

UNIVERSITE CATHOLIQUE DE LOUVAIN
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**A multiscale multiphysics
investigation of aluminum friction
stir welds**

**from thermal modelling to mechanical
properties through precipitation
evolution and hardening**

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par

Aude Simar

Membres du jury :

B. de Meester (promoteur)
T. Pardoen (promoteur)
J-C. Samin (président)
Y. Bréchet
F. Delannay
A. Denquin
F. Dupret

à Sébastien, mon mari
à Aurélien, mon fils
à mes parents

Résumé

L’objectif de cette thèse est la compréhension des phénomènes thermiques, mécaniques et métallurgiques qui se produisent lors du soudage par friction-malaxage de l’alliage d’aluminium 6005A et la détermination des propriétés mécaniques des joints soudés.

Un dispositif expérimental permet de mesurer tant les efforts et le couple nécessaires au soudage que les cycles thermiques encourus dans les différentes zones des joints. Un premier modèle permet de prédire l’influence des paramètres opératoires sur les cycles thermiques observés dans les différentes zones des joints soudés, et en particulier sur l’asymétrie des évolutions de température par rapport au centre de la soudure. Un second modèle relie les évolutions locales des microstructures, en particulier les cinétiques de précipitation, aux cycles thermomécaniques. Un troisième modèle vise à relier les microstructures locales aux propriétés mécaniques locales et inclut un nouveau modèle d’écrouissage à base physique. Finalement, le lien entre les propriétés mécaniques des joints en service, les propriétés mécaniques locales et les microstructures est modélisé dans une approche multi-échelle incluant un modèle constitutif d’endommagement à fondement micromécanique.

Les modèles sont étalonnés et validés à partir de la caractérisation fine des microstructures et des essais mécaniques effectués tant sur le matériau de base et sur le matériau traité thermiquement que sur des joints soudés par friction malaxage.

Abstract

The aim of the thesis is the understanding of the thermal, mechanical and metallurgical phenomena occurring during friction stir welding of a 6005A aluminum alloy and the determination of the mechanical properties of the welded joints.

The forces and the torque needed for welding as well as the thermal cycles were measured in the various zones of the joint. A first model predicts the influence of the welding parameters on the thermal cycles, and especially on the asymmetry of the temperature evolution with respect to the weld centerline. A second model links the local microstructural evolutions, and in particular the precipitation kinetics, to the thermo-mechanical cycles. A third model relates the local microstructures to the local mechanical properties, based on a novel physics-based strain hardening model. Finally, the link between the mechanical properties of the joint in service, the local mechanical properties and the microstructures is modeled using a multi-scale approach including a micro-mechanics damage constitutive model.

The models are calibrated and validated through in-depth microstructure characterization and mechanical tests on the base material, on heat treated samples and on the friction stir welds.

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List of symbols

Latin symbols

Symbol and units	Description
A_0 [kJ/mol]	Parameter for the energy barrier for nucleation
b [m]	Magnitude of Burger's vector
C	Function of S_v and f
C_0 [wt %]	Magnesium content of the base alloy
CC_A [W/m ² K]	Contact conductivity between the workpiece and the backing plate under the shoulder
CC_B [W/m ² K]	Contact conductivity between the workpiece and the backing plate at the back of the tool
C_{Cu} [wt %]	Copper content in the solid solution
$C_{Cu_{init}}$ [wt %]	Initial copper content in the solid solution
C_e [wt %]	Equilibrium magnesium concentration in solid solution at the precipitate/matrix interface
C_{e0} [wt %]	Pre-exponential term to C_e
C_i [wt %]	Magnesium content at the precipitate/matrix interface
C_{lim} [wt %]	Limit magnesium content for natural aging
C_{Mg} [wt %]	Magnesium content in the solid solution
c_p [J/kgK]	Specific heat of the base alloy
C_p [wt %]	Magnesium content in Mg ₂ Si
C_{Si} [wt %]	Silicon content in the solid solution
d [m]	Distance between the two thermocouples inserted in the tool
D [m ² /s]	Diffusion coefficient
D_0 [m ² /s]	Pre-exponential term to the diffusion coefficient
e	Elongation
E [Pa]	Young modulus
E_a [J/m ²]	Antiphase boundary surface energy
e_u	Ultimate tensile strain
e_x, e_y, e_z	Unit vectors parallel to the x, y and z direction respectively
f	Porosity

Symbol and units	Description
f_0	Initial porosity
$\overline{F}$ [N]	Mean obstacle strength
F_i [N]	Obstacle strength of the precipitate in the size class i
f_v	Volume fraction of precipitates
F_x, F_y, F_z [N]	Forces in the x, y and z direction respectively
g	Function of S_v and f
G [Pa]	Shear modulus
h_1	Function of S_v and f
h_2	Function of S_v and f
h_p [m]	Pin height
HV [HV1]	Vickers hardness
j [#/ m^3s]	Nucleation rate
J	Tensor associated to the void axis
j_0 [#/ m^3s]	Pre-exponential term to the nucleation rate
K [N/ $m^{5/2}$]	Precipitation strengthening constant for $r < r_{trans}$
K' [N/m]	Precipitation strengthening constant for $r > r_{trans}$
k_1 [m^{-1}]	Proportionality constant for dislocation storage
k_2	Proportionality constant for dynamic recovery
k_β	Constant linking the yield strength to the dynamic recovery rate β
k_{Cu} [Pa wt% $^{2/3}$]	Constant for the effect of the copper content in solid solution on the yield strength
k_{Mg} [Pa wt% $^{2/3}$]	Constant for the effect of the magnesium content in solid solution on the yield strength
$k(Mg, Si)_{eq}$ [Pa wt% $^{2/3}$]	Constant for the effect of the magnesium and silicon content in solid solution on the yield strength
K_{NA} [Pa]	Constant for the natural aging model
k_{Si} [Pa wt% $^{2/3}$]	Constant for the effect of the silicon content in solid solution on the yield strength
$k_{\theta dp}$ [Pa]	Constant for the dynamic precipitation effect on θ
l [m]	Particles distance in the glide plane of the dislocation
L_0 [m]	Distance between two moving dislocations of opposite sign
L_{jjkl} [Pa]	Elastic moduli
l_L [m]	Largest edge thickness of the reduced section of a micro-tensile sample
l_S [m]	Smallest edge thickness of the reduced section of a micro-tensile sample
M	Taylor factor
M_z [Nm]	Torque along z axis
n	Strain hardening exponent
N [#/ m^3]	Density of precipitates

Symbol and units	Description
N_i [$\#/\text{m}^3$]	Density of precipitates in the size class i
n_u	Strain hardening exponent at necking
n_t	Number of thermocouples
P [W]	Total mechanical power
P_d	Projector tensor
P_{in} [W]	Total mechanical power inputed in the weld
$p(m)$	Mean probability for a moving dislocation to meet m precipitates having an Orowan loop surrounding it
$p_{shoulder}$ [Pa]	Pressure under the shoulder
q	Parameter calibrated as a function of f_0 and W_0
Q_d [J/mol]	Activation energy for diffusion
Q_e [J/mol]	Apparent solvus boundary enthalpy
q_S [W/m ²]	Specific surface heat source
Q_S [W]	Surface heat source
Q_{tool} [W]	Heat going into the tool
q_V [W/m ²]	Specific volume heat source
Q_V [W]	Volume heat source
r [m]	Mean equivalent precipitate radius
R [Pa]	Engineering stress
r_c [m]	Critical equivalent precipitate radius
r_{cl} [m]	Precipitate radius of loss of coherency
R_g [J/K mol]	Universal gas constant
r_i [m]	Mean equivalent precipitate radius of the size class i
R_m [Pa]	Ultimate tensile strength
R_{mBM} [Pa]	Ultimate tensile strength of the base material
r_{min} [m]	Minimum radius of the TMAZ
r_p [m]	Pin radius
R_p [Pa]	Yield strength
r_S [m]	Shoulder radius
r_{trans} [m]	Radius of transition between dislocation shearing the precipitates and dislocations bypassing the precipitates
R_x, R_y, R_z [m]	Radius of voids in the x , y and z direction respectively
S [m ²]	Minimum section of a tensile sample
S_i [m ²]	Initial section of a tensile sample
S_r [m ²]	Reduced section of a tensile sample at fracture
S_v	Logarithmic aspect ratio of voids
t [s]	Time
T [K]	Temperature
T_0 [K]	Room temperature
T_3 [K]	Maximum temperature measured at 3 mm from the tool shoulder
T_{13} [K]	Maximum temperature measured at 13 mm from the tool shoulder

Symbol and units	Description
t_d [s]	Traveling time associated to the mean spacing between two dislocations of opposite sign
T_{exp}^i [K]	Maximum temperature measured by thermocouple i
T_{model}^i [K]	Maximum temperature predicted at the position of thermocouple i
t_{preci} [s]	Inverse of the frequency of precipitates distribution
T_r [m]	Reduced thickness of a tensile sample
v [m/s]	Advancing speed
$v_{d/c}$ [m/s]	Rate of dissolution or coarsening of precipitates
V_m [m ³ /mol]	Molar volume of Mg ₂ Si
v_{mat} [m/s]	Material velocity
V_p [m ³]	Volume of the TMAZ
v_r [m/s]	Relative velocity of two moving dislocations
W	Aspect ratio of the voids
W_0	Initial aspect ratio of the voids
W_{area} [J]	Area energy of a dislocation
W_{l+p} [J]	Free energy of the system loop + precipitate
W_{PK} [J]	Work of the Peach K��hler force
W_r [m]	Reduced width of a tensile sample
x [m]	Welding direction
X	tensor associated to the void axis
y [m]	Transverse direction
y_0 [m]	Annihilation distance between two dislocations of opposite sign in a pure material
y_a [m]	Annihilation distance between two dislocations of opposite sign in an alloy
y_{preci} [m]	Annihilation distance between two dislocations of opposite sign when at least one precipitate sur-rounded by an Orowan loop is met during the time required to meet another moving dislocation
z [m]	Through thickness direction

Greek symbols

Symbol and units	Description
α	Constant of the dislocation density contribution to the flow stress
α_2	Function of S_v and f
α_d	Damage model parameter, function of the strain hardening exponent
β	Dynamic recovery rate strain hardening parameter
β_0	Dynamic recovery rate strain hardening parameter of a pure material
β_{min}	Minimum of β
β_{SHT}^{init}	Dynamic recovery rate strain hardening parameter β of a solutionized sample
β_Γ	Dislocation line tension constant
γ	Proportion of P_{in} associated to volume heat sources
Γ [N]	Dislocation line tension
γ_d	Geometric factor depending on the voids arrangement
γ_i [J/m ²]	Precipitate/matrix interface energy
γ_p	Local plastic shear strain
δ	Ratio of the material velocity to the tool velocity at the tool/workpiece interface
ΔG_{het}^* [J/mol]	Energy barrier for heterogeneous precipitation
δ_K	Kronecker tensor
ε	True strain
ε_f	Macroscopic true fracture strain
ε_f^{micro}	Microscopic true fracture strain
ε^p	Plastic strain tensor
ε_p	Equivalent plastic strain
ε_u	True strain at the onset of necking
ζ	Parameter of heat source generated by plastic deformation
η	Welding efficiency
η_d	Function of S_v and f
θ [Pa]	Dislocation storage strain hardening parameter for an alloy
θ_0 [Pa]	Dislocation storage strain hardening parameter for a pure material
θ_{dp} [Pa]	Dynamic precipitation contribution to θ
θ_{SHT} [Pa]	Dislocation storage parameter θ of a solutionized sample
κ	Function of S_v and f
λ [W/mK]	Thermal conductivity of the base alloy
λ_d	Particle distribution factor
λ_{steel} [W/mK]	Thermal conductivity of steel

Symbol and units	Description
μ	Friction coefficient
ν	Number of precipitates encountered before two moving dislocations meet
ν_φ	Number of precipitates having an Orowan loop efficiently stored around it encountered before two moving dislocations meet
ρ [#/ m^3]	Dislocation density
ρ_a [kg/m^3]	Density of the base alloy
ρ_{init} [#/ m^3]	Initial dislocation density
σ [Pa]	Cauchy stress tensor
σ' [Pa]	Deviatoric tensor part of the Cauchy stress tensor
σ_0 [Pa]	Yield strength of a pure aluminum
σ_c [Pa]	Critical flow stress for Orowan loops annihilation
σ_d [Pa]	Grain size contribution to the flow stress
σ_f [Pa]	Flow stress
σ_h^g [Pa]	Generalized hydrostatic stress
σ_{pl} [Pa]	Dislocation density contribution to the flow stress
σ_{preci} [Pa]	Precipitation contribution to the flow stress
σ_{sat} [Pa]	Saturation stress
σ_{ss} [Pa]	Solid solution contribution to the flow stress
σ_u [Pa]	Flow stress at necking
σ_y [Pa]	True yield strength
σ_y^{max} [Pa]	Maximum yield strength
σ_{yNA} [Pa]	Yield strength after natural aging
σ_{ySHT}^{init} [Pa]	Yield strength after a solution heat treatment
σ_{ySHT}^{NA} [Pa]	Yield strength after a solution heat treatment and natural aging
τ [Pa]	Applied shear stress
τ_c [Pa]	Critical flow shear stress for Orowan loops annihilation
$\tau_{contact}$ [Pa]	Shear stress at the tool/workpiece interface
τ_f [Pa]	Flow shear stress
τ_y [Pa]	Flow shear stress at the beginning of yielding
φ	Efficiency of dislocation storage
Φ	Flow potential
χ	Void spacing
ω [rad/s]	Rotational speed

Part I

General introduction

Chapter 1

Introduction

Aluminum alloys are characterized by high strength to weight ratio, low density, good corrosion resistance, adequate formability and moderate to high fracture toughness. In the transportation industry, the constant need to diminish the CO₂ emissions provides an impetus for weight reductions favoring the use of aluminum rather than steel. Extruded profiles of the 6xxx aluminum alloy family and in particular the 6005A aluminum alloy are therefore interesting in many applications (see Figure 1.1).

The problem with the extrusion presses is that they are limited in size and capacity. Therefore a sound welding method is useful to join extruded or rolled Al alloy sheets and plates, especially if it is less expensive than mechanical fastening. Fusion welding aluminum causes important loss in ductility due to solidification defects (cracking, porosities). Furthermore, not all the fusion welding processes are fully automated nor offer high productivities. Laser welding could be an alternative to more classical fusion welding techniques but the high level of defects produced

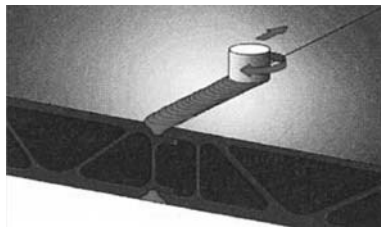


Figure 1.1. Industrial application of the friction stir welding process: welding of an extruded profile for the railway industry (© Hitachi website, Ltd. 1994, 2006)

when welding aluminum alloys is a clear drawback of that process. Friction stir welding offers the advantages of a potentially productive process without the defects typical of fusion welds. Hence, many industries already use friction stir welding for specific applications.

Many aspects of the friction stir welding process and of the ensuing mechanical properties are still badly understood and require further studies, especially with respect to quantitative structural integrity assessment and forming issues. Therefore, a fundamental study about the origins of the mechanical and damage properties of the weld has a great interest in the optimization of the process for further applications. High welding speeds should not be in conflict with the quality of the welds produced. A good understanding and prediction of the variation of the strength of the weld for higher advancing speeds makes certainly sense in that perspective.

Figure 1.2 summarizes the underlying methodology and program followed in this Ph.D. research work. The process parameters are often linked to the mechanical properties of the weld without understanding the thermo-mechanical cycles and the microstructural evolutions which created these properties. To get more insight into the process, a series of modeling tools have been developed and validated by experiments on the unwelded alloy and on friction stir welds with different welding conditions.

The first part of this thesis is made of three chapters. It provides an introduction to the metallurgy of aluminum alloys and to the friction stir welding process as well as to the experimental methods. The material studied in this work is a 6005A-T6 aluminum alloy provided by the company SAPA RC profiles. The properties of this alloy in the as received condition are also presented in part I.

The second part of this thesis is made of two chapters. The results of the thermomechanical measurements (i.e. the forces, torque and temperature) performed during welding are described in chapter 4. In chapter 5, a model for the thermal cycles during the welding process is developed, identified and validated.

The third part of this thesis presents the complete microstructural and mechanical characterization of some of the friction stir welds produced. Three models make the link between the thermal cycles and the ensuing

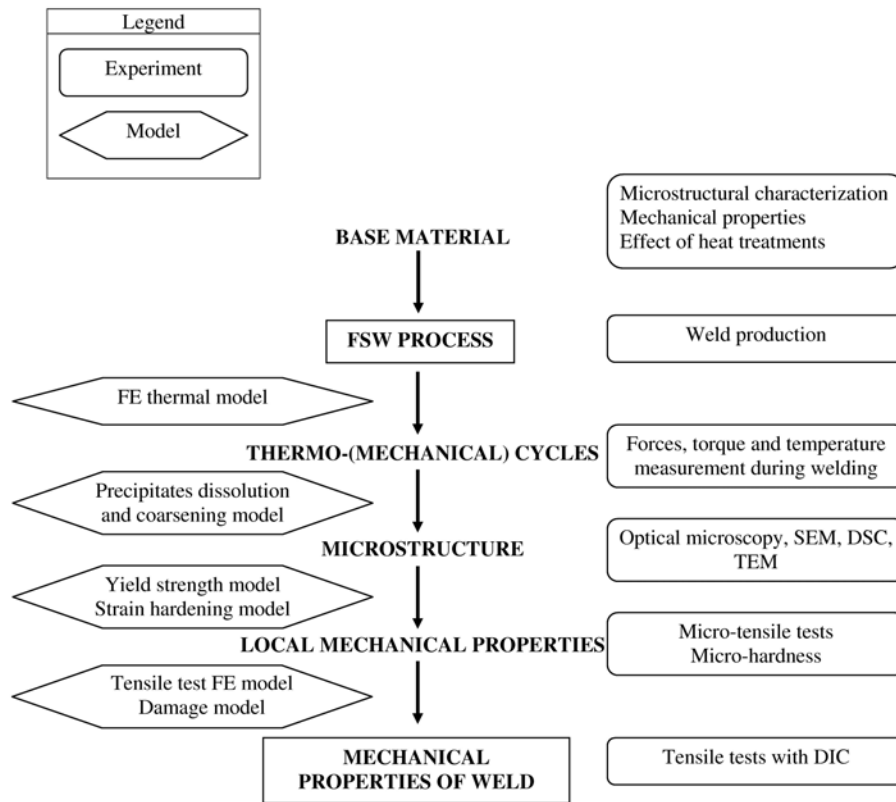


Figure 1.2. Principle of the research project

mechanical properties. A first model, described in chapter 7, predicts the microstructural evolutions during welding. The second model, also presented in chapter 7, predicts the yield strength. Chapter 8 concerns the modeling of strain hardening. The strain hardening model developed has the advantage compared to the existing models found in the literature to be mainly based on the microstructure of the alloy rather than dependent on phenomenological parameters.

The final part of this thesis (part IV) concerns the global mechanical properties of friction stir welds and the modeling chain. Chapter 9 describes the macro-mechanical properties measured on different welds and the model which links the local mechanical properties to the global ones through a finite element model. This model was first tested on 6056 aluminum alloy friction stir welds [51]. A physically based damage model allows to predict the true fracture strain of the welds. Before the conclusions, chapter 10 assesses the entire modeling chain and tends to conclude on the effects of the welding parameters on the global behavior of friction stir welds in 6005A-T6 aluminum alloy.

Chapter 2

Background

2.1 6xxx aluminum alloys

2.1.1 Classification and composition

The 6xxx series aluminum alloys have magnesium and silicon as principal alloying elements which, generally speaking, harden the material by the precipitation of precursors of the stable phase Mg_2Si . Manganese and chromium additions have a limited effect on the mechanical properties but allow the control of the recovery, recrystallization, grain growth and sometimes nucleation of the strengthening precipitates [40, 50, 79]. Copper addition enhances the hardness and refines the microstructure [91].

Two other sub-classification criterion for the 6xxx series alloys are of importance when looking at the microstructures.

- The silicon content: The alloys often contain an excess in silicon which respect to what is needed to form Mg_2Si precipitates. The consequence is the precipitation of the excess silicon in silicon rich precipitates. The excess of silicon promotes the formation of additional particles which do not contribute to hardening (B' , Type A , Type B). When the excess silicon content is very high (1.32% silicon for 0.6% magnesium [57]) the effective amount of hardening phase is increased compared to alloys with lower excess silicon.
- The copper content: Copper modifies the precipitation sequence and favors the precipitation of the Q -phase ($\text{Al}_5\text{Cu}_2\text{Mg}_8\text{Si}_6$) [56, 85]. Literature is not clear about the limit copper concentration required to favors this type of precipitation since a confusion often

exists between the B' (typical of high excess silicon alloys) and Q phases due to their close compositions [85].

The 6xxx series aluminum alloys are age hardenable alloys. Classical state of material delivery are:

- T4 state: solution treated, quenched and naturally aged at room temperature, this state offers a good formability but relatively low strength [90];
- T6 state: solution treated, quenched and artificial aged to the peak strength;
- T5 state: similar to the T6 state but no solution treatment followed by quench has been performed beforehand;
- T78 state: slightly over-aged material (offers sometimes better corrosion properties for high copper content alloys).

2.1.2 β phase and its meta-stable hardening precursors

In low copper and low excess silicon content 6xxx aluminum alloys¹, the hardening phases are the metastable precursors of the stable Mg_2Si phase. The generally accepted precipitation sequence is the following [34, 36, 56, 58, 90]²:

Supersaturated solid solution $\rightarrow$ clusters/ GP zones $\rightarrow \beta'' \rightarrow \beta' + B' + Si + (\text{Type } A \text{ and } B) \rightarrow \beta(Mg_2Si) + Si$

The GP zones are produced during low temperature artificial aging or natural aging at room temperature. They provide moderate hardening. The precipitates β'' are fully coherent precipitates, formed in particular during the artificial heat treatment T6, and responsible for the peak hardness when their size is optimal (10–15 nm long in the 6061 aluminum alloy [36]). The precipitates following β'' in the precipitation sequence have a minor hardening potential [56]. Their formation correspond to over-aged conditions. The β' precipitates are semi-coherent precipitates. The main precipitates shape, crystalline structure and compositions are

¹The alloy 6005A of this study is an excess silicon, low copper content, 6xxx series alloy. Therefore, we will focus in the following on the hardening phases present in these specific types of alloys.

²Note that the general tendencies of the precipitation sequences are similar for all these authors but small differences exist, especially concerning the clusters and the non-hardening phases formation.

represented in Table 2.1 (note that sometimes literature presents variations on that subject).

2.1.3 Non-hardening precipitates

During the casting of industrial aluminum alloys of the series 6xxx, the presence of other alloying elements than magnesium and silicon as well as the excess silicon can lead to incoherent coarse phases. These can be classified in two main categories: the intermetallics which are iron rich inclusions and the dispersoids which contain less iron and are manganese and/or chromium rich precipitates.

Coarse Mg_2Si phases are formed during the material elaboration and the very large ones are not dissolved during homogenization. Si rich phases can be formed due to the excess silicon which respect to what is needed to form Mg_2Si precipitates. These phases will be assimilated to intermetallics in the following since they have similar effects on the mechanism of damage of the alloy and have similar sizes.

Intermetallics are Al-Fe, Al-Fe-Si and Al-Fe-Mn-Si phases formed between the aluminum dendrites during cooling [70]. These phases have a typical size of 1 to 10 μm . The homogenization treatment after casting aims at transforming interconnected plate-like $\beta\text{-Al}_5\text{FeSi}$ in more rounded and discrete $\alpha\text{-Al}_{12}(\text{FeMn})_3$ and dissolving coarse Mg_2Si precipitates [69, 73].

Dispersoids are $\alpha\text{-Al}(\text{FeMn})$ phases formed during the homogenization of the alloy. These phases have a typical size of 100 nm. They nucleate heterogeneously on the β' precipitates during the homogenization treatment and are incoherent with the aluminum matrix. The β' phases segregate toward the dendrites boundaries during casting [62] (and will later dissolve during the homogenization treatment). This explains the inhomogeneous distribution of dispersoids in the aluminum matrix [78].

2.2 The friction stir welding process

2.2.1 Principle of the friction stir welding process and main features

Friction stir welding (FSW) is a solid state welding technique patented by TWI in 1991 [135]. The parts or components to be welded are rigidly clamped on the machine support. A rotating tool, composed

	Shape and preferential orientation	Crystalline structure	Composition
β''	needle along $\langle 100 \rangle_{Al}$ [56, 90] and polyhedral cross-section [148]	monoclinic $a = b = 6.16\text{\AA}$ $c = 7.1\text{\AA}$ $\gamma = 82^\circ$ [116] $a = 3.0\text{\AA}$ $b = 3.3\text{\AA}$ $c = 4.0\text{\AA}$ $\gamma = 71^\circ$ [116] $a = 15.3\text{\AA}$ $b = 4.05\text{\AA}$ $c = 6.8\text{\AA}$ $\beta = 106^\circ$ (BC) [36] hexagonal $a = 7.05\text{\AA}$ $c = 4.05\text{\AA}$ [65]	$\text{Mg}_{1.15}\text{Si}$ [36] Mg_5Si_6 [2] varies with alloy composition [95]
β'	rod and circular cross-section along $\langle 100 \rangle_{Al}$ [56]	hexagonal $a = 7.05\text{\AA}$ $c = 4.05\text{\AA}$ [36, 65, 80, 84, 143]	$\text{Mg}_{1.7}\text{Si}$ [86] $\text{Mg}_{0.44}\text{Si}$ [80]
B'	lath-like and rectangular cross-section with long-face oriented at 11° to $\langle 100 \rangle_{Al}$ [36, 33]	orthorhombic [33, 36] $a = 9.0\text{\AA}$ $b = 5.2\text{\AA}$ $c = 4.05\text{\AA}$; $a = 18\text{\AA}$ $b = 10.4\text{\AA}$ $c = 4.05\text{\AA}$. hexagonal $a = 10.4\text{\AA}$ $c = 4.05\text{\AA}$.	$\text{Mg/Si} = 0.9...1.2$ [86]
β	platelets on $\{100\}_{Al}$	FCC CaF_2 (anti-fluorite) $a = 6.39\text{\AA}$ [65, 134]	Mg_2Si

Table 2.1. Shape, preferential orientation, crystalline structure and composition of the meta-stable precursors and of the stable β phase.

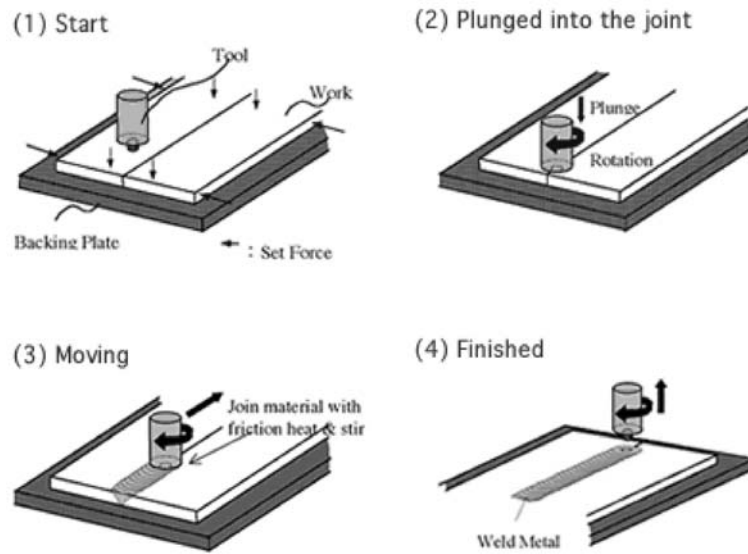


Figure 2.1. Principle of the friction stir welding process (© Hitachi, Ltd. website 1994, 2006)

of a threaded pin and a shoulder, is introduced into the material at the joint axis until the shoulder is in contact with the upper surface of the plates. Heat generated by friction and deformation brings the material in an extremely malleable state that allows the tool to move forward with limited effort and the material to flow from the front to the back of the tool where it cools down (see Figure 2.1). The weld is asymmetric since on one side of the weld, called the advancing side, the rotation of the tool is in the direction of the advancing velocity, while on the other side, called the retreating side, it is moving in the opposite direction.

The quality of a friction stir weld is highly dependent on the tool geometry. The presence of a tread on the pin allows welding thick plates without defects but many other geometries have been imagined by tool designers in order to improve the welds quality. Figure 2.2 shows two examples of tools developed by The Welding Institute (TWI), UK. The friction stir welding machines classically used for weld production are of two types: displacement or force controlled. The displacement controlled machines are highly rigid milling machines. The force controlled machines impose the desired vertical force at all times during welding. One of the advantages of force controlled machines is the tolerance to workpiece gaps and small thickness differences [93].

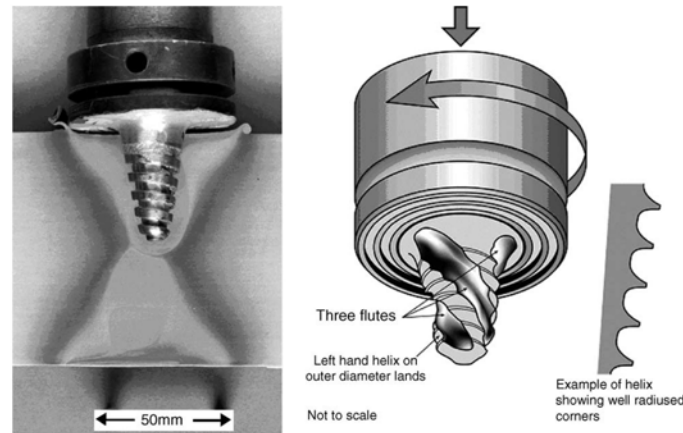


Figure 2.2. WorlTM and MX TrifluteTM tools developed by TWI, UK (Copyright 2001, TWI Ltd) (from [136])

Friction stir welding was initially developed for welding aluminum alloys but nowadays many other materials have been successfully welded with this process. Studies have been made on copper, magnesium, titanium, thermo-plastics, steel and stainless steel (see references [175-232] of [92]). The problem when welding high melting temperature alloys (i.e. steel) is the need for specific tooling made of highly resistant materials, which constitutes the main limitation of the process for friction stir welding those materials.

2.2.2 Advantages and limitations of the process

Friction stir welding has many advantages, including the following:

- FSW is a solid phase process avoiding thus, in principle, problems of hot cracking and porosity.
- Low distortions are observed due to the relatively low temperatures associated to this solid state process and to the rigid clamping of the plates.
- There are no consumables, filler metal or gas.
- There are no fumes, no UV radiations, no spatters and, therefore, the process is environmentally friendly.
- The procedure can easily be automated, requiring limited training of the welders.

- Welding can be carried out in any position since no fused metal bath is present.
- High joint efficiencies³ has been achieved in aluminum alloys (see Table 8 of [92] and Table 3 of [55]).
- Alloys generally classified as unweldable by fusion processes (e.g. 2xxx [110] and 7xxx [83] series aluminum alloys) give high quality friction stir welds.
- Dissimilar alloys can be welded together giving high quality welds (see Table 24 of [92]).
- The ability to butt join two materials of different thickness allows reducing the weight of structures to be welded by an appropriate design.
- It is an energy efficient process, only 2.5% of the energy used for laser welding is used [92].

The following limitations must be underlined:

- Specific FSW defects are observed (kissing bonds and tunnel defects) when the welding parameters are not adapted.
- The rigid clamping of the plates leads to significant residual stresses (see Figure 2.3).
- An exit hole is left over after withdrawing the FSW tool, unless a pin retractable tool is used (NASA patent).
- Significant down force and transverse forces are applied and consequently very stiff machines are required.
- FSW machines are not very flexible even though the robotizing of the process has been improved over the last years.
- The length of the tool pin has to be adapted to the different thicknesses to be welded.
- An expensive TWI patent license is required for using the process.

³The definition of the joint efficiency will be given later. It relies on the loss of strength with respect to the base material.

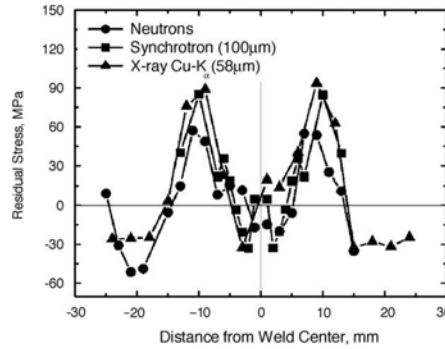


Figure 2.3. Longitudinal residual stress distribution in 6013 – T4 friction stir welds determined by different measurements methods, the shoulder diameter is of 15 mm (from [22]).

The high quality of the welds produced and the reproducibility of this fully automated process has provided the impetus for many industries to already implement it in production. The first friction stir welding installation was made at Hydro Marine Aluminum (Norway) to assemble large panels for base structure of ferries. Later, aeronautics and aerospace applications have been developed. The Eclipse Aviation Corporation’s business jet and the Boeing Company’s fuel tanks of the space shuttles Delta II and Delta IV are some of the early applications of the process in these industries. Automotive and railway industries have also jointed parts by friction stir welding.

2.2.3 Microstructural zones in a friction stir weld

A friction stir welding joint consists of various zones involving different microstructures and mechanical properties, as shown on Figure 2.4. The heat affected zone (HAZ) is the furthest away from the joint line and is not deformed but the dissolution and coarsening of precipitates due to the thermal cycles influence the mechanical properties of this zone. The thermo-mechanically affected zone (TMAZ) and the nugget have been highly deformed. Contrarily to the TMAZ, the nugget is dynamically recrystallized [76, 129].

The dislocation density in the nugget zone is very low compared with the TMAZ [11, 75, 76, 111, 115, 142]. The nugget grains are equiaxed and much smaller size (several μm) than the base material grains. This small grain size has apparently a second order effect on the hardness of the nugget in age-hardenable aluminum alloys [75]. Under specific

welding conditions, so-called onion rings can be observed in the nugget (see macrograph in Figure 2.4).

2.2.4 Precipitates evolution of friction stir welds in 6xxx series aluminum alloys

The coarse phases in the friction stir welds are essentially broken in smaller particles in the central zone of the weld [75, 76]. However, C. Gallais [50] observed no significant reduction in the size of these phases in the nugget zone of a 6056 friction stir weld. Nevertheless, he suggested that the reduction of volume fraction observed in that region compared to the base material could be due to the breakage of these phases in so tiny phases that they were under the detection limit. Sutton *et al.* [130, 131, 147] have shown that the coarse phases observed by SEM are non uniformly distributed in the nugget zone of a 2024-T3 friction stir weld. Particles rich bands are separated by particles poor bands and their spacing seems to be related to the welding tool advance per revolution. Similar conclusions to those proposed by Gallais [50] were drawn by these authors: the lower value of the particles volume fraction in the weld center cannot be caused by dissolution at the temperatures imposed by the friction stir welding process. Sutton *et al.* [131, 147] obtained a surprising result: the average particle diameter was clearly lower in the weld center but higher on the retreating side than on the advancing side.

Sato *et al.* [113] studied the evolution of the hardening precipitates by TEM observations throughout a 6063-T5 friction stir weld. The base material contains a great amount of needle-shaped precipitates. In the region where the hardness starts to drop, the density of needle-shaped precipitates decreases while the density of rod-shaped precipitates increases. The minimum hardness region has a low density of large rod-shaped precipitates. In the central zone of the weld, all the precipitates are dissolved. Figure 2.5 shows the relationship between hardness and hardening precipitates evolution. The TEM campaign of Sato *et al.* [115] hardly revealed any apparent differences in precipitate distribution between the advancing and retreating sides. Lee *et al.* [75] got similar conclusions to Sato *et al.* [113] from their TEM observation of a 6005-T6 FSW. The needle-shaped precipitates are identified as β'' precipitates and the rod-shaped precipitates as β' precipitates. Sato *et al.* [114] showed that a post-weld heat treatment on a 6063-T5 friction stir weld forces the precipitation of β'' phases in regions where at least a part of the magnesium and silicon is in solid solution.

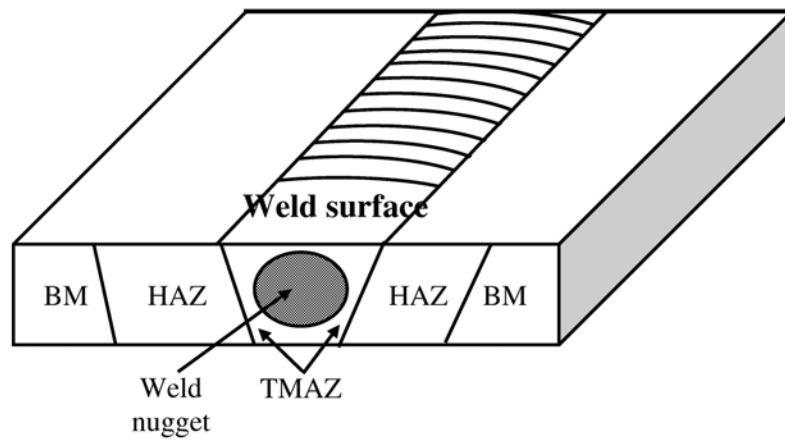
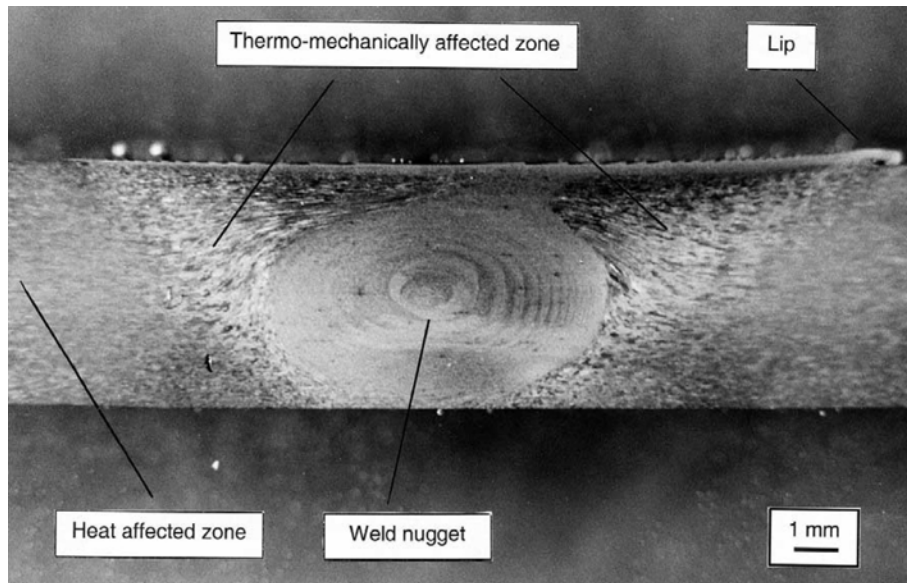


Figure 2.4. Microstructural zones of a friction stir weld (macrograph from [10]). BM = base material; HAZ = heat affected zone; TMAZ = thermo-mechanically affected zone.

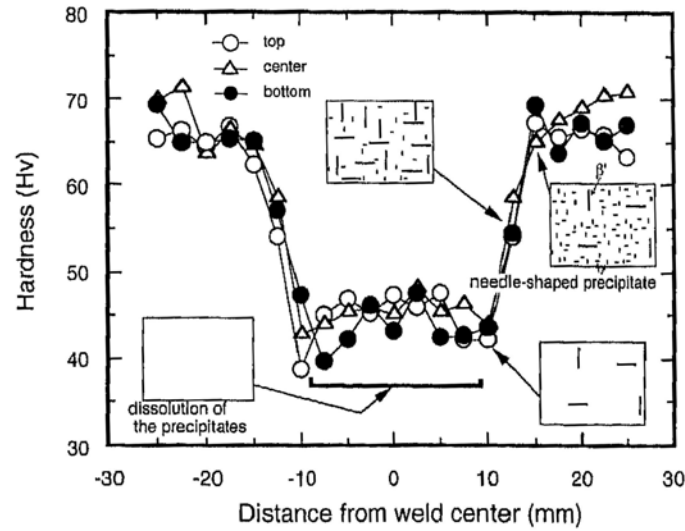


Figure 2.5. Relationship between precipitate distribution and the hardness profile in a 6063-T5 friction stir weld (from [113])

The precipitation evolution in friction stir welds of higher copper content 6xxx series aluminum alloys seems to be different. Liu *et al.* [76] identified a complex fine precipitation evolution in the heat affected zone by observing 6061-T6 friction stir welds with TEM. They concluded that the hardness drop zone is composed of lath-like precipitates containing more copper than the base material fine precipitates. They observed far fewer large precipitates in the weld center. Svensson *et al.* [132] and Denquin *et al.* [29] studied respectively a 6082 and a 6056 friction stir weld and showed heterogeneous precipitation on the dispersoids that have respectively been identified as β' phases and Q phases. Denquin *et al.* [29] showed that a post-weld heat treatment induces a homogeneous fine precipitation inside the grains.

2.2.5 Mechanical tensile properties of 6xxx series aluminum alloys friction stir welds

Two types of tensile tests can be performed on the welds in order to determine their mechanical properties:

- classical transverse tensile tests where all the zones of the weld are deformed at the same time, and
- local tensile tests on the different zones of the weld.

The transverse tensile samples are made of an inhomogeneous material and does thus not provide intrinsic material properties. These tests mostly allow the identification of the location of fracture and the determination of the joint efficiency (ratio of the joint to the base material ultimate tensile strength). These are two basic indications of the quality of a weld allowing the comparison between different welding conditions and processes. Joint efficiency values for friction stir welded 6xxx series aluminum alloys are case dependent with values ranging from 60% to 90% [75, 92, 110, 142].

Fracture of transverse tensile samples generally occurs in the heat affected zone on the retreating side of the weld for the welds of the 6xxx aluminum alloy series containing low copper concentration [77, 110, 115]. Fracture of friction stir welds of other aluminum series does not systematically occur on the same side of the weld nor systematically in the heat affected zone. Sato *et al.* [115] and Denquin *et al.* [29, 30] showed that the position of fracture is linked to the minimum hardness and corresponds to the outer limit of the stirred zone if no post-weld heat treatment is applied. Sato *et al.* [115] showed that this minimum hardness moves away from the centerline when a post-weld heat treatment of the T6 type is applied. This change of position is linked to the microstructure and in particular to the solid solution concentration evolution in the friction stir welds. Denquin *et al.* [29, 30] did not observe this move of the minimum hardness but the alloy studied (a 6056-T78) contained more copper which drastically changes the precipitation modes. Liu *et al.* [77] studied 6061-T6 friction stir welds and showed that the position of fracture gets closer to the weld centerline when the advancing speed increases.

The local tensile properties of a friction stir weld have not been so extensively studied since extracting small tensile samples from the welds is not obvious and is time consuming. Nevertheless the studies by von Strombeck *et al.* [142], Sato *et al.* [115], Denquin *et al.* [29, 30] and Cabibbo *et al.* [11] have to be mentioned. These authors concluded that the yield strength variation is similar to the hardness variation. The definition of the fracture strain parameters being unclear, it is not easy to draw general conclusions on its evolution. Nevertheless, a general trend is that the fracture strain of the weld central region is lower than the fracture strain of the base material if the base material is in the T4 or in the T6 state. von Strombeck *et al.* [142] extracted tensile specimens from the different zones of a 6061-T6 friction stir weld. The HAZ had

	Base material	HAZ	TMAZ	Weld nugget	TMAZ	HAZ	Base material
6061-T6 (von Strombeck <i>et al.</i>)	R_p maximum R_m high n_u low	R_p low R_m low n_u low		R_p low R_m medium n_u medium		R_p low R_m high n_u high	
6056-T4 (Gallais <i>et al.</i>)	R_p medium R_m high n_u high	R_p low R_m low n_u low		R_p medium R_m medium n_u medium		R_p medium R_m high n_u high	
6056-T78 (Gallais <i>et al.</i>)	R_p high R_m medium n_u low	R_p low R_m low n_u low		R_p medium R_m medium n_u medium		R_p medium R_m high n_u high	

Figure 2.6. Evolution of the local tensile properties throughout a friction stir weld for different base materials of the 6xxx aluminum alloy series (out of von Strombeck *et al.* [142] and Gallais *et al.* [51]). Lowest values are in bold. R_p is the yield strength; R_m is the ultimate tensile strength and n_u is the strain hardening exponent at necking.

a lower strain hardening ability than the weld center region. This gives a different distribution of the ultimate tensile strength from the yield strength distribution.

The author has contributed to the study by Gallais *et al.* [51] which aimed at understanding and predicting the tensile behavior of 6056-T78 and T4 friction stir welds. The weld center region had a good hardening ability (high strain at necking value) equivalent to the T4 base material level. Minimum values of the strength and strain at necking were observed in the heat affected zone closest to the TMAZ. The strain to fracture of the base material is lower than that of the HAZ weakest zone. The T4 state has lower values of the strain to fracture than the T78 state. The importance of the coarse phases and the dispersoids density on the fracture process are put forward in that study.

Figure 2.6 shows the variation of the local tensile properties throughout a friction stir weld for different base materials as found in the work by von Strombeck *et al.* [142] and by Gallais *et al.* [51]. Even though the distribution in tensile properties vary for different base materials, in particular regarding the yield strength distribution, the weakest zone is always the heat affected zone.

Chapter 3

Experimental methods, tooling and base materials

3.1 Experimental welding device

3.1.1 Conventions

The z direction is the direction of the plate thickness ($z = 0$ on the top surface of the workpiece), the x direction is the welding direction ($x = 0$ at the center of the tool when starting the welding) and the y direction is the transverse direction pointing toward the retreating side of the weld ($y = 0$ at the center of the weld). The tool always rotates in the clockwise direction.

3.1.2 Welding machine and tools

A HERMLE 3-axis CNC milling machine (UWF 1001 H) has been adapted to make friction stir welds. The main characteristics of that milling machine are:

- Maximal nominal power : 12 *kW*,
- Working space (x, y, z) : 700 x 550 x 500 mm,
- Nominal position accuracy : 0.01 mm,
- Advancing speed : varying continuously from 1 to 5000 mm/min,
- Rotational speed : varying continuously from 25 to 4200 rpm.

The rotational speed and advancing speed are continuously measured and accurately controlled by the CNC controller of the milling machine.

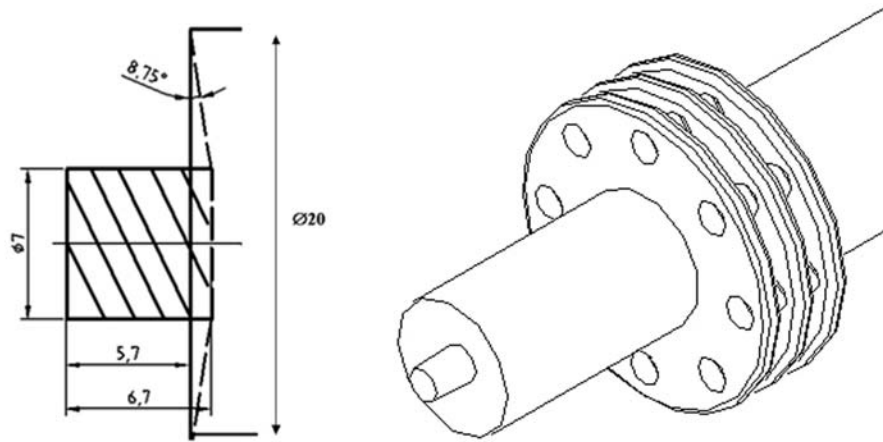


Figure 3.1. Geometry of the friction stir welding tool used for the high advancing speed welds.

Two friction stir welding tools have been mounted on the machine. The first having a 5.5 mm long inversely treaded pin is used for making hot welds (advancing speeds lower than 350 mm/min) and the other has a 5.7 mm long treaded pin. The choice of using two different tools is made to weld without root defects with a wide variety of welding parameters. The shoulders are both 20 mm in diameter and the pin is 7 mm in diameter. The tread is 1 mm deep and has a pitch of 1 mm. Blades are mounted on the tool body to avoid excess heating of the machine's head while welding (see Figure 3.1). The tool should preferably be in a wear resistant H13 hardened tool steel but for 6xxx series aluminum alloys, mild steel is sufficiently hard. The tool is tilted backward by 2° .

In order to rigidly clamp the workpiece, a backing bar is fixed on the table of the milling machine. This CK95 steel backing bar is 450 mm wide, 630 mm long and 80 mm thick. The two plates to be welded are both 70 mm wide, 590 mm long and 6 mm thick. In order to limit the vertical movement of the plates during welding, four spring clips have been fixed on the backing bar on each side of the workpiece. Heat transfer in the spring clips was limited by placing PVC sheets between the extremity of the spring clips and the workpiece. The movement of the workpiece in the advancing direction is blocked by two steel pins 10 mm in diameter that are positioned at the end of the backing bar. The movement in the y direction is blocked by four of these pins located on each side of the weld.

3.1.3 Measurements during welding

The forces in the three directions of space (F_x , F_y and F_z) as well as the torque M_z (along the tool axis) were measured using a rotating Kistler 9124A dynamometer adapted to the welding head located just above the friction stir welding tool. The dynamometer was calibrated using a tensile machine and an adapted traction system. Measurements during welding of welds performed at the beginning of the research have also been compared with a fixed dynamometer results. No major differences were measured. The forces F_x and F_y are measured in rotating coordinates with a zero fixed by the zero-count output. This zero count output allows measuring also the tool's rotating speed. The rotating speed given by the CNC controller was identical and no variation was observed during welding, therefore no further interest was given to this type of measurement.

Type K Thermo-Electric MTS-56050 thermocouples (0.5 mm in diameter) were embedded into the workpiece on the advancing and the retreating sides and within the backing plate by drilling holes of 0.6 mm in diameter and introducing a conducting pasta in the hole. The locations of the thermocouples are provided in Table 3.1 (see also Figure 3.2). Thermocouples 7 to 10 were located under the tool shoulder by drilling holes in diagonal. Two additional thermocouples were also inserted into the tool axis, respectively 3 and 13 mm above its shoulder. Figure 3.3 shows a picture of the welding device.

3.2 Microstructural characterization methods

3.2.1 Optical and electron microscopy

Optical and scanning electron microscopy. After having classically polished up to $1/4 \mu\text{m}$ the sections to be observed, they were observed using a high resolution camera and optical and scanning electron microscopy (SEM). The macrosection, the grain size and the shape of the welds were observed on transverse sections and the coarse phases were observed at mid-thickness in the plate section (see Figure 3.4). Prior to camera and optical microscopy observations of respectively the weld shape and the grain size, the sections were chemically etched in a Keller solution [94] for 2 to 5 minutes until the best contrast was reached. The best contrast in scanning electron microscopy for the observation of the iron rich phases was obtained using the backscattered electrons. The base material was observed on the same section for comparison.

Thermo- couple number	Distance from start (center of tool) [mm]	Distance to the weld centerline [mm]	Depth below the surface [mm]	Side
1	430	20	3	A
2	430	16	3	A
3	440	14	3	A
4	430	12	5	A
5	440	12	3	A
6	440	12	3	R
7	420	9.2	2.8	A
8	420	9.2	2.8	R
9	410	7.2	3.3	A
10	410	7.2	3.3	R
11	420	0	7	B

Table 3.1. Position of the workpiece and backing plate thermocouples (A = advancing side, R = retreating side, B = within the backing plate)

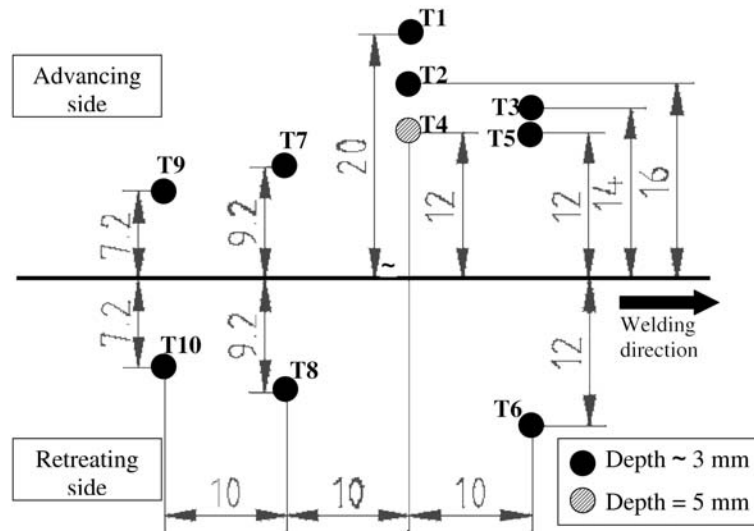


Figure 3.2. Schematic showing the position of the thermocouples in the workpiece.

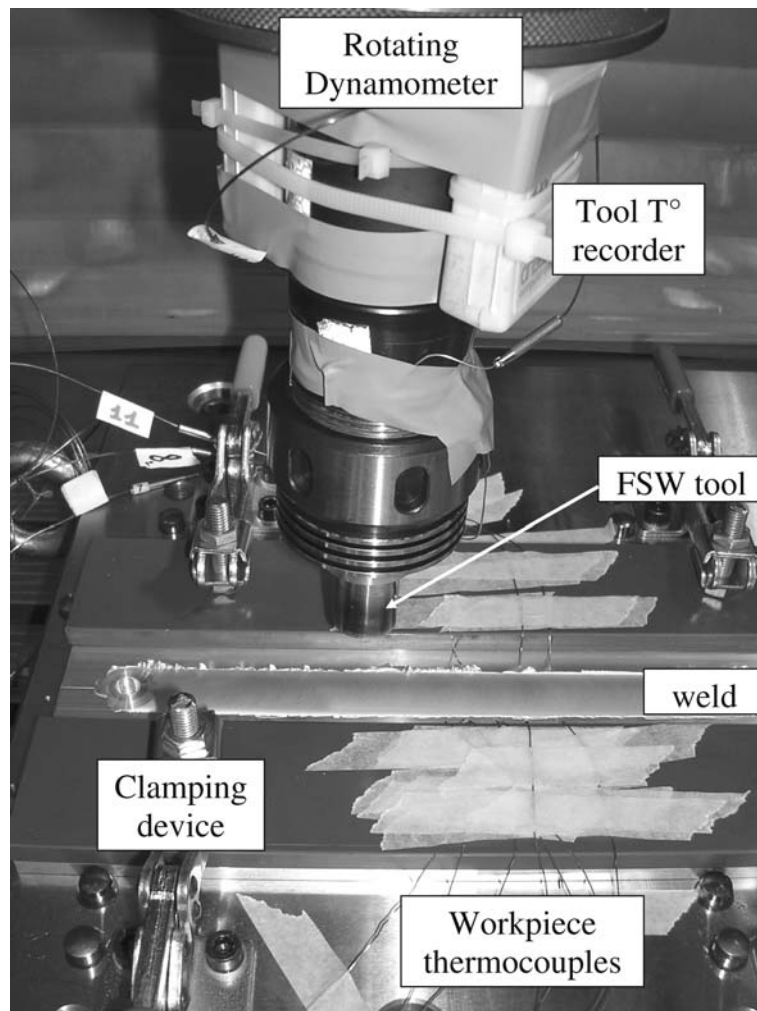


Figure 3.3. Friction stir welding device

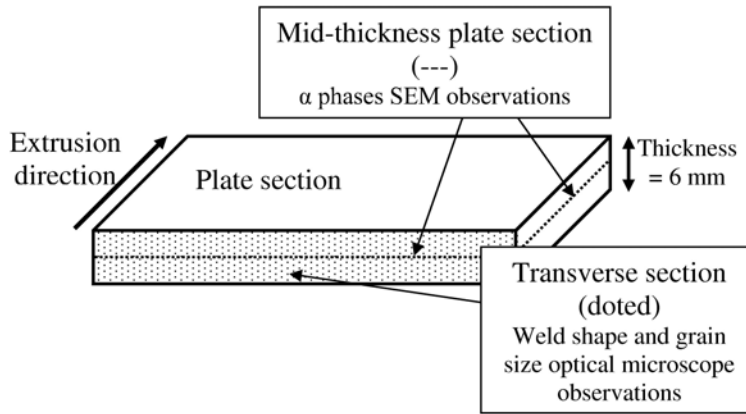


Figure 3.4.

Transmission electron microscopy. The dispersoids and the fine precipitates were observed in a Leo 922 transmission electron microscope (TEM) under 200 keV and the EFTEM (Energy-Filtered Transmission Electron Microscopy) mode. Samples were prepared from a 3 mm diameter rod extracted transversely to the welding direction of the welds (along the y direction) or extracted from the base material. These rods were sliced into 300 μm thick samples. The thickness was then reduced to 100 μm by manual polishing. The final thickness reduction was obtained by electro-polishing with a HNO_3 solution (HNO_3 30% in volume in methanol at -30°C under 9 V). The observation of the β'' and β' precipitates was always done under the 001 zone axis.

A complete analysis of the base material precipitation was overtaken. The thickness of the samples was estimated using the EELS method (Electron Energy Loss Spectroscopy). Myhr *et al.* [101] announce an accuracy of the thickness prediction by this method of $\pm 9\%$. Two friction stir welds were analyzed by TEM looking only at the β'' and β' precipitates.

3.2.2 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) measures the liberation or absorption of energy in a sample due to a thermal cycle. Figure 3.5 shows the principle of a DSC apparatus. The temperature of the heating chamber follows a given thermal cycle and the temperature of the samples are measured simultaneously. The first sample is a reference and the

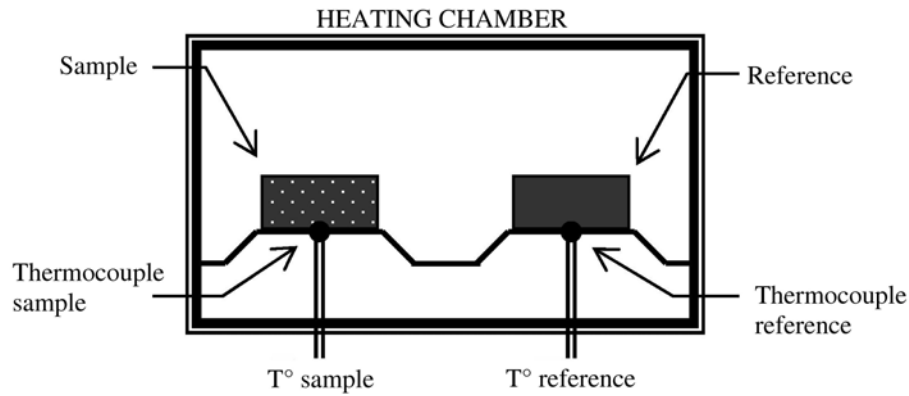


Figure 3.5. Principle of Differential Scanning Calorimetry (from [50])

second one is the sample of interest. Calibration of the apparatus allows determining the heat flux from the measured temperature difference. The heat flux is representative of the phase transformations which do not occur in the reference and which do occur in the investigated sample.

DSC measurements have been performed on a TA instrument MDSC 2920 apparatus at the ONERA, Chatillon, France. The samples have been prepared similarly to the TEM samples but their diameter was of 5 mm. The slices of the rods were 1.5 mm in thickness and were reduced by manual polishing to a mass of 75 mg. The DSC reference is pure aluminum in order to capture the effect of the alloying elements present in the studied sample. The heating rate was constant and equal to 20 °C/min. Since a slight increase in the heat flux is observed when using a pure aluminum as sample, it is classical to subtract this base line to the results obtained for alloyed samples [50]. Different samples have been studied:

- two base material samples;
- two solution heat treated samples measured respectively after 3 weeks of natural aging and after 1.5 years of natural aging;
- samples extracted along the section of a weld with parameters 1000 mm/min and 1000 rpm (measured after 1 year of natural aging after welding);

Phase transformation	Temperature	Base references
GP zones precipitation	160°C	[9, 144, 148, 150]
GP zones dissolution	200 – 220°C	[9, 57, 90, 144]
β'' precipitation	260°C	[9, 35, 56, 58, 148, 150]
β'' dissolution (if initially T6)	265°C	[90, 144] announce about 240°C
β' and B' precipitation	290 – 310°C	[36, 56, 58, 90, 56], [150, 9, 34, 35]
β' and B' dissolution	380 – 420°C	/
Si 1 st precipitation peak	300 – 320°C	[57, 58, 90, 150]
Si 2 nd precipitation peak	450°C	[57]
Si dissolution	520°C	[90]
Mg ₂ Si precipitation	460 – 470°C	[34, 36, 56, 57, 58]
Mg ₂ Si dissolution	500 – 510°C	[34, 58, 90]

Table 3.2. Temperatures of DSC peaks corresponding to different phase transformations

The identification and interpretation of the DSC peaks and valleys is based on literature and is summarized in Table 3.2¹. An exothermic peak ($Q > 0$) corresponds to a precipitation and an endothermic peak ($Q < 0$) corresponds to a dissolution. The peak area corresponds to the extent of the precipitation [56]. The positions of the peaks shift to higher temperatures if the precipitates are coarser. The presence of excess silicon can have an influence on the temperatures of the peaks [57]. The peaks of aluminum alloys of the 6xxx series are highly convoluted which can affect the identification of the peaks [50, 57].

3.3 Mechanical tests

3.3.1 Global mechanical properties characterization

Tensile tests on the base material (aluminum alloy 6005A-T6) and on heat treated specimens were performed on a ZWICK 50kN machine. The samples were 6 mm thick, 12.5 mm wide and had 45 mm of measurement basis for extensometry. Three tests were performed on the base material samples both in the extrusion and transverse long directions in order to quantify the scatter and anisotropy of the mechanical properties. Indeed,

¹The references are given only to confirm that the identified values of the temperatures of transformation are in a reasonable range since their values are very sensitive to the test conditions.

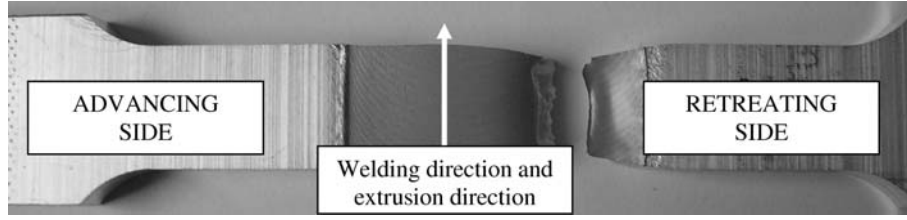


Figure 3.6. Global transverse tensile test on a weld

the alloy 6005A used in our study was extruded as 150 mm wide and 6 mm thick plates which induces significant anisotropy in the mechanical properties.

Global tensile tests on the welds were performed in the transverse long direction since the welding direction was the extrusion direction. The geometry of the weld tensile specimens and base material specimens were similar (see Figure 3.6). The yield strength of a weld is difficult to define since the sample is heterogeneous and plasticity starts in a very confined region. Therefore, the yield strength of the welds was conventionally defined at 0.1% of offset to linearity.

The reduced section S_r of these two types of macro-samples after fracture were obtained using a profile projector. S_r is then defined as:

$$S_r = W_r * T_r \quad (3.1)$$

where W_r and T_r are respectively the reduced width and thickness.

3.3.2 Local mechanical properties characterization

The 1 kg Vickers hardness of five welds was measured with a ZWICK 3202 machine along the transverse section at various through thickness positions before and after a post-weld heat treatment. To insure correct positioning of the hardness points, the position of a given hardness point was measured on an electronic profile projector. Finally, results were interpolated to determine the mid-thickness hardness evolution.

Digital Image Correlation (DIC) was performed on the transverse section of the same five welds in order to obtain by image correlation the logarithmic in-plane strain maps during tensile testing. The transverse section was initially painted in white and then painted with small black

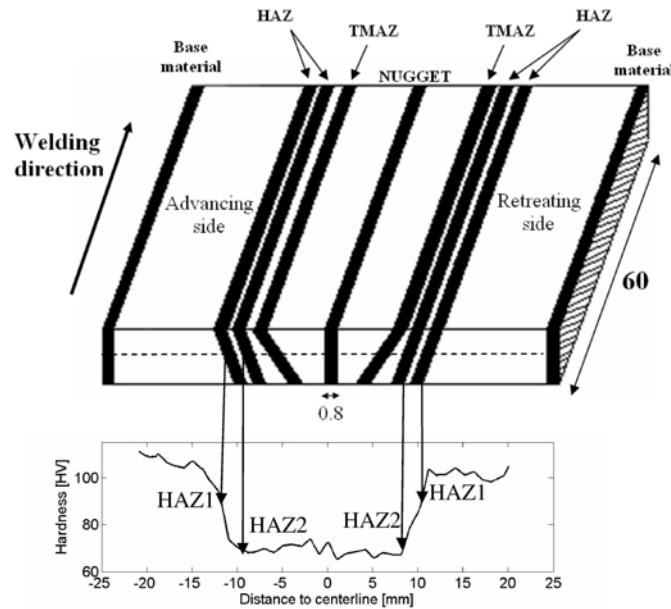


Figure 3.7. Extraction of the local tensile samples on a friction stir weld based on the hardness measurements

dots. Marks were also painted on the other side of the specimens in order to accurately determine the specimen fracture location.

Micro-tensile samples 0.8 mm thick were machined by electro-erosion parallel to the weld axis in each relevant zone of the five reference welds. Three samples were tested for each welding condition and region of the weld, giving a total of 135 samples. The shape of these zones being tilted for certain welding parameters (high advancing speeds, see hardness measurements in Figure 6.20(a,b)), some samples had to be machined at specific angles with respect to the axis normal to the plate (see Figure 3.7). Two samples were extracted from the HAZ. The first specimen, called HAZ1, presented an intermediate hardness (80 – 95 HV1) and the other, called HAZ2, had the lowest hardness (60 – 70 HV1) (Figure 3.7). The mid-thickness hardness of the tensile samples were also measured before the tensile tests in order to ensure correct positioning of the HAZ1 samples and to link the yield strength with the hardness. Macrographs of the welds allowed correct positioning of the TMAZ samples.

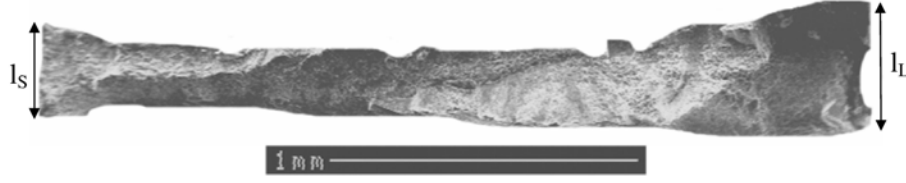


Figure 3.8. Rupture section of a micro-tensile sample

The fracture strain of the micro-tensile specimens was measured directly on the fracture surface in a scanning electron microscope. The reduced section S_r was measured by image analysis. A mechanical inhomogeneity index was defined as the ratio of the smallest edge thickness to the largest edge thickness l_S/l_L (see Figure 3.8).

3.3.3 Analysis of the tensile curves

In addition to the yield strength R_p , the ultimate tensile strength R_m and the true strain at the onset of necking ε_u , other mechanical properties are obtained through the tensile test.

- The true fracture strain ε_f is defined as:

$$\varepsilon_f = \ln\left(\frac{S_i}{S_r}\right) \quad (3.2)$$

where S_i and S_r are respectively the initial and reduced section of the tensile samples.

- The strain hardening exponent n is defined as:

$$n = \frac{\varepsilon}{\sigma_f} \frac{\partial \sigma_f}{\partial \varepsilon} \quad (3.3)$$

- The strain hardening exponent at necking n_u is defined as:

$$n_u = \frac{\varepsilon}{\sigma_f} \frac{\partial \sigma_f}{\partial \varepsilon} \Big|_{\varepsilon=\varepsilon_u} \quad (3.4)$$

- The dislocation storage rate θ and the recovery rate β are defined as:

$$\frac{d\sigma_f}{d\varepsilon_p} = \theta - \beta(\sigma_f - \sigma_y) \quad (3.5)$$

where σ_f is the flow stress, ε_p is the plastic strain and σ_y is the yield strength. θ and β are obtained by a linear fit on the variation

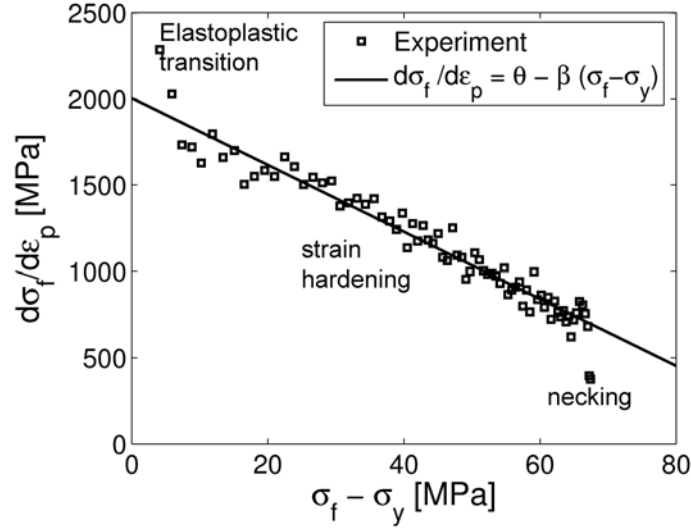


Figure 3.9. Strain hardening θ and β parameters identification from a tensile curve (tensile curve of a micro-tensile sample extracted from a friction stir weld heat affected zone). σ_f is the flow stress; σ_y is the true yield strength

of the strain hardening rate $d\sigma_f/d\varepsilon$ with the true flow stress in plasticity (σ_{pl}) (see Figure 3.9). The elastoplastic transition and the localization (necking) stages are excluded of the linear fit. The physical basis of these parameters will be presented in chapter 8.

3.4 Base material properties

3.4.1 Chemical composition of the alloy

The main alloying elements of the aluminum alloy 6005A-T6 are magnesium and silicon. Two different 6005A alloys have been investigated in this study, 6005A alloy number 1 and 6005A alloy number 2. These alloys are furnished in 6 mm thick extruded plates. The composition of the alloys have been determined by a chemical analysis and are summarized in Table 3.3. The main difference between these two alloys is the copper content. Alloy 1 contains more copper. If the opposite is not explicitly mentioned, the alloy used for experiments and modeling is the alloy 6005A number 1.

Alloy	Al	Si	Mg	Fe	Mn	Cu	Cr	Ti	Zn
$n^\circ 1$	98.18	0.81	0.48	0.24	0.11	0.09	<0.01	0.02	0.07
$n^\circ 2$	98.23	0.87	0.47	0.22	0.11	0.02	0.03	0.03	0.02

Table 3.3. Composition of the two 6005A aluminum alloys [wt%]

Direction of loading	Extrusion	Transverse long
Yield strength at 0.2%, R_p [MPa]	265.5 ± 2.2	255.5 ± 0.7
Tensile strength, R_m [MPa]	287.8 ± 1.2	276.8 ± 0.7
True strain at onset of necking, ε_u	0.086 ± 0.001	0.078 ± 0.002
Strain hardening exponent, n_u	0.085 ± 0.002	0.081 ± 0.003
True fracture strain, ε_f	0.20 ± 0.04	0.18 ± 0.01
Dislocation storage rate, θ [MPa]	918 ± 29	806 ± 4
Recovery rate, β	11.8 ± 0.4	10.1 ± 0.3

Table 3.4. Tensile properties of the base material 6005A-T6 in the extrusion direction and transverse long direction

3.4.2 Mechanical properties

The tensile properties of base alloy number 1 in the extrusion direction and transverse long direction are given in Table 3.4. The yield strength of base alloy number 2 is of 261 MPa (value given by the alloy manufacturer, SAPA RC Profile).

The hardness of the base alloy number 1 measured on a transverse surface is equal to 105 HV1 and does not vary through the thickness (a slight decrease of hardness is sometimes observed at mid-thickness). The hardness is only slightly anisotropic. A small HV1 difference is measured between the upper or the side surfaces but this average difference falls within the measurement error bar. The base alloy number 2 is a bit softer with a hardness equal to 95 HV1.

Part II

Thermal cycles during welding

ABSTRACT

Predicting the thermal history during the friction stir welding process is an important step in the understanding of the behavior of friction stir welds. Indeed, the thermal cycles are at the origin of the microstructural evolution in the different zones of a weld. Studying the effect of welding parameters on the thermal cycles will also help revealing the cause of the difference in behavior between hot welds (low advancing speed and high rotational speed) and cold welds (high advancing speed and low rotational speed). In addition to the prediction of the thermal cycles during the process, the thermal model developed in chapter 5 provides insights about the contact conditions both at the tool / workpiece interface and at the workpiece/backing plate interface that influence the thermal cycles measured during welding (chapter 4).

The measurement of the torque during welding gives the total power needed to execute a weld. The power input is more affected by the advancing speed than by the rotational speed and increases linearly with the advancing speed. The measurement of the tool temperature at two different distances from the tool shoulder gives an estimate of the heat loss into the tool. Hence the power that is really introduced in the plates to be welded can be inferred from those measurements.

Chapter 5 presents a simple finite element model predicting the thermal cycles during the welding process. This pseudo steady-state model includes the effect of convection of material from the retreating side to the advancing side of the weld by introducing a simple distribution of the circumferential velocity.

The power input is the sum of all the heat sources in the thermal model. The thermal model distributes this power input between surface and volume heat sources. The surface heat sources are attributed to frictional effect while the volume heat sources are attributed to the heat generated by plastic deformation due to material stirring. The comparison of the measured and predicted thermal cycles brought the conclusion that heat is generated by material deformation in a very thin layer surrounding the tool. Hence, if material convection is introduced

in the model the heat generation can be simply modeled by surface heat sources even though heat is generated by material deformation.

The thermal model shows that the contact between the workpieces and the backing plate contributes substantially to cooling down the workpiece during welding. The contact is not perfect everywhere at the interface. Comparing the predicted and measured thermal cycles showed that the contact is the best where a visible indentation is observed at the bottom of the welded plates, i.e. where the tool has squeezed the workpiece against the backing plate.

The temperature measurements presented in chapter 4 show that the advancing side of the weld is generally hotter than the retreating side of the weld. The thermal model has allowed understanding this effect by the introduction of material convection. Hence, it is the movement of hot material from the retreating side of the weld to the advancing side of the weld that is at the origin of the asymmetry of friction stir welds much more than the difference in friction between the sides of the weld which would cause a hotter retreating side. This is coherent with the identification of a more important effect of the "stirring" than of the "friction" in the friction stir welding process. The thermal model also reproduces correctly the lower level of asymmetry for low advancing speed welds.

Chapter 4

The mechanical and thermal welding conditions

This chapter presents the results of the measurements done during welding and the observed weld shape. These measurements include the forces and the torque as well as the thermocouple measurements. The important features discussed in this chapter are the evolution of the friction coefficient with the welding parameters, the relevance of the weld pitch as the key welding parameter, the determination of the welding efficiency and the asymmetry in temperature distribution. The results of this chapter will be the basis and the validation material for the thermal modeling presented in chapter 5.

4.1 Results

4.1.1 Welding conditions

Table 4.1 gives a summary of the welding conditions of the welds extensively studied in this work. The plunging speed was of 20 mm/min and the plunge depth was such that the "theoretical"¹ penetration of the shoulder in the material was 0.25 mm. The longitudinal movement of the tool was started as soon as the total plunge depth was attained.

4.1.2 Power and forces during welding

Figure 4.1 shows the evolution with time of the torque and the forces in the three directions of space. A first stage corresponds to the plunge

¹The deformation of the machine causes the true penetration to be slightly less than the theoretical one.

Experiment number	1	2	3	4	5
Advancing speed [mm/min]	200	200	200	765	1000
Rotational speed [rpm]	500	750	1000	1000	1000

Table 4.1. Welding conditions for the welds extensively studied.

of the pin in the material ($0 \text{ s} < \text{time} < 17 \text{ s}$). The vertical force (F_z) increases rapidly in that stage but the maximum value can be mitigated if the plunging speed is decreased. The torque is relatively constant during that stage and the in-plane forces (F_x and F_y) are negligible. When the shoulder touches the workpiece surface, a small peak force is sometimes observed if the plunging speed is too high. This peak must be limited as much as possible since it is the maximum force the machine has to hold up. The choice of a slow plunging speed of 20 mm/min eliminates this peak. The second stage is reached when the tool starts advancing ($17 \text{ s} < \text{time} < 51 \text{ s}$). A plateau is very rapidly obtained. The forces and torque values are much higher than the ones in the first stage if the plunging speed is not too high. These steady values of the forces and torque will be the only one discussed later on. The final stage consists in the extraction of the tool from the welded plates ($\text{time} > 51 \text{ s}$). This slightly affects the plate forces (F_x and F_y) due to the pin tread.

The scatter was determined by repeating the same weld many times. By repeating experiment number "4" four times, the maximum deviation from the mean of the measured torque M_z in the stationary plateau was found to be equal to 0.4 Nm. Measurements of the force in the welding direction F_x and the transverse force F_y never varied by more than 5 kg. The measurement of the vertical force F_z varied more, up to 52 kg. This higher value is due to the fact that the friction stir welding equipment used is displacement-controlled and not force-controlled. The last experiment (number "5") was also repeated three times and similar maximum deviations were observed.

The total power input is inferred from the torque measurements (P is equal to $M_z * \omega$, where P is the total power [W], M_z is the torque [Nm] and ω is the rotational speed [rad/s]). The experimental data show that the contribution of the force F_x in the welding direction to the total power is negligible.

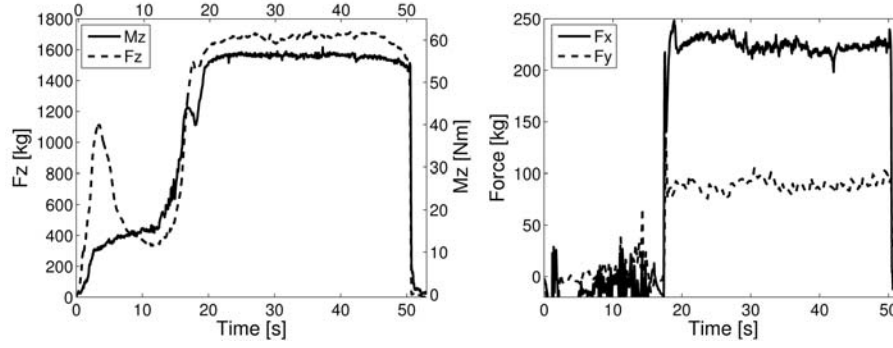


Figure 4.1. Forces and torque evolution with time for weld number "5" (1000 mm/min and 1000 rpm). The initial time (0 s) corresponds to the moment the tool pin gets in contact with the plate surface. The tool starts to move forward after about 17 s.

Figure 4.2(a) shows the variation of the total mechanical power with the welding parameters. The total power input is influenced more by the advancing speed than by the rotational speed. A higher rotational speed corresponds to a slightly higher total power. In the range of process parameters tested in the present study, the variation of P with respect to the advancing speed is almost linear. Equation (4.1) gives a good estimation of the total mechanical power.

$$P[kW] = 2.66 + 0.00322 * v[mm/min] \quad (4.1)$$

where v is the advancing speed [mm/min].

Figure 4.2(b) shows the variation of the vertical force with the welding parameters. The vertical force increases with increasing welding speed. An increase of the rotational speed tends to increase the vertical force (F_z) in analogy with the total mechanical power.

Figure 4.2(c) and (d) give the evolution of the in-plane forces (F_x and F_y) for different welding parameters. These forces globally increase with increasing welding speed. The effect of the rotational speed is not clear in all cases but the global tendency is to a decrease of the in-plane forces with increasing rotational speed.

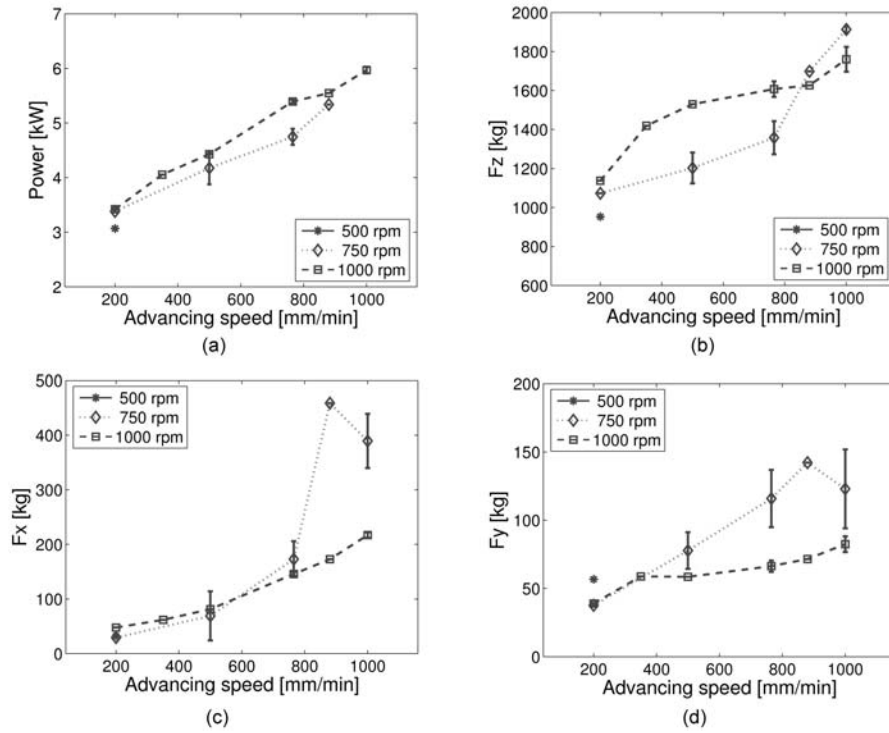


Figure 4.2. Forces and torque in stationary state for different welding parameters (a) Total mechanical power (b) Vertical force (F_z) (c) Force in the welding direction (F_x) (d) Transverse force (F_y). Error bars show the experimental scatter when more than one weld was executed for a given welding parameter.

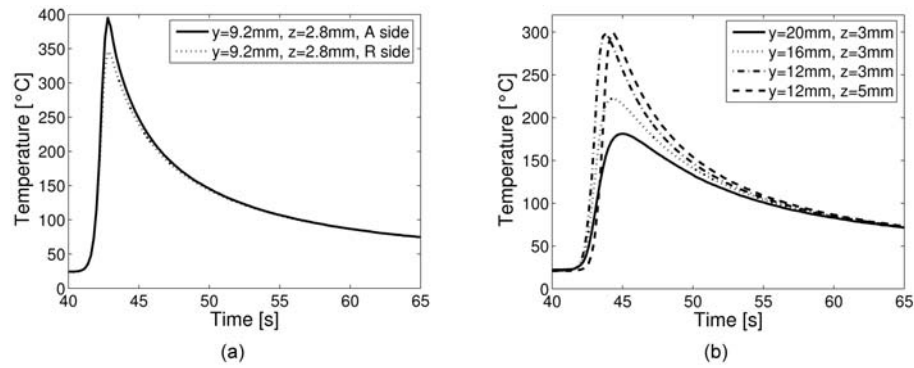


Figure 4.3. Temperature evolution with time for weld number "5" (1000 mm/min and 1000 rpm): (a) Temperature cycles on advancing and retreating side of the weld (b) Temperature cycles at different distances from the centerline and at a different depth.

4.1.3 Temperatures measured in the aluminum plate and in the tool

The temperature cycles measured by different thermocouples for weld number "5" are given in Figure 4.3. Temperature decreases rapidly when moving away from the centerline. The temperature measured at a 5 mm depth is similar to the one measured at a 3 mm depth. Hence, the effect of depth is not marked at 12 mm from the centerline.

In order to show the effect of the advancing speed on the heating and cooling rates, thermocouples giving similar maximum temperatures (about 350°C) have been selected. Indeed, the weakest zone of a weld has always a maximum temperature of about 350°C. Figure 4.4(a) shows that the heating and cooling rates increase significantly when the advancing speed increases. In the range of welding parameters used for this study, the rotational speed has no perceptible effect on the heating and cooling rate. The heating and cooling rates are key parameters when looking at the microstructural evolution due to a thermal cycle.

A study of the scatter in the temperature measurements has been undertaken because imprecise thermocouple positioning can always affect the quality of the temperature measurements since steep thermal gradients are present in the near tool region. By repeating experiment number "4" four times, the maximum deviation from the mean of the measured maximum temperature was found to be equal to 7°C. A similar level of

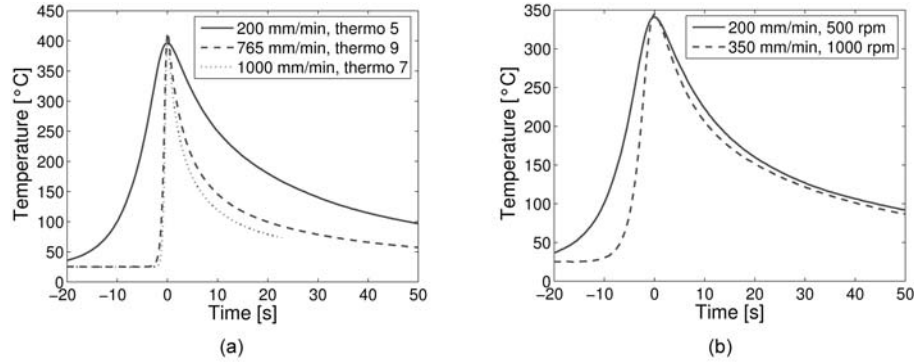


Figure 4.4. Heating and cooling rates for different welding parameters. (a) Effect of the advancing speed (for a rotational speed of 1000 rpm). Different thermocouples on the advancing side at a 3 mm depth are compared in order to get the same maximum temperature. Thermocouples 5, 9 and 7 are respectively at 12 mm, 9.2 mm and 7.2 mm from the centerline. (b) Assessment of the relevance of the weld pitch (advancing speed/rotational speed) as parameter for the heating and cooling rates for thermocouple 3. Time = 0 s corresponds to the maximum temperature.

deviation also applies to the temperatures measured in the tool except in one case (maybe the thermocouple moved out of the hole in that case). Dickerson *et al.* [17] found a standard deviation on their temperature measurements of about 10°C.

Figure 4.5 shows the effect of the welding parameters on the maximum temperature. In all cases, temperature increases when the advancing speed decreases and when the rotational speed increases whether it concerns positions close to the centerline (Figure 4.5(a)), away from the centerline (Figure 4.5(b)), the tool temperature (Figure 4.5(c)) or in the backing plate (Figure 4.5(d)).

4.1.4 Effects of the welding parameters on the weld shape

Figure 4.6 shows the effect of the welding parameters on the weld shape. The thermo-mechanically affected zone (TMAZ) starts on top of the weld at the tip of the shoulder and has a width at the bottom of the plate of about 7 mm for all the welding parameters. This width corresponds to the pin diameter. Other aspects of the weld shape are influenced by the welding parameters:

- for low advancing speeds and high rotational speeds, the excess flash increases.

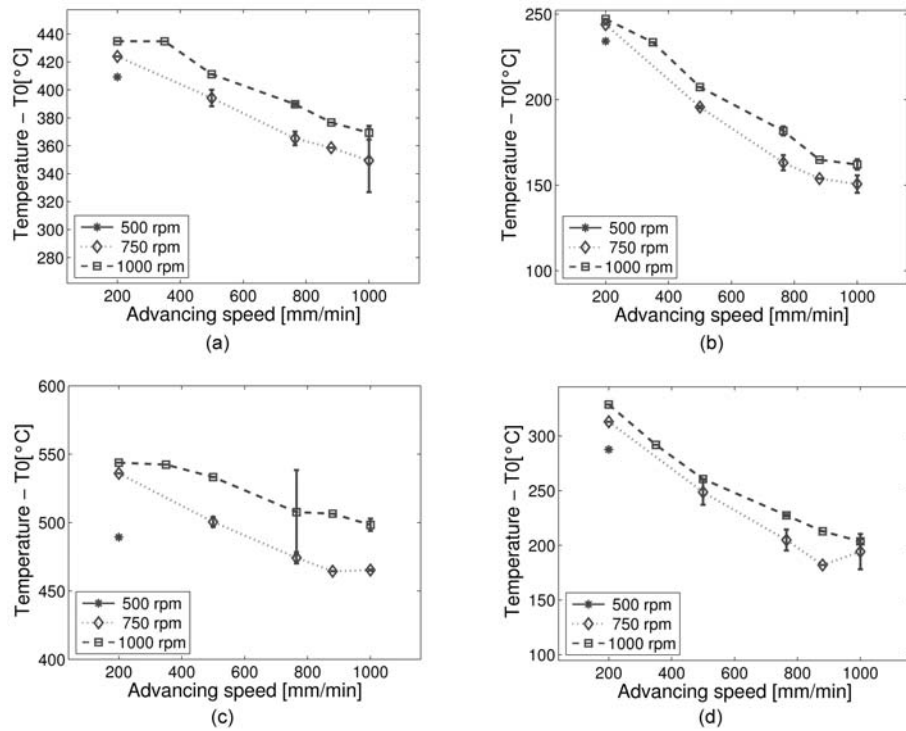


Figure 4.5. Maximum temperature measured by thermocouples for different welding parameters: (a) Thermocouple 7 (9.2 mm from the centerline) (b) Thermocouple 1 (20 mm from the centerline) (c) Thermocouple in the tool 3 mm above the shoulder (d) Thermocouple 11 (in the backing plate). Error bars show the experimental scatter when more than one weld was instrumented for a given welding parameter. T_0 is the initial plate temperature.

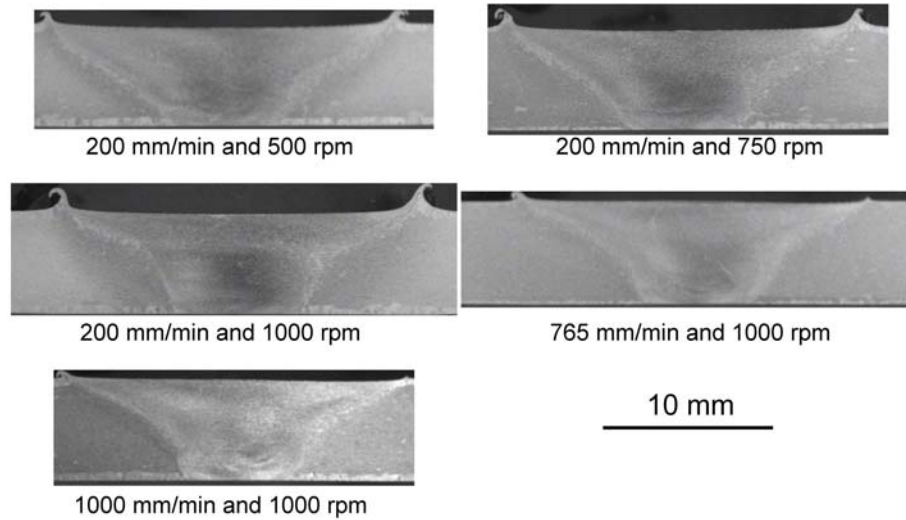


Figure 4.6. Weld shape for different welding conditions (the advancing side of the welds are on the left hand side)

- the shape of the TMAZ does not seem to significantly depend on the advancing speed but rather on the rotational speed. The TMAZ sticks more closely to the tool for high rotational speeds. Lower rotational speeds tend to increase the TMAZ mid-thickness width leading to a trapezoidal shape.

There are no clear evidences of asymmetries between sides of the welds when looking at the TMAZ shapes.

4.2 Discussion

4.2.1 Evolution of the total mechanical power input with the welding conditions

The literature is not clear about the dependence of the total mechanical power input on the advancing and rotational speeds. Tang *et al.* [133] interpreted temperature measurements on 6061-T6 aluminum alloy welds (60 mm/min advancing speed) by concluding that with a higher rotational speed, temperature should increase but that increasing temperature reduces the material flow stress which in turn limits the power generation and temperature increase. Colegrove *et al.* [17] concluded from temperature measurements on 16 mm thick welds of 7075 aluminum alloy that the maximum temperature was close to the solidus temperature

and therefore that heat generation was limited, which gives a weld power independent of the rotational speed. They also suggested that the power input is proportional to the square root of the advancing speed. On the other hand, Record *et al.* [109] concluded from a statistical study on 7075 aluminum alloy welds that the power input is influenced by both the rotational and the advancing speed.

From the literature it can be concluded that the material being welded and the process parameters influence the effect of the rotational speed on the power input since friction stir welding is a self regulating process. In hot welds (low advancing speeds and high rotational speeds), the power input seems to be relatively independent of the rotational speed. Experimental results from the present study concern rather hot welds which might explain the small influence of the rotational speed on the mechanical power input.

4.2.2 Evolution of the forces with the welding conditions

The increase of the in-plane forces with increasing advancing speed can be attributed to the fact that the material is colder and thus less soft when moving more rapidly. We have shown that increasing the advancing speed induces a decrease of the temperature measured away from the centerline. The effect of the rotational speed on the temperature is opposite to the evolution of the in-plane forces with the rotational speed. This lead us to conclude that the temperature is not the only variable affecting the in-plane forces but that the material stirring clearly plays an important role in the magnitude of these forces. Nevertheless, these forces are so small compared to the vertical force that they do not constitute limiting factors for machine design.

Colegrove *et al.* [19] showed for different tools that the force in the transverse direction F_y increases with increasing welding speeds. The welding parameters were found to have a minor effect on the vertical force F_z . They mentioned two effects to explain the evolution of the force in the welding direction F_x . Firstly, if the amount of stirring that the tool shape induces in its surrounding increases, the force in the welding direction F_x decreases. Secondly, if the amount of material rotating around the tool increases, F_x increases. Record *et al.* [109] concluded from their experimental statistical study on 7075-T7351 aluminum alloy welds that the force in the welding direction F_x and the vertical force F_z are mainly influenced by the advancing speed and that the force in the transverse direction F_y is mainly influenced by the rotational speed.

Ulysse [139] developed a model of the friction stir welding process. His parametric study of the influence of the welding speed on the forces lead to the conclusion that all the force components increase when welding at a higher welding speed or at a lower rotational speed. Chen *et al.* [14] model provided similar conclusion concerning the effect of the rotational speed but found only a minor effect of the rotational speed on the force F_y .

In conclusion, the effect of the welding conditions on the forces and torque is not clear. A more thorough study of the material flow would probably be useful for providing a more general and in-depth understanding of the effect of not only the welding parameters but also the tooling and the machine type on the forces.

4.2.3 Shape of the TMAZ

The shape of our TMAZ is mainly affected by the rotational speed. Colegrove *et al.* [17] drew opposite conclusions from their macrosection observations. In their experiments, the size of the TMAZ diminished if the welding speed increases but the rotational speed had no influence on the TMAZ size.

4.2.4 Friction coefficient

It is rather classical to link the vertical force (F_z) to the total mechanical power (P) through a friction coefficient μ assuming that all the heat is generated by friction. The surface heat generated by the friction of the tool on the material can be simply estimated by equation (4.2) assuming constant pressure equal to $p_{shoulder}$ [Pa] [45]. Note that this expression neglects the pin lateral surface contribution.

$$Q_S = \frac{2}{3}\pi\mu p_{shoulder}\omega r_S^3 = \frac{2}{3}\mu F_z\omega r_S \quad (4.2)$$

where r_S is the radius of the tool shoulder. If we assume that the power is only generated by friction (hence $P = Q_S$) we can estimate the friction coefficient with the following expression:

$$\mu = \frac{P}{\frac{2}{3}F_z\omega r_S} \quad (4.3)$$

Figure 4.7 shows the evolution of the friction coefficient calculated by expression (4.3) for different welding parameters. The friction coefficient

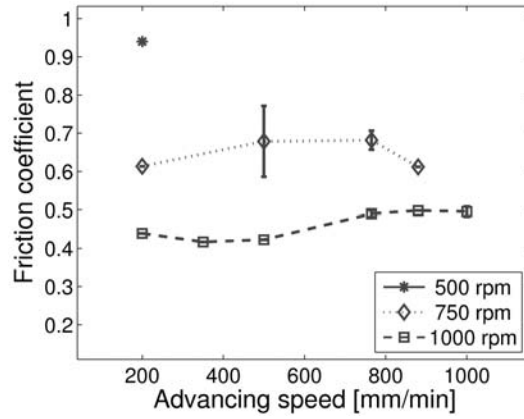


Figure 4.7. Evolution of the friction coefficient with the welding parameters

is clearly more influenced by the rotational speed than by the advancing speed since the vertical force F_z and the power P depend more on the advancing speed than on the rotational speed and that they both increase when the advancing speed increases. In the thermal modeling literature, we find all kinds of values for the friction coefficient. Some are realistic like the one proposed by Frigaard [46] ($\mu = 0.4$ for welding parameters of 300 mm/min and 1500 rpm and a shoulder diameter of 20 mm). Oppositely, the value proposed by Zahedul *et al.* [149] sounds very low ($\mu = 0.15$ for welding parameters of 139.7 mm/min and 500 rpm and a shoulder diameter of 25.4 mm). Nevertheless, the hypothesis on which this calculation is based are certainly too simplistic. The influence of material deformation is clearly not negligible.

4.2.5 Relevance of the weld pitch as key welding parameter

The weld pitch is defined as the ratio of the advancing speed (in mm/min) to the rotational speed (in rpm). It is classical in the friction stir welding community to use the weld pitch to compare the effect of the friction stir welding parameters.

Figure 4.8(a) shows that the weld pitch is not the unique parameter describing the process since the total mechanical power depends more on the advancing speed than on the rotational speed as mentioned earlier. Nevertheless, when looking at the maximal temperatures measured by the thermocouples (see Figure 4.8(b)), it seems that the weld pitch is

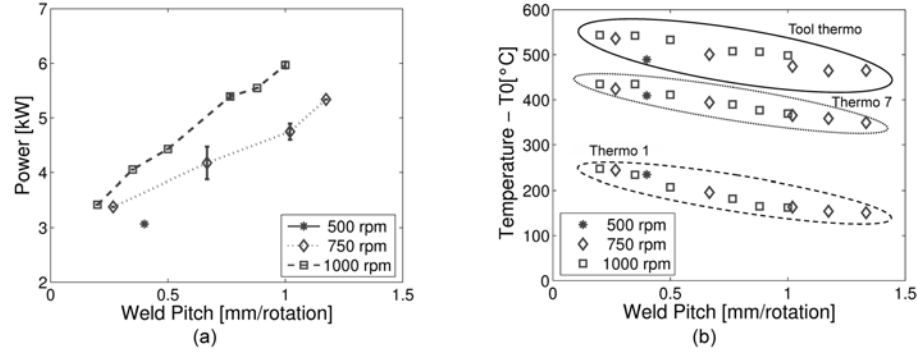


Figure 4.8. Effect of the weld pitch. (a) Total mechanical power evolution with the weld pitch (b) Maximum temperature evolution with the weld pitch for thermocouple 1 (20 mm from the centerline) and 7 (9.2 mm from the centerline) and the tool thermocouple embedded 3 mm from the shoulder.

a relevant parameter to rank monotonously the maximum temperatures for different welding conditions. Care should still be taken since giving the maximum temperature is not sufficient to describe a thermal cycle. The heating and the cooling rate are more dependent on the advancing speed than on the rotational speed and therefore the weld pitch does not describe correctly their evolution. Figure 4.4(b) shows the thermal cycles for a given thermocouple for two welds having similar weld pitches but different welding parameters. Even if the maximum temperatures coincide, the heating and cooling rates are different.

Reynolds *et al.* [120] have developed a 2-D computational fluid dynamics model to study the flow of material considering the heat input to be due to viscous dissipation. They concluded that the weld pitch is not the unique process parameter to consider. Temperature measurements by Fujii *et al.* [48] show that the maximum temperature is mainly dependent on the weld pitch.

4.2.6 Efficiency and heat loss in the tool

The measurements of temperature at two locations along the tool axis allow a simple evaluation of the heat losses into the tool. From the temperature gradient one can calculate the heat loss into the tool:

$$Q_{tool} = \frac{\lambda_{steel}(T_3 - T_{13})}{d} \quad (4.4)$$

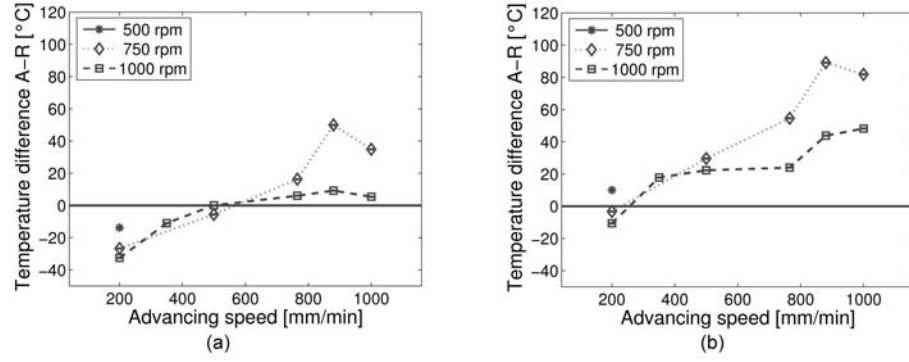


Figure 4.9. Maximum temperature difference between the advancing and the retreating side of the welds (a) 12 mm and (b) 9.2 mm from the centerline

where λ_{steel} is the thermal conductivity of the tool steel ($\lambda_{steel} = 47$ W/mK); T_3 and T_{13} are the maximum temperatures measured inside the tool at 3 mm and 13 mm from its shoulder, respectively, and d is the distance between these two thermocouples ($d = 10$ mm).

An estimation of the welding efficiency is obtained by subtracting the heat loss from the total power input:

$$\eta = \frac{P - Q_{tool}}{P}. \quad (4.5)$$

In the range of welding parameters investigated in this study, the efficiency does not seem to be dependent on the welding parameters and is approximately equal to 95%.

The value of the heat loss into the tool has also been studied in the literature, leading to similar conclusions. Chao *et al.* [13] and Dickerson *et al.* [31], after modeling the temperature distribution in the tool and comparing it to experiments, conclude that the heat loss is about 5%. Dickerson *et al.* [31] give a value of 10% under steady-state conditions. Many other authors use process efficiencies of about 90% in their model of the process (Shi *et al.* [124], Schmidt *et al.* [119]).

4.2.7 Asymmetry in the temperature distribution

As observed in Figure 4.3(a), the maximum temperature on the advancing side is higher than on the retreating side². Nevertheless this con-

²Note that the flexibility of the dynamometer causes the displacement of the weld center of about 0.3 mm to the retreating side for all the welds such that errors in

clusion, valid for most of the welding parameters of the present study, is not always verified. For low advancing speeds and high rotational speeds, the temperature can be slightly higher on the retreating side. Figure 4.9 shows the temperature difference between the advancing side and the retreating side of the weld for various welding parameters 12 mm and 9.2 mm from the centerline.

The scientific literature globally agrees about the fact that the advancing side of a friction stir weld is hotter [3, 82]. But, Maeda *et al.* [82] and Lawrjaniec *et al.* [74] concluded from their experimental results that a slightly hotter retreating side could be observed for some welding conditions. Tang *et al.* [133] found no significant difference in the temperature distribution on the two sides of the weld. Clearly, the welding conditions play an important role on the temperature difference between the advancing and the retreating side of the weld.

4.3 Conclusions

This chapter presented and made a first analysis of the results of the forces, torque and temperature measurements during welding. The following conclusions have been drawn:

- The total mechanical power and the forces increase with increasing advancing speed. The effect of the rotational speed is not so clear but the tendency goes for an increase of the in-plane forces for decreasing rotational speeds and an increase of the vertical force and the total mechanical power for increasing rotational speeds. The effect of the rotational speed on the total mechanical power is clearly of the second order compared to the effect of the advancing speed on the total mechanical power.
- The cooling and heating rate are increasing for increasing advancing speeds. No marked effect of the rotational speed has been identified.
- The maximum temperatures measured by the thermocouples decrease with increasing advancing speeds and increase with increasing rotational speeds.
- The advancing side is clearly hotter for cold welds (high advancing speeds) but for hot welds (low advancing speeds) the retreating side could be hotter.

positioning do not explain this temperature difference between sides.

- The weld shape is more clearly affected by the rotational speed than by the advancing speed.
- The friction coefficient is of the order of 0.4 to 1 for the welding conditions studied in this thesis and is mainly affected by the rotational speed.
- The weld pitch is a relevant parameter when looking at the maximum temperature but not when looking at the total mechanical power evolution nor when looking at the heating and cooling rates.
- The efficiency of all friction stir welds studied in this thesis was found to be of 95%.

Chapter 5

Thermal modeling

The thermal model presented in this chapter and the experimental results used for the identification and validation are the compilation and the improvement of work published by the author *et al.* [125, 126, 127]. These experimental results have been presented in the previous chapter.

5.1 Background

In the friction stir welding community it is generally accepted that heat can be generated by frictional effect and/or by the plastic deformation associated with material stirring. The introduction in models of those two heat generations can be done by surface heat sources when modeling the frictional heat generation or by volume heat sources when modeling the effect of material deformation on the heat generation. A more complex way to treat the problem of heat generation is to model the material flow and deduce the heat generation. The problem is the need for laws of material behavior at the high temperatures reached during the process. Furthermore, the question whether the material slides or sticks at the tool/material interface is linked to the question of heat generation by friction or by plastic deformation. The backing plate has been shown to contribute to cool down the plates to be welded and hence is of importance when modeling the process.

No consensus has emerged yet about the proper partitioning of frictional and plastic deformation in friction stir welding. It is not even clear which is the dominant heat generation mechanism. By using a rather low value for the friction coefficient, Zahedul *et al.* [149] concluded that a purely frictional heating model is probably not adequate. Khandkar *et al.* [68] took the measured mechanical power as input to a friction stir

welding thermal model with frictional surface heat sources distributed based on the assumption of a uniform shear stress at the tool/material interfaces. Tang *et al.* [133] measured the temperatures in the near pin region; the isothermal plateau near the pin suggested that heat is generated mainly through plastic deformation during the friction stir welding process.

The problem of distributing the heat sources around the tool has also been addressed in the literature. Schmidt *et al.* [117] studied the influence of the tool shape on the relative amount of heat generated at the tool interfaces. Schmidt *et al.* [118] applied this proportion to the prediction of the thermal cycles in FSW. Their model includes material convection by the introduction of a simple model for the velocity field. Shi *et al.* [124] distributed a total power input of 1600 W (obtained from the measured torque) between a surface heat source at the shoulder interface (75% of the total power input) and a volume heat source inside the pin (25% of the total power input). Mc Clune *et al.* [87] proposed to reduce the contribution of the pin to 20% of the total power input in an application to thinner sheets. Tang *et al.* [133] temperature measurements with a pinless tool gave only a 4% reduction in the measured maximal temperature. They concluded that the pin has a minor influence on the heat input.

The fact that friction stir welding is a solid-state welding process is generally accepted and is sometimes artificially taken into account in models. Frigaard *et al.* [47] limited the maximal temperature of their model to a value close to the eutectic temperature of the alloys studied by adapting the coefficient of friction to prevent the maximal temperature being overpassed. Song *et al.* [128] set the heat input of their model to zero when the melting temperature was reached.

The loss of heat through the contact interface between the bottom of the workpiece and the backing plate has been introduced in numerical models following different approaches. Chao *et al.* [12] proposed a value of 200 W/m²K for the bottom convection coefficient by comparing the results of 3D finite element simulations to the experimental results by Mc Clure *et al.* [88] on 6061-T6 aluminum alloy. Foment *et al.* [43] proposed a similar value for the bottom convection coefficient. Zahedul *et al.* [149] compared four different bottom convection coefficients and concluded that a too high value of this coefficient (4000 W/m²K) leads to underestimating the maximum temperature. They also concludes that

adiabatic conditions ($0 \text{ W/m}^2\text{K}$) give maximum temperatures far above the melting point. An intermediate value of $1000 \text{ W/m}^2\text{K}$ was therefore chosen. Ulysse [139] did not include the backing plate assuming simple adiabatic conditions at the workpiece-backing plate interface.

Some authors introduced a backing plate in the model and simulated the contact conditions between the workpiece and the backing plate. Colegrove *et al.* [16] proposed a contact conductance of $1000 \text{ W/m}^2\text{K}$ between the workpiece and the backing plate. The bottom of the backing plate was set to a temperature equal to 300 K . In another paper, Colegrove *et al.* [20] kept the same contact conductance over the whole interface surface except under the tool region where a perfect contact was introduced. Khandkar *et al.* [68] introduced an annular distribution of the contact conductance which simulates the heat loss in the backing plate. The contact between the workpiece and the backing plate in front of the tool is probably not complete as a result of the backward inclination of the tool. Shi *et al.* [124] proposed a temperature-dependent contact conductivity between the workpiece and the backing plate. De Vuyst *et al.* [27] showed that such a variation of the contact conductance with temperature ensures a good correlation between experimental and modeling time-temperature curves for the friction stir welded 6005A-T6 and 2024-T3 aluminum alloys.

Schmidt *et al.* [118] proposed a simple distribution of circumferential material velocity to study the effect of material convection on the temperature distribution in the sticking and the sliding cases. More complex models include material flow. Bendzsak *et al.* [5], Shercliff *et al.* [123], Seidel *et al.* [120], Colegrove *et al.* [18, 19, 20] developed computational fluid dynamics models to study the material flow assuming the heat input to be due to viscous dissipation. Ulysse [139], Askari *et al.* [3], Smith *et al.* [119], Foment *et al.* [43] and P. Heurtier *et al.* [61] developed coupled thermomechanical models which provide excellent insight into the material flow during the process. Buffa *et al.* [8] developed an uncoupled thermomechanical model of the friction stir welding process. All these models give key insight into the material flow during the process.

Some models in the literature include residual stress predictions (e.g. Shi *et al.* [124], Chen *et al.* [14], De Vuyst *et al.* [27]). They are generally based on uncoupled thermal and mechanical models.

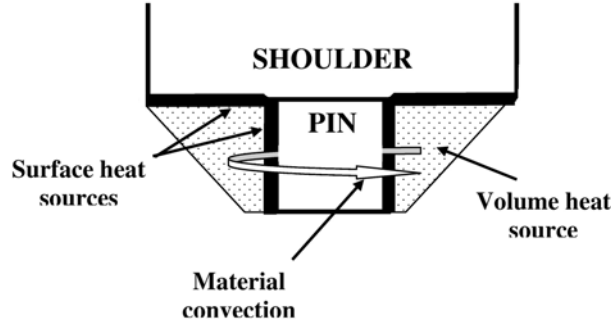


Figure 5.1. Principle of the thermal model.

5.2 Model description

5.2.1 General assumptions

A finite element model of the pseudo steady-state heat transfer during welding was developed using the general-purpose commercial code ABAQUS [1]. Figure 5.1 shows the different elements that the thermal model introduces including surface and volume heat sources as well as the effect of material convection. The underlying equation is:

$$\rho_a c_p \left(\frac{\partial T}{\partial t} + v_{mat} \cdot \nabla T \right) = \lambda \nabla \cdot \nabla T + q_{heat} \quad (5.1)$$

where ρ_a , c_p and λ are respectively the density, the specific heat and the thermal conductivity of the material, here, aluminum alloy 6005A. T is the temperature, t is the time and v_{mat} is the velocity of material. q_{heat} is a heat source. Far away from the tool region v_{mat} is equal to the advancing speed. In the near tool region, the effect of the movement of material around the tool on the value of v_{mat} is taken into account by imposing a chosen velocity distribution.

The finite element mesh of the thermal model is made up of hexahedrons with a high density in the near tool region (see Figure 5.2). The model is long enough not to influence the thermal field in the zone surrounding the tool.

A temperature of 25°C was imposed upstream. The top and side surfaces were considered as adiabatic. The effect of convection at these surfaces on the temperature distribution was shown to be negligible. The model takes into account the heat loss through the backing plate by

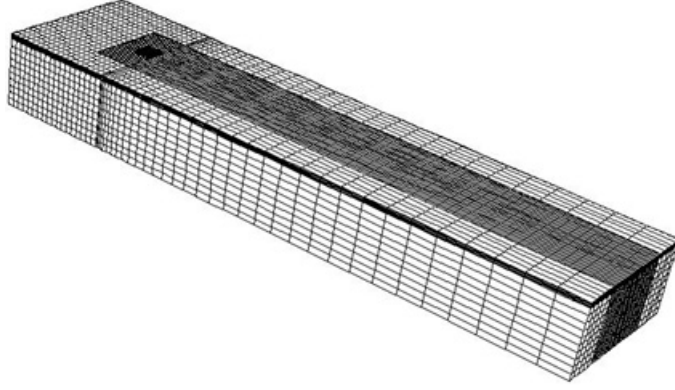


Figure 5.2. Finite element mesh for the thermal model

modeling a 80 mm thick steel plate under the workpiece. A temperature equal to 25°C was imposed at the bottom of this thick steel plate. The contact conditions between the workpiece and the thick steel plate will be detailed later.

In order to avoid discontinuities in the flow field of aluminum, the pin was ignored. Taking the steel pin into account would require to model the way the aluminum flows around the pin.

5.2.2 Heat sources

(a) Heat dissipated by friction at the tool / workpiece interface - surface heat sources

The heat generated by friction between sliding surfaces q_S [W/m²] is equal to the work done by the interfacial shear stress $\tau_{contact}$:

$$q_S = \tau_{contact}(v_{tool} - v_{mat})|_{interface}. \quad (5.2)$$

The tool velocity v_{tool} can be decomposed in its velocity ωr due to the rotation and the velocity of the linear movement. In most practical cases, the latter can be neglected. The material velocity v_{mat} at the tool / workpiece interface is unknown. For simplicity, the material velocity v_{mat} is introduced in the model as a fraction δ of the velocity ωr . The surface heat source due to friction at the tool / workpiece interface expresses as:

$$q_S = \tau_{contact}\omega r(1 - \delta) \quad (5.3)$$

with $0 \leq \delta \leq 1$. The value of δ characterizes the type of contact at the tool / workpiece interface:

- If $\delta = 0$, the contact is perfect *sliding*;
- If $\delta = 1$, the contact is perfect *sticking*.

The scientific literature does not provide a clear identification of the condition prevailing in FSW.

It is convenient to divide the total power dissipated at the tool / workpiece interface Q_S [W] in three parts corresponding to the different working surfaces of the tool : the shoulder (assumed here to be a plane annular disk with an external radius equal to the shoulder radius r_S and an inner radius equal to the radius of the tool pin r_p), the lateral surface of the pin (assumed here to be a cylinder with a radius equal to the tool pin radius r_p and a height equal to the height of the tool pin h_p) and the surface of the tool pin tip (assumed here to be a plane disk with a radius equal to the tool pin radius r_p).

The total power dissipated at the tool / workpiece interface thus writes:

$$Q_S = Q_{S,shoulder} + Q_{S,pin} + Q_{S,pin\ tip} \quad (5.4)$$

with

$$\begin{aligned} Q_{S,shoulder} &= \int_0^{2\pi} \int_{r_p}^{r_S} \tau_{contact} \omega r (1 - \delta) r d\theta dr, \\ Q_{S,pin} &= \int_0^{2\pi} \int_{r_p}^{r_S} \tau_{contact} \omega r (1 - \delta) r d\theta dz, \\ Q_{S,pin\ tip} &= \int_0^{2\pi} \int_{r_p}^{r_S} \tau_{contact} \omega r (1 - \delta) r d\theta dr. \end{aligned} \quad (5.5)$$

Assuming that $\tau_{contact}$ and δ are constant along all tool / workpiece interfaces lead to

$$\begin{aligned} Q_{S,shoulder} &= \tau_{contact} \omega (1 - \delta) \frac{2}{3} \pi (r_S^3 - r_p^3), \\ Q_{S,pin} &= \tau_{contact} \omega (1 - \delta) 2\pi r_p^2 h_p, \\ Q_{S,pin\ tip} &= \tau_{contact} \omega (1 - \delta) \frac{2}{3} \pi r_p^3. \end{aligned} \quad (5.6)$$

The total power dissipated at the tool / workpiece interfaces is therefore equal to

$$Q_S = \tau_{contact}\omega(1 - \delta)\frac{2}{3}\pi(r_S^3 + 3r_p^2h_p). \quad (5.7)$$

Using the numerical values of the parameters characterizing the tool geometry in the present study ($r_S = 10$ mm, $r_i = 3.5$ mm and $h_p = 5.7$ mm), one easily obtains the relative proportions of the friction heat dissipated on the shoulder, the pin and the pin tip of the tool:

$$\begin{aligned} Q_{S,shoulder}/Q_S &= 79.2\%, \\ Q_{S,pin}/Q_S &= 17.3\%, \\ Q_{S,pin\ tip}/Q_S &= 3.5\%. \end{aligned} \quad (5.8)$$

The part of the frictional heat power being associated with the pin tip being very small, it will be neglected in the following. We will therefore consider that

$$\begin{aligned} Q_S &= \tau_{contact}\omega(1 - \delta)\frac{2}{3}\pi((r_S^3 - r_p^3) + 3r_p^2h_p) \\ Q_{S,shoulder}/Q_S &= \frac{r_S^3 - r_p^3}{(r_S^3 - r_p^3) + 3r_p^2h_p} = 82\%, \\ Q_{S,pin}/Q_S &= \frac{3r_p^2h_p}{(r_S^3 - r_p^3) + 3r_p^2h_p} = 18\%. \end{aligned} \quad (5.9)$$

If we now distribute the surface heat sources corresponding to the heat power per unit of area dissipated by friction at the tool / workpiece interfaces, we obtain along the shoulder with a linear variation with the radius:

$$q_S = \frac{0.82Q_S r}{\frac{2}{3}\pi(r_S^3 - r_p^3)} \quad (5.10)$$

with $r_S \leq r \leq r_p$ and $z = 0$, and along the lateral surface of the tool pin;

$$q_S = \frac{0.18Q_S}{2\pi r_p h_p} \quad (5.11)$$

with $0 \leq z \leq h_p$ and $r = r_p$.

(b) Heat dissipated by material stirring (plastic deformation) - volume heat sources

Heat is also dissipated throughout the volume of the material by plastic deformation. The corresponding volume heat sources are equal to :

$$q_V = \zeta \dot{\epsilon}_{ij}^p \sigma_{ij} \quad (5.12)$$

where $\dot{\varepsilon}_{ij}^p$ and σ_{ij} are respectively the components of the plastic strain rate tensor and the Cauchy stress tensor and ζ is a parameter ranging typically between 0.8 and 0.99 [112] which accounts for the fact that a small part of the energy is stored in the material in the form of defects. Several research groups (Heurtier-Monteillet, Deepweld project, Schmidt *et al.* etc...) are currently developing models to numerically simulate the plastic flow and accompanying heat generation. The problem is extremely complex not only because the flow is complex but also because the constitutive relationships are complex. Indeed, the plastic response depends on rate and temperature dependent internal variables characterizing the hardening and softening behavior associated with the microstructural evolution (precipitate dissolution, recrystallization, recovery).

A purely phenomenological approach is used in the present study. Little can be predicted concerning the location and the intensity of the heat dissipation through plastic deformation during FSW, except that it occurs in the so-called thermo-mechanically affected zone (TMAZ) as is experimentally confirmed and explained in the literature. Dong *et al.* [32] concluded from their models that plastic slip at the surface due to mechanical interactions between the tool and the base material seems to correlate with the friction stir weld profile obtained by macrographs. The inverse model of Lambrakos *et al.* [71] allowed them to conclude that there is a strong correlation between the physical shape of the stirred region and the volume distribution of heat generated by the stirring process. The thermomechanical model of Askari *et al.* [3] shows that the deformed region is confined to the area immediately surrounding the pin and under the shoulder. The deformation of the material is very large close to the friction stir welding tool but, due to heating, the flow stress there is the lowest. Conversely, away from the tool, the deformation is lower but the material flow stress is higher because the temperatures are lower.

In the absence of any other relevant information, an uniform distribution of the volume heat sources q_V [W/m^3] resulting from the plastic deformation in the TMAZ will be assumed. The total heat power dissipated through plastic deformation is imposed to be

$$Q_V = q_V V_p \quad (5.13)$$

where V_p [m^3] is the volume of the TMAZ and Q_V [W] is the volume heat source power.

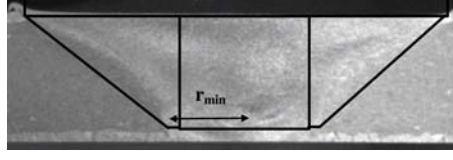


Figure 5.3. Shape of the TMAZ

The volume V_p of the TMAZ where plastic deformation takes place during FSW has been determined from macrographic observations on a transverse section as a trapezoid (see Figure 5.3). The volume of this trapezoid is equal to:

$$V_p = \frac{1}{3} \pi h_p (r_s^2 + r_{min}^2 + r_s r_{min} - 3r_p^2). \quad (5.14)$$

The advantage of choosing this very simple description of the shape of the TMAZ avoids the need to prepare a macrography of the weld in order to use the model. The value of r_{min} (introduced in Figure 5.3) was defined as $r_p + 0.5 \text{ mm} = 4 \text{ mm}$ and we will assume that it does not vary with the welding parameters even though we have observed that the weld shape is slightly affected by the rotational speed.

(c) Distribution of Mechanical power between volume and surface heat sources

The total power P_{in} dissipated by friction and plastic deformation during welding can be calculated from the measurements of the force and torque. Almost all that power is transformed into heat. As explained above, the energy stored in the material in the form of dislocations and other microstructural defects, such as new grain boundaries and dislocations for instance, is very small. The portion of the power lost within the tool was previously found to be 5% of the total mechanical power.

$$\eta P = P_{in} = Q_S + Q_V \quad (5.15)$$

The relative importance of Q_V and Q_S is unknown. In the present study, a parameter γ is introduced for that purpose:

$$\begin{aligned} Q_V &= \gamma P_{in}, \\ Q_S &= (1 - \gamma) P_{in}. \end{aligned} \quad (5.16)$$

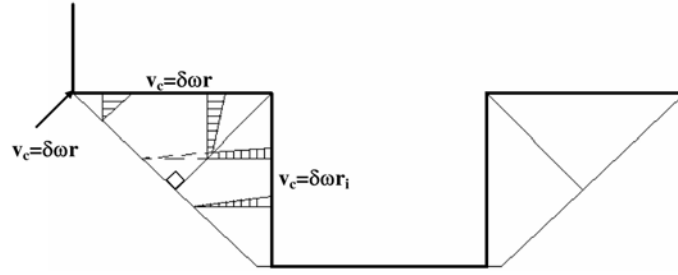


Figure 5.4. Simple model of the circumferential velocity field

5.2.3 Material convection around the friction stir welding tool

To study the effect of material convection around the tool, a very simple model for the velocity field around the tool is proposed. Material flow is considered only in the TMAZ. The local circumferential velocity due to the shoulder increases with increasing radius and decreases with increasing depth. The local circumferential velocity due to the pin decreases with increasing radius. Figure 5.4 represents the assumed velocity field. Note that the parameter δ was defined previously as the ratio of the material velocity on the tool velocity at the material/tool interface.

The problem with such a simple model for the circumferential velocity field is that it introduces velocity discontinuities at the outer radius of the shoulder.

5.2.4 Decoupling of parameters γ and δ

The work by Schmidt *et al.* [118] does not distinguish between parameters γ and δ , which is similar to assuming that the two parameters evolve in the same way. Hence, a sliding interface condition corresponds to surface heat sources and a sticking interface condition corresponds to volume heat sources. It could be more complex.

Let us discuss the four extreme cases:

- $\gamma = 0$ (surface heat source) and $\delta = 0$ (no material circumferential velocity): this is the case generally considered in simple models [12, 13, 68, 149]; the material is not moving around the tool and all the heat is generated by friction.

- $\gamma = 1$ (volume heat source) and $\delta = 0$ (no material circumferential velocity): there is no circumferential velocity but the stirring of the material around the pin generates heat in the TMAZ.
- $\gamma = 0$ (surface heat source) and $\delta = 1$ (material circumferential velocity = tool circumferential velocity): the material rotates with the tool but heat is generated only by material shearing in a thin layer at the tool/material interface. Dong *et al.* [32] showed through their model that such a thin plastic boundary layer is relevant to the FSW process. This is very close to a surface heat source generation and can be modeled as such. Note that in this case the surface heat source distribution proposed here, based on friction equations, might not be totally adequate. Nevertheless with higher circumferential velocities (like at the edge of the shoulder) more heat is generated. A linear variation with radius seems to be reasonable. Schmidt *et al.* [118] used a thin layer as TMAZ in their model and later showed the relevance of this choice [119].
- $\gamma = 1$ (volume heat source) and $\delta = 1$ (material circumferential velocity = tool circumferential velocity): this is another classical assumption [3, 5, 123]; the material rotates with the tool and heat is generated by the deformation of material in the entire TMAZ.

5.2.5 Contact conditions between the workpiece and the backing plate

The contact conditions between the workpiece and the backing plate must be carefully described in the model. Many authors have shown that various options can be considered.

- **CASE 1:** No backing plate. In that situation the lower surface of the workpiece is assumed to be adiabatic. This case would be representative of a very poor contact between the workpiece and the backing plate.
- **CASE 2:** Perfect contact between workpiece and backing plate.
- **CASE 3:** Good contact under the tool region only. The contact under the tool is chosen to be a perfect aluminum/steel contact under the pin and a worse contact (with contact conductance parameter CC_A [W/m²K]) under the shoulder. This case is suggested by experimental observations : the high pressures under the pin and in the surrounding lead to a visible indentation of the upper surface of the backing plate along a width approximately equal to

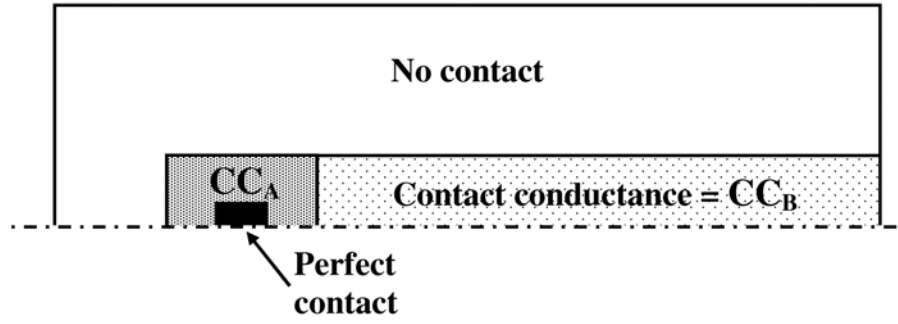


Figure 5.5. Contact conditions between the workpiece and the backing plate in CASE 3 (sketch not to scale)

the diameter of the tool. There, the contact is certainly good. The indentation is more pronounced under the pin region.

At the back of the tool, the contact is worse than under the tool region but difficult to quantify. Therefore a contact conductance parameter CC_B is introduced behind the tool. Figure 5.5 pictures the different contact condition zones.

The different contact conductances are introduced in the finite element code by artificially inserting a layer of 0.2 mm thickness between the workpiece and the backing plate having a thermal conductivity equal to the contact conductance in the given region multiplied by the layer thickness. For the sake of simplicity, the contact surfaces under the tool shoulder and pin are assumed to be square.

5.3 Parameter identification

Table 5.1 summarizes the parameters of the thermal model and the values that have been chosen for these parameters. The optimized parameters have been chosen by minimizing the error between experimentally measured and predicted temperature values. These choices will be extensively discussed in the following.

5.3.1 Thermal properties of the aluminum alloy 6005A

Measurements of the thermal properties of aluminum alloy 6005A-T6 with temperature have been performed at ULg (Université de Liège) [127]. Table 5.2 gives the results of these measurements. The melting

Param.	Significance	Value	Origin
ρ_a	Density	Table 5.2, pg 68	Measurements on 6005A
c_p	Specific heat		
λ	Thermal conductivity		
r_{min}	TMAZ shape parameter	4 mm	Figure 5.3
η	Welding efficiency	95 %	Power and temperature in tool
CC_A	Contact conductivity under the shoulder	100 kW/m ² K	Optimized
CC_B	Contact conductivity at the back of the tool	3 kW/m ² K	Optimized
γ	Q_V/P_{in}	0.2	Optimized
δ	$(v_{mat}/v_{tool}) _{interface}$	1	Optimized

Table 5.1. Summary of the parameters chosen for the application of the thermal model to alloy 6005A

point was also measured and the solidus was found to be equal to 613°C in a DSC heating up test performed at 10°C/min.

5.3.2 Maximum temperature

(a) Definition of the error on the temperature predictions

A mean error definition is introduced in order to compare the various parameters of the model and their influence on the quality of the prediction:

$$Error = \frac{1}{n_t} \sum_{i=1}^{n_t} |T_{exp,i} - T_{model,i}| \quad (5.17)$$

where n_t is the number of thermocouples¹, $T_{exp,i}$ and $T_{model,i}$ are the maximum temperatures at thermocouple position i respectively measured and predicted. Note that these maximum temperatures are not reached at the same time for various transverse positions (y).

This mean error is expressed in °C. The choice of an absolute error rather than a relative one has the advantage of giving the same weight to the error at low temperatures, in the heat affected zone and at high

¹The thermocouples in the backing plate and in the tool are excluded of the error definition since we wish to correctly predict the microstructural evolutions in the workpiece based on that model.

Temperature T [°C]	Density ρ_a [kg/m ³]	Specific heat c_p [J/kg K]	Thermal conductivity λ [W/mK]
25	2680	/	/
50	2680	920	206.6
100	2670	930	208.3
150	2660	950	210.3
200	2650	970	210.3
250	2630	990	210.9
300	2620	1010	208.6
350	2610	1020	205.6
400	2600	1040	201.9
450	2590	1060	197.5
500	2580	1080	190.0
550	/	1110	182.8

Table 5.2. Thermal properties of base alloy number 1 versus temperature

temperature in the weld center. In fact, a good prediction of the temperatures in the heat affected zone is fundamental for the prediction of the local microstructure evolution and the ensuing mechanical properties of the weld.

(b) Exclusion of contact conditions CASE 1 and 2

Figure 5.6(a) compares the predicted maximum temperatures reached at the hottest point under the shoulder behind the pin for weld number "5" (1000 mm/min and 1000 rpm). CASE 1 (no backing plate) leads to unrealistic predictions with temperatures far above the liquidus. The other cases have little influence on the maximum temperature and provide more reasonable values for the maximum temperature. Figure 5.6(b) compares the maximum temperature at mid-thickness at various transverse positions for CASE 2 (perfect contact everywhere) and CASE 3 (good contact under the tool). It is shown that CASE 2 underestimates to a large extent the maximum temperatures measured by the thermocouples away from the centerline. Furthermore, the cooling rate after the passing of the tool is overestimated. Hence, in the following, only contact condition CASE 3 will be considered.

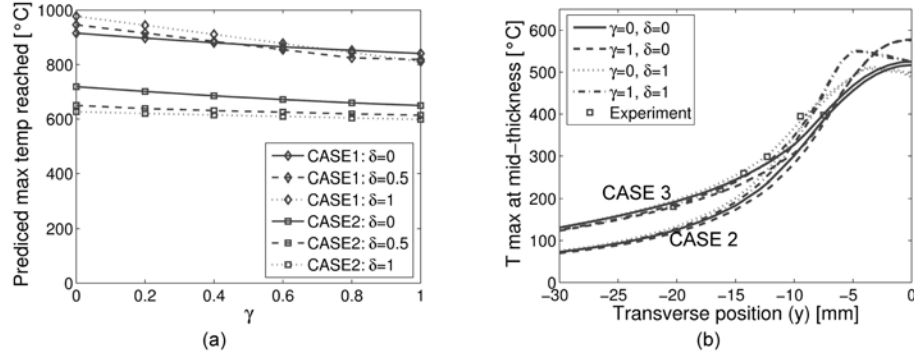


Figure 5.6. CASE 1 and 2 exclusion for a weld with welding parameters 1000 mm/min and 1000 rpm (a) Predicted maximum reached temperature for CASE 1 and 2 for different γ and δ ; (b) Maximum predicted and measured temperature distributions at mid-thickness for CASE 2 and CASE 3 (with $CC_A = 100 \text{ kW/m}^2\text{K}$ and $CC_B = 0 \text{ kW/m}^2\text{K}$) for different values of γ and δ .

(c) Choice of CC_A

The value of the contact conductance behind the tool CC_B has little influence on the predictions of the maximum temperature at the thermocouple positions. Hence, the optimization of the contact conductance under the shoulder CC_A and later the parameters γ and δ is made with a value of $CC_B = 0 \text{ kW/m}^2\text{K}$ (no contact behind the tool).

The maximum temperatures away from the centerline are underestimated when too high a value of the contact conductance under the shoulder region CC_A is chosen. Inversely, too low a value of this contact conductance over-estimates the maximum temperatures away from the centerline. Figure 5.7 shows that the choice of $CC_A = 100 \text{ kW/m}^2\text{K}$ gives a mean error of 10°C to 20°C for a wide range of welding parameters and of γ and δ .

(d) Choice of γ and δ

Figure 5.8 shows the effect of the extreme values of parameters γ and δ on the maximum temperature distribution for weld number "5" (1000 mm/min and 1000 rpm). Figure 5.9 shows the maximum temperature and the mean error on the maximum temperatures predictions for all possible values of γ and δ for three welds with different welding parameters.

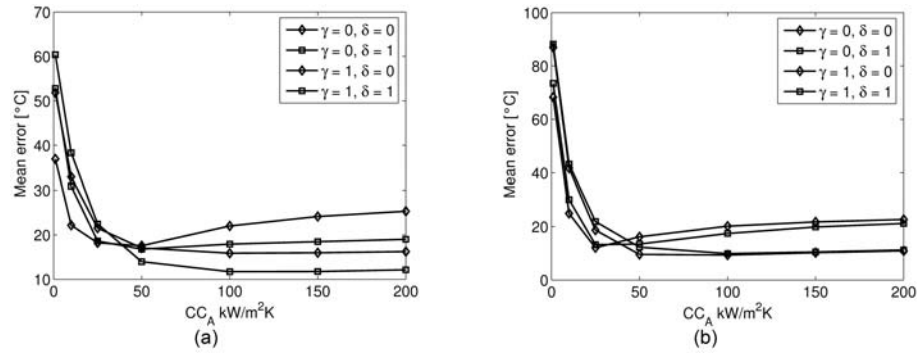


Figure 5.7. Choice of CC_A . Error on the maximum temperatures prediction for different values of γ and δ with welding parameters; (a) 1000 mm/min and 1000 rpm; (b) 200 mm/min and 1000 rpm

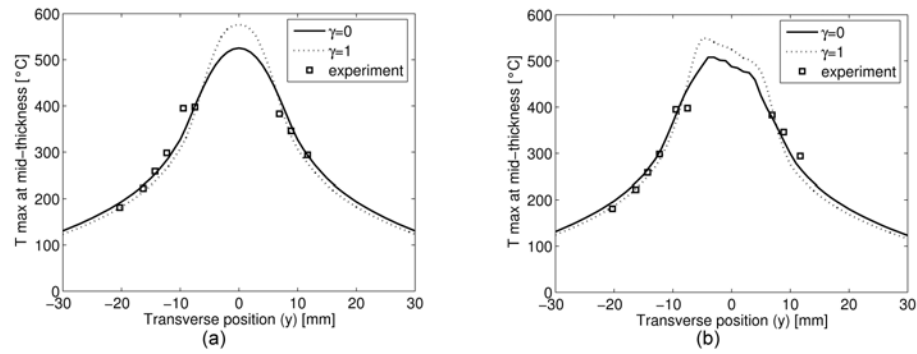


Figure 5.8. Maximum temperature distribution for a weld with welding parameters 1000 mm/min and 1000 rpm; (a) $\delta = 0$; (b) $\delta = 1$.

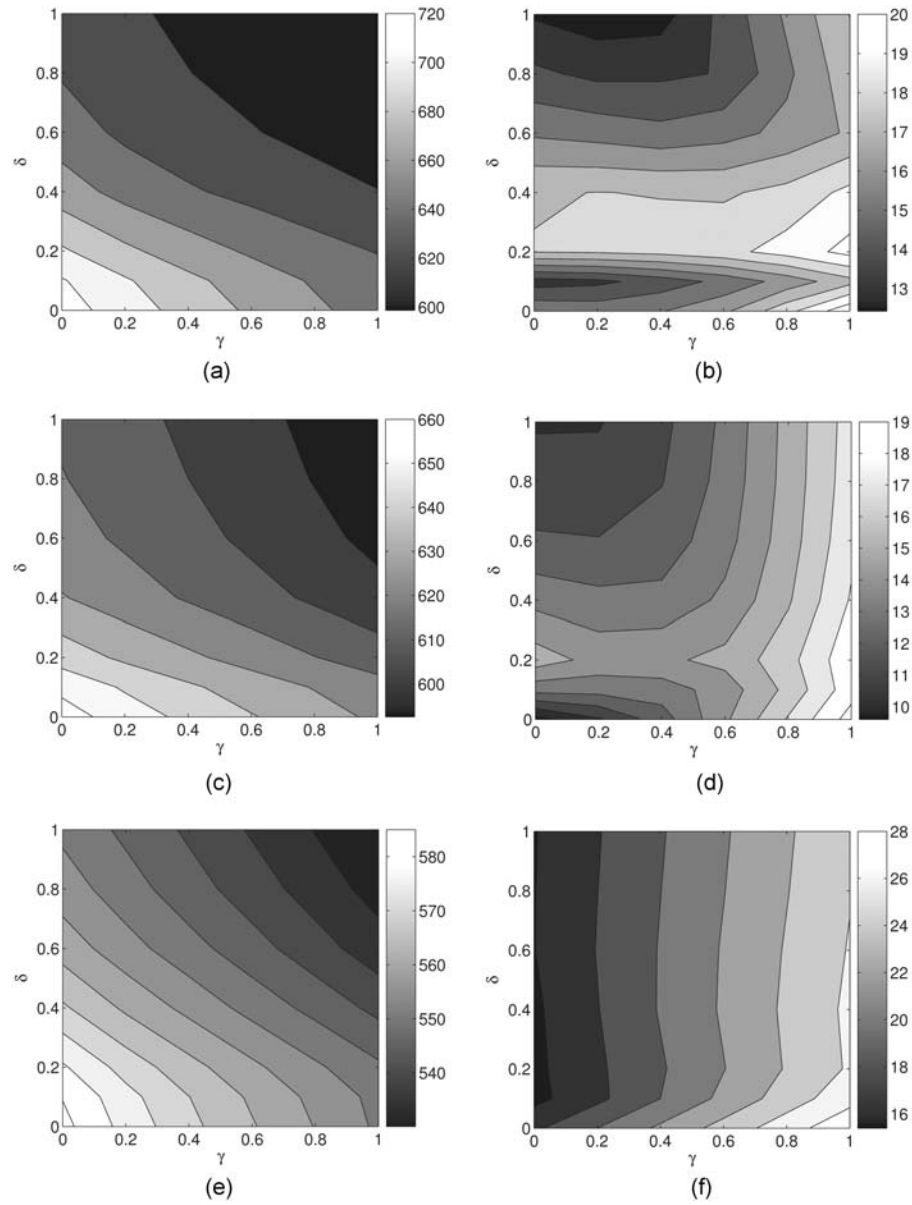


Figure 5.9. (a,c,e) Predicted maximum temperature reached at hottest point and (b,d,f) mean error on the maximum temperature predictions for all possible γ and δ for welds with welding parameters; (a,b) 1000 mm/min and 1000 rpm; (c,d) 200 mm/min and 1000 rpm; (e,f) 200 mm/min and 500 rpm

The maximum predicted temperature reached when $\delta = 0$ and $\gamma = 0$ is unacceptable for some welding conditions (above 700°C , see Figure 5.9(a)) considering that friction stir welding is a solid state welding technique and that the melting point of aluminum alloy 6005A is somewhat above 613°C . Since heating rates are much higher in FSW, the effective solidus temperature will be higher than the 613°C measured by DSC at $10^{\circ}\text{C}/\text{min}$. Nevertheless, a predicted maximum temperature reached at hottest point larger than 700°C for $\delta = 0$ and $\gamma = 0$ is certainly too high. We can safely conclude that the assumption that the heat source is purely frictional and that no material stirring occurs, is not representative of the friction stir welding process. This leads to the conclusion that if a simple model has to be developed without introducing material convection it is more adequate to introduce volume heat sources to represent the heat generated by the process [125, 127].

The choice $\delta = 0$ (see Figure 5.8(a)) would give maximum temperatures symmetrical with respect to the centerline. It has been shown in Figure 4.9 that the advancing side is generally hotter close to the centerline in our experimental friction stir welds and that this asymmetry is reduced when the advancing speed diminishes. This asymmetry can be introduced in the model by taking the material convection into account ($\delta > 0$) (see Figure 5.8(b)). Colegrove *et al.* [17] also concluded that the introduction of a convective heat flow induces asymmetry in the temperature distribution causing the advancing side to be hotter.

Figure 5.9(b,d,f) shows that the mean error on the maximum temperature predictions is minimal in two regions of the γ - δ field for a large variety of welding parameters. The first minimum ($\delta \approx 0$) corresponds to the situation where the material velocity is only a very small fraction of the tool velocity at the tool interface and heat is mainly produced by friction. This case must be excluded due to the too high predicted maximum temperatures reached at hottest point for weld number "5" (1000 mm/min and 1000 rpm) which are incompatible with a solid state welding process as discussed earlier (Figure 5.9(a)). The second mean error minimum corresponds to the case $\delta \approx 1$ (sticking conditions) and $\gamma \approx 0$ (surface heat source). The predicted maximum temperatures reached at hottest point are in a much more acceptable range.

Values of $\delta = 1$ and $\gamma = 0.2$ will be used in the following since the assumption of no volume heat source ($\gamma = 0$) is not reasonable. Indeed a large TMAZ is clearly observed on macro-sections of the welds. Note

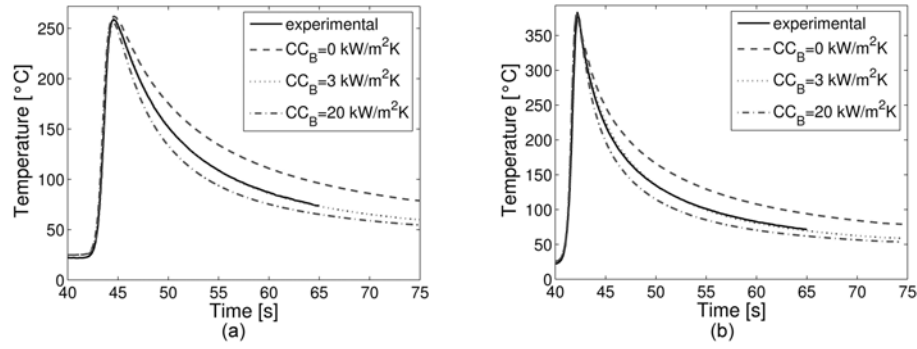


Figure 5.10. Choice of CC_B . Thermal cycles in a weld with welding parameters 1000 mm/min and 1000 rpm for (a) thermocouple 3, positioned at 14 mm from the centerline on the advancing side; (b) thermocouple 10, positioned at 7.2 mm from the centerline on the retreating side.

that the identification of the best value for the δ parameter is not so clear when looking at welds with lower rotational speeds.

5.3.3 Cooling rates

The contact condition of CASE 3 with $CC_B = 0$ kW/m²K behind the tool accurately reproduces the maximum temperature away from the centerline. The cooling rates are however underestimated (see Figure 5.10). This effect can be corrected by using a small contact resistance in the regions surrounding the tool. A value of 3 kW/m²K gives a very good prediction of the cooling rate² for thermocouples placed either away from the centerline (see Figure 5.10(a)) or close to the centerline (see Figure 5.10(b)) for weld number "5" (1000 mm/min and 1000 rpm). The conclusions given earlier concerning the other parameters choice based on the maximum temperature are still valid since the choice of CC_B has a very limited effect on the maximum temperature.

5.3.4 Discussion with confrontation to literature

(a) γ and δ values

Chao and colleagues [12, 13] assumed a frictional heat source without including material flow with a fitted total power input of 1.4 kW. The welding parameters were a rotational speed of 400 rpm and an advancing speed of 120 mm/min. Their 6061-T6 aluminum alloy plates were 6.4

²In Figure 5.10 the quality of the prediction is so good that the curves cannot be distinguished for the case $CC_B = 3$ kW/m²K.

mm thick. For similar welding conditions, the power input measured in the present study is much higher, about 3 kW. The very low value of the fitted power input reported in their article confirms that a purely surface heat source is probably not adequate. Similarly, by using a rather low value for the friction coefficient, Zahedul *et al.* [149] conclude that a purely frictional heating model is probably not adequate.

The presented parametric study leads to the conclusion that the material at the tool interface adheres to the tool and that heat is generated in a small layer with high shear strains. Smith *et al.* [119] did not impose a contact condition at the tool-workpiece interface in their model. They could therefore conclude on that important aspect of the process modeling. They showed that non-uniform contact conditions close to sticking are present along most of the interface except at the vicinity of the tool shoulder and at the lower trailing advancing region of the probe/matrix interface. Xu *et al.* [146] proposed two models of the process: one for sliding contact and the other for frictional conditions at the interface. They concluded that their visualization experiments are correctly reproduced by the models when the interface is partially sticking ($\delta = 0.48$ and 0.30 for two different welding conditions).

It is a tautology to state that the conclusions of this study are correct if the assumptions at the basis of the calculation are correct. Let us be more specific. If the volume heat source distribution supposed here was incorrect, the conclusions concerning the value of δ should be modified especially if the exact distribution of the volume heat source is asymmetric. All these effects could balance the material convection asymmetry and lead to slightly different conclusions. Seidel *et al.* [120] and Colegrove *et al.* [18] concluded from their 2-D computational fluid dynamics model that the width of the deformed region is smaller on the advancing side than on the retreating side. Our macrographs of the welds do not show any clear evidence of asymmetry in the TMAZ shape between the advancing and the retreating sides of the weld. Another aspect not taken into account in this model is the vertical flow of material due to the presence of a thread on the pin surface. This would tend to diminish the maximum temperature reached at the hottest point when surface heat sources are introduced and might affect the conclusions concerning the correct values of γ and δ . A decrease of the maximum temperature reached at hottest point would be in favor of choosing a lower value for the δ parameter leading to conclusions similar to those of Xu *et al.* [146].

(b) Contact conditions between the workpiece and the backing plate

Shi *et al.* [124] proposed a value of the contact conductivity similar to our CC_B for temperatures below 150°C and equal to about 20 kW/m²K for temperatures higher than 300°C. Khandkar *et al.* [68] used a contact conductivity between the workpiece and the backing plate equal to 10 kW/m²K in front of the tool and 100 kW/m²K at the backside of the tool. They also introduced a contact conductivity between the workpiece and the backing plate equal to 1 kW/m²K in the other regions. The choice made in the present study is very similar, i.e. a contact conductivity equal to 3 kW/m²K behind the tool and 0 kW/m²K elsewhere except under the tool where a much higher value is taken but without distinguishing between the front and the back of the tool. The value of CC_A proposed earlier (100 kW/m²K) corresponds to the value proposed by Khandkar *et al.* [68] at the rear of the tool. Hence, the scientific literature seems to agree with the order of magnitudes chosen for CC_A and CC_B .

It would be interesting to perform more measurements of the temperature in the backing plate at various positions away from the centerline just under and just above the contact in order to have a better understanding of the contact conditions prevailing in the friction stir welding process. The only temperature measured in the backing plate in the course of this study is overestimated by the model, which predicts a maximum temperature of about 400°C for weld number "5" while a value of 224°C has been measured. This is probably due to the simplistic assumption of a perfect contact under the pin region.

5.4 Effects of the welding parameters

For the sake of validation, the model was applied to various welding conditions with the values of the parameters identified previously mainly on weld number "5". Figures 5.11(c) and (d) and 5.12 show that the model provides very good predictions of the maximum temperatures for a wide range of welding parameters. The maximum temperatures are generally slightly underestimated close to the centerline. This effect is more obvious when the rotational speed is lower. It was shown earlier that the choice of δ is not self-evident for a low rotational speed weld (Figure 5.9(e,f)).

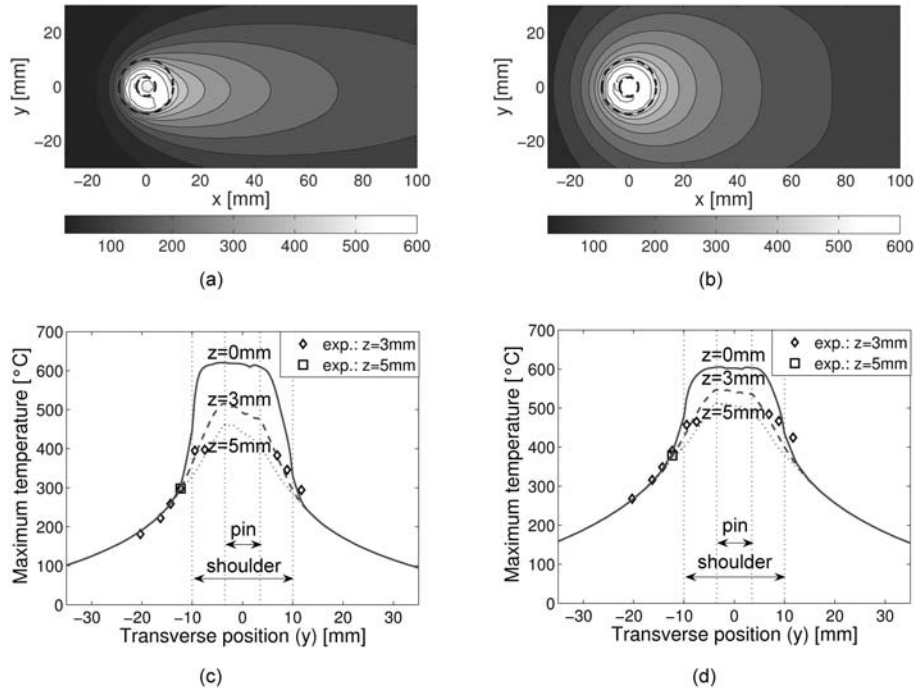


Figure 5.11. (a,b) Cartography of the temperatures on the top surface of the plates (c,d) Maximum temperatures distribution at different position in depth, comparison of model and experiment. Welding parameters: (a,c) 1000 mm/min and 1000 rpm; (b,d) 200 mm/min and 1000 rpm

The maximum temperature distribution in a cold weld (see Figure 5.11(a) and (c)) is quite different from the maximum temperature distribution in a hot weld (see Figure 5.11(b) and (d)). The predicted maximum temperatures reached at the hottest point are actually similar in these so-called hot weld and cold weld (see Figure 5.12(d)). It is the temperature distribution away from the centerline, in the heat affected zone which justifies these designations of hot and cold weld.

The temperature difference between the advancing and the retreating side of the weld is only partially well predicted. Figure 5.13 shows the maximum temperature difference between the advancing and the retreating side of the weld 9.2 mm from the weld centerline, comparing predicted and experimental values for different welding parameters. The model overestimates the temperature differences in nearly all cases but the trends are correctly reproduced: namely, no marked effect of the rotational speed and an increase of the temperature difference with

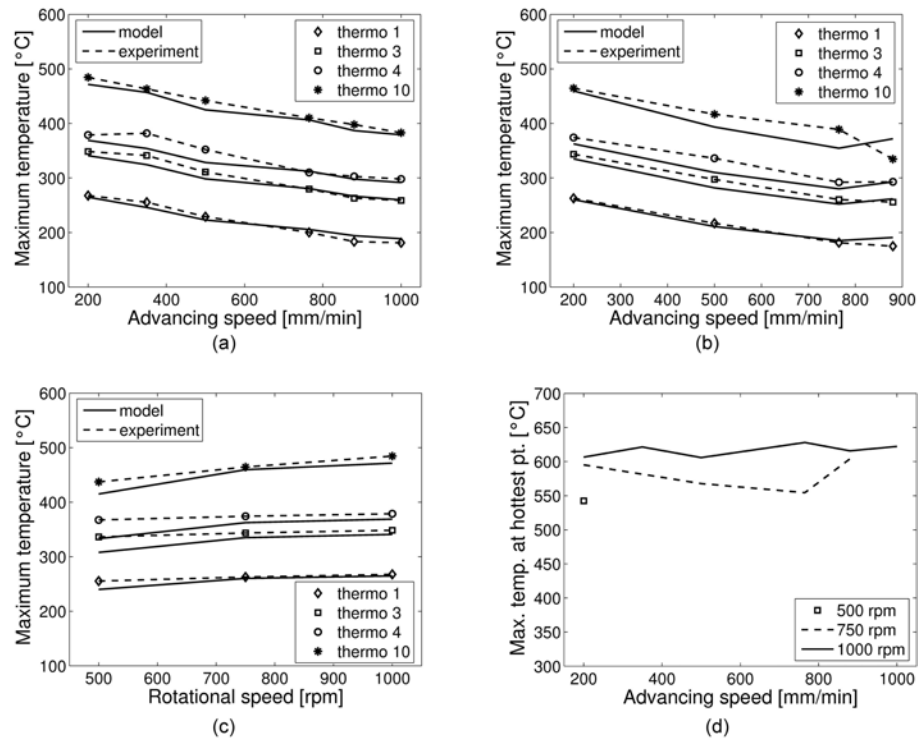


Figure 5.12. Assessment of the model for different welding parameters. Maximum temperatures at some thermocouple positions for different advancing speeds for a rotational speed of (a) 1000 rpm and (b) 750 rpm. (c) Maximum temperature at some thermocouples position for different rotational speeds for an advancing speed of 200 mm/min. (d) Evolution of the predicted maximum temperature reached at hottest point for different welding parameters

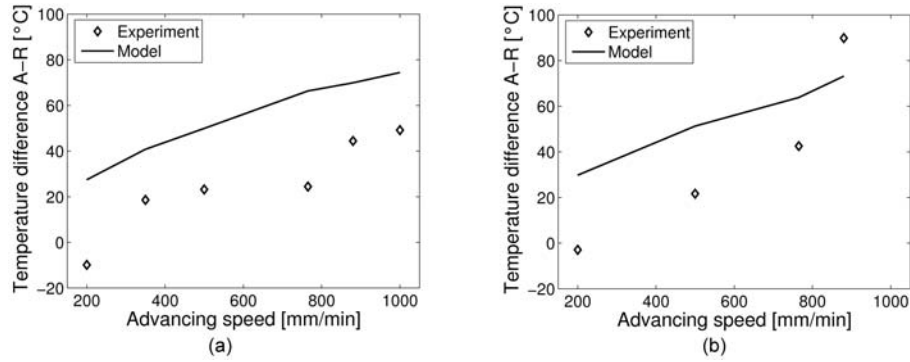


Figure 5.13. Effect of the welding parameters on the maximum temperature difference between the advancing and the retreating side of the weld, 9.2 mm from the weld centerline. Rotational speeds are (a) 1000 rpm and (b) 750 rpm

increasing advancing speeds. The experimental observation of a hotter retreating side for low advancing speeds needs to be confirmed and cannot be reproduced by the model. It is not the frictional difference between the two sides of the weld (the two sides have different relative speeds) which can cause this difference since it has a negligible effect on the temperature difference. Maybe a more sophisticated flow pattern can explain the hotter retreating side measured for some welding parameters.

5.5 Conclusions

An asymmetric finite element model of the friction stir welding process has been developed and successfully validated based on the large variety of welding experiments presented in chapter 4. The importance of the contact conditions between the workpiece and the backing plate are of tremendous importance when looking at temperatures away from the centerline and at the cooling rates. A simplified model of the material flow around the tool allowed to take into account the forced material convection effect around the tool. The experimentally observed asymmetry of the weld and a warmer advancing side are well modeled through this simple assumption.

The contact conditions between the tool and the workpiece have been identified as sticking on the basis of the maximum temperature measured by the thermocouples. Heat is generated mainly close to the surface between the tool and the workpiece and hence heat generation can

be modeled by surface heat sources when taking the effect of material convection into account.

Part III

Microstructure evolution and mechanical behavior

ABSTRACT

Chapter 2 has presented the different types of precipitates found in aluminum alloys of the 6xxx series with low copper content. These are in 3 main categories: the intermetallics, the dispersoids and the hardening precipitates. This last category of precipitates will be largely affected by the welding process. The other precipitates are essentially unaffected by the welding process except in the thermo-mechanically affected zone where they can be fragmented. The experimental results of this part of the thesis show that the volume fraction and aspect ratio of the intermetallics are globally unaffected by the welding process while the size of these phases is reduced in the thermo-mechanically affected zone. The base material used in this study, i.e. aluminum alloy 6005A-T6, corresponds initially to a maximum hardened state. Hence, the welding process can only deteriorate the local strength affecting the transverse tensile properties of the weld. The main question that will be discussed in this part of the thesis, is to what extent the microstructure is affected by the welding process and how this microstructural evolution affects the tensile properties of the various zones of the weld. This discussion includes experimental and modeling results.

Chapter 7 presents a numerical microstructure evolution model based on a discretization in size classes of the hardening precipitates taken as the nucleation, dissolution and coarsening of only one phase of composition Mg_2Si . In the yield strength model, also presented in chapter 7, the contributions of the solid solution and precipitation to the yield strength are added up. Finally, a natural aging model predicts the increase of the yield strength after long term natural aging. In particular, it has been identified that a minimum supersaturation in solid solution concentration is required for natural aging. This last observation is crucial when looking at the evolution of precipitates in welds since the heat affected zone does not present a sufficient solid solution concentration. Hence, since the solid solution concentration is not sufficient, no natural aging occurs in the heat affected zone reducing thus the strength of that zone.

Chapter 8 presents a novel model of strain hardening. The strain hardening parameter θ (which, in a pure metal, would reflect the dislocations storage rate) is affected by the following physical mechanisms:

- Dynamic precipitation is the precipitation induced by the movement of dislocations during plastic deformation. When the solid solution is supersaturated, dynamic precipitation will increase the flow stress during deformation and cause an apparent increase of the strain hardening parameter θ .
- The formation of Orowan loops when the precipitates are by-passed by the dislocations increases the storage of dislocations and hence the strain hardening parameter θ . Nevertheless, in classical approaches, the effect on the strain hardening parameter θ would be inversely proportional to the precipitate radius and thus would decrease continuously when precipitate radii grow beyond the shearing / by-passing transition. Experimentally, an increase is observed for medium size precipitate radius. This is attributed to the inefficient storage of Orowan loops around precipitates that are coherent or semi-coherent with the matrix. Indeed, in that case, the Orowan loops penetrate the precipitates and auto-annihilate.

The strain hardening parameter β (the dynamic recovery rate) is affected by the following physical mechanisms:

- If the yield strength increases, the annihilation distance of two dislocations of opposite sign decreases and hence the strain hardening parameter β increases. There is probably an effect of the solid solution concentration on dynamic recovery but it has not been taken into account at this level.
- The efficient storage of Orowan loops increases the dynamic recovery rate by favoring the de-localization of the dislocation glide. This effect, called the "dislocation lift", has been quantified by estimating the probability for two moving dislocations of opposite sign to meet a precipitate having an Orowan loop efficiently stored around it in the time needed for their mutual annihilation.

The microstructure evolves as follows when moving from the base material to the weld center.

- The base material is composed of a large density of fine β'' precipitates.
- In the medium-level hardness zone, some β'' precipitates dissolve while others form β' precipitates. The dissolution of β'' precipitates in that region of the weld has been identified by the DSC measurements and confirmed by post-welding heat treatments and the microstructure evolution model.
- The low-hardness heat affected zone is composed of large β' precipitates and a certain amount of solid solution.
- The weld nugget presents a high solid solution concentration. No β'' , nor β' precipitates were observed by TEM in that zone of the weld. Some β precipitation takes away a fraction of the solute. This precipitation was identified by DSC measurements of the weld nugget and confirmed by post-welding heat treatments.

The microstructure of the heat affected zone is more affected by the advancing speed than by the rotational speed except for what concerns the width of the thermally affected zone.

- The heat affected zone of low advancing speed welds presents much larger precipitates than the heat affected zone of high advancing speed welds. This has been identified by TEM image analysis. The microstructure evolution model shows that this is due to the high heating and cooling rates in high advancing speed welds.
- The heat affected zone of low advancing speed welds has a larger volume fraction of precipitates (and hence a lower solid solution concentration) than the heat affected zone of high advancing speed welds. This was first identified by the microstructure evolution model but is confirmed by the measured lower hardness in the heat affected zone after a post-welding heat treatment of a low advancing speed weld compared to that of a high advancing speed weld. The low solid solution concentration in the heat affected zone (after welding and natural aging) of low advancing speed welds explains the lower hardness and yield

strength of that zone since no natural aging is possible when the solid solution concentration is not sufficient.

The strain hardening parameters evolve as follows when moving from the base material to the weld center.

- The base material presents a minimum value of the two strain hardening parameters θ and β since no dynamic precipitation is possible; the yield strength is maximum and no Orowan loops are stored.
- The heat affected zone presents a high value of the dislocation storage rate θ since the large precipitates present in that zone of the weld favor the stability of the Orowan loops. The dynamic recovery rate β of that zone is also high since the Orowan loops formed favor the "dislocation lift" effect.
- The weld center presents a high value of the dislocation storage rate θ due to the dynamic precipitation which is favored by the residual solid solution concentration of that zone of the weld when it has aged naturally. The dynamic recovery rate is rather low in that region of the weld since no "dislocation lift" effect is possible and the effect of the yield strength on dynamic recovery is clearly a second order effect.

When the advancing speed decreases the precipitate radius in the heat affected zone is higher and hence both strain hardening parameters increase with decreasing advancing speed.

The consequence of the distribution of the strain hardening parameters throughout the weld zones is that the weld center has a higher strain hardening capacity than the heat affected zone. Hence, it is mainly strain hardening that causes the heat affected zone to be the weakest zone of a friction stir weld. Indeed, the yield strength is very similar in those two zones of the weld, in particular in low advancing speed welds, while the tensile strength of the weld center is higher than the tensile strength of the heat affected zone. The strength mismatch between these two zones increases with decreasing advancing speed.

Chapter 6

Microstructure and local mechanical characterization

This chapter presents the microstructure and mechanical characterization of both the base material (in section 6.1) and the friction stir welds (in sections 6.2 and 6.3). This chapter aims at presenting the experimental results which will be discussed in the modeling chapters (7 and 8). Nevertheless, some aspects of the results will already be discussed at this stage.

6.1 Characterization of the base material 6005A-T6

6.1.1 Microstructural characterization

(a) Coarse phases

These phases have been observed by both back-scattered scanning electron microscopy (SEM) and transmission electron microscopy (TEM). They are of three types: the intermetallics (essentially the $\alpha(\text{AlFeMnSi})$ phase), the $\beta(\text{Mg}_2\text{Si})$ coarse phases and the silicon rich precipitates.

Figure 6.1 shows SEM and TEM images of α particles. Table 6.1 shows the results of an image analysis of these phases¹ based on 30 images containing each about 100 precipitates. The Feret diameter is defined as the maximum distance between two extreme points of a particle.

¹Since the α coarse phases are by far the dominant second phase, the volume fractions of the other coarse phases (β - Mg_2Si coarse phases and Si rich phases) are not studied.

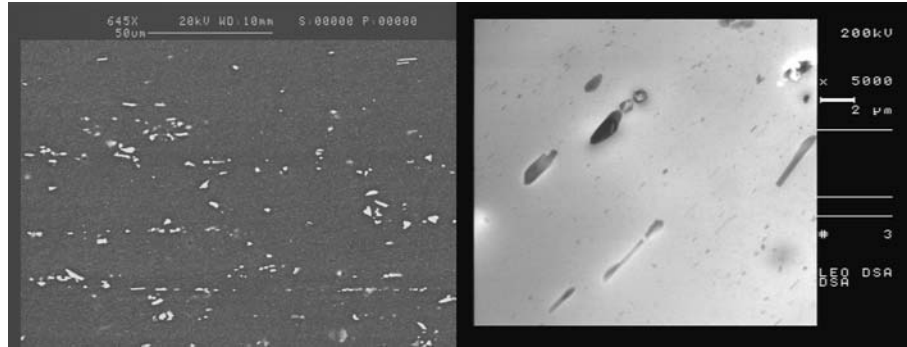


Figure 6.1. Coarse α phases in the base material (observed by SEM and by TEM)

Precipitate property	Mean value	Standard deviation
Surface fraction	1.2%	0.44%
Shape ratio	1.7	0.14
Area radius	0.9 μm	0.33 μm
Feret radius	1.1 μm	0.17 μm
Largest axis of the closest ellipse	2.1 μm	0.16 μm
Smallest axis of the closest ellipse	1.2 μm	0.08 μm

Table 6.1. Base material α phases analysis

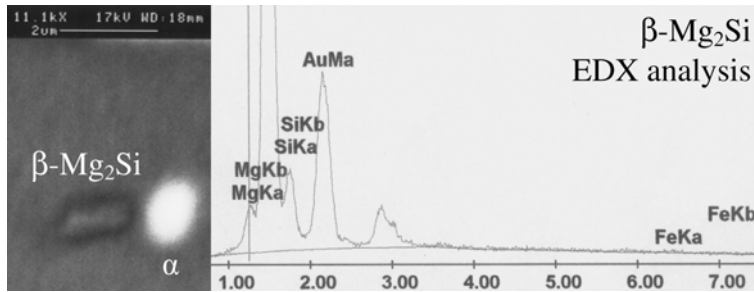


Figure 6.2. $\beta(Mg_2Si)$ phase SEM image and EDX analysis

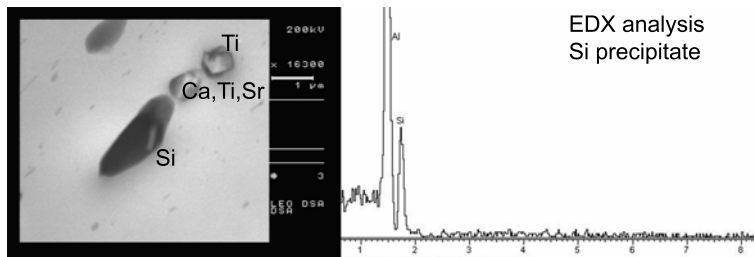


Figure 6.3. Si phase (and other): TEM image and EDX analysis of the Si phase.

Some $\beta(Mg_2Si)$ coarse phases can also be observed by SEM but studying their distribution by image analysis is delicate since they are highly dispersed. Their typical size is similar to the α phase. Figure 6.2 shows a SEM micrograph of a $\beta(Mg_2Si)$ phase and the EDX analysis of this $\beta(Mg_2Si)$ phase (note that the presence of gold is due to the coating). This EDX analysis shows the high concentration of magnesium in these phases which is clearly not observed when analyzing $\alpha(AlFeMnSi)$ particles.

Silicon precipitates have been observed by transmission electron microscopy. Their formation is the consequence of the excess silicon in the alloy composition remaining after the formation of the $\beta(Mg_2Si)$ phases, the α phases and the dispersoids. Figure 6.3 shows a TEM micrograph of a silicon rich precipitate and its EDX analysis. The size of Si precipitates varies in a wide range. Particles of 90 to 1300 nm have been observed. The aspect ratio of the Si precipitates is always close to 3. Finally, minor amount of other phases rich in the other, low content, alloying elements, like titanium, are also shown in Figure 6.3.

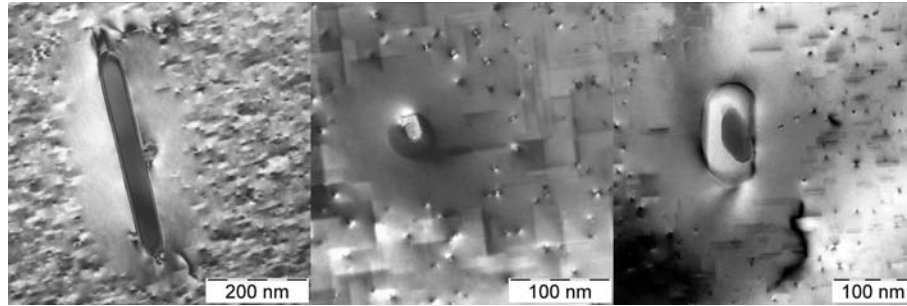


Figure 6.4. Dispersoids in the base material

(b) Dispersoids

Dispersoids are much smaller than the coarse phases but still do not contribute to material hardening. Their size is typically equal to 100 – 150 nm. Those precipitates are the background "dots" observed in Figures 6.1 and 6.3. These particles contain much more manganese than the intermetallics and an appreciable amount of silicon.

Figure 6.4 shows that these precipitates are often found by pair. Although EDX analysis reveals that both precipitates of a pair contain manganese, it might be possible that one of these precipitates is a coarse $\beta(\text{Mg}_2\text{Si})$ or β' since the EDX magnesium peak might be hidden by the other more dominant peaks. Nevertheless, if these particles are both dispersoids, this could be due to heterogeneous precipitation of one dispersoid on the other. Before the final T6 heat treatment, the material overcomes different heat treatments. These heat treatments could cause different dispersoids precipitation stages. It is hence possible that a second dispersoid has precipitated heterogeneously on a pre-existing one.

The precipitates of Figure 6.4 are surrounded by a depleted zone in which no fine precipitation is observed. This depletion is probably due to the high silicon content of the dispersoids (and, possibly, due to the magnesium content if one of these precipitates is a $\beta(\text{Mg}_2\text{Si})$) which leaves insufficient silicon for the $\beta'' - \beta'$ precipitation later in the alloy heat treatment stages. Nevertheless, the diffusivity of elements in solid solution should be sufficient to avoid this effect. The stress field caused by the dispersoids could also be a reason for the depletion.

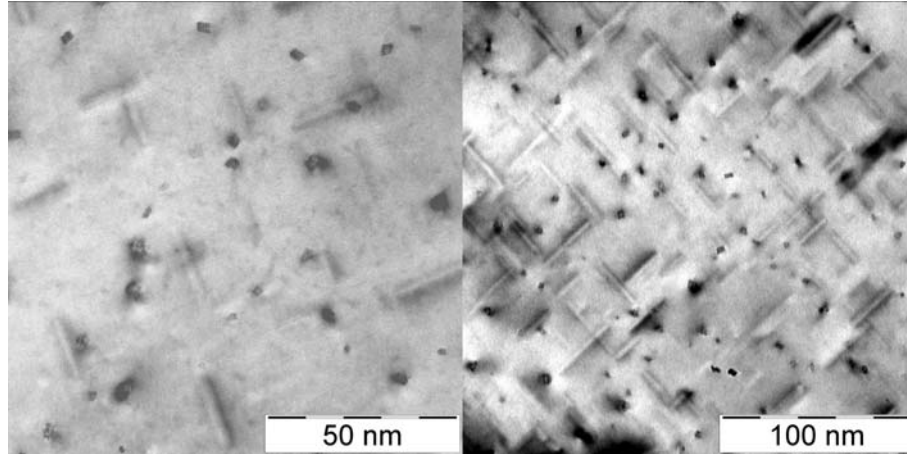


Figure 6.5. β'' precipitation in the base material ($[100]_{Al}$ zone axis).

(c) Hardening precipitates

As shown in Figure 6.5, a high density of fine precipitates is observed by transmission electron microscopy (TEM) in the base material aluminum, i.e., the alloy 6005A-T6. The DSC response of the base material presented in Figure 6.6 shows indeed a large β'' dissolution peak at about 260°C and a β' precipitation peak at about 300°C . The fine precipitates in the micrographs of Figure 6.5 are thus β'' precipitates. The precipitates observed in Figure 6.5 have squared sections as can be expected for β'' precipitates, see Table 2.1 (page 10).

Analyzing 620 end-on precipitates (i.e. precipitates with the long axis perpendicular to the plane) and 140 precipitates parallel to the plane (i.e. precipitates with the long axis parallel to the plane) gives the precipitates size distribution shown in Figure 6.7. The number of β'' precipitates is three times the number of end-on precipitates to take the two other possible orientations into account [38]. Radius and length size classes are chosen to be respectively 0.25 nm and 10 nm wide in order to obtain smooth size distributions.

Figure 6.8(a) shows for all the precipitates with the long axis in the plane of observation that there is no strict correlation between the length and the width of these particles. An equivalent radius is defined based on equivalent volumes for precipitates having their length equal to the mean length. Figure 6.8(b) shows the equivalent radius distribution of

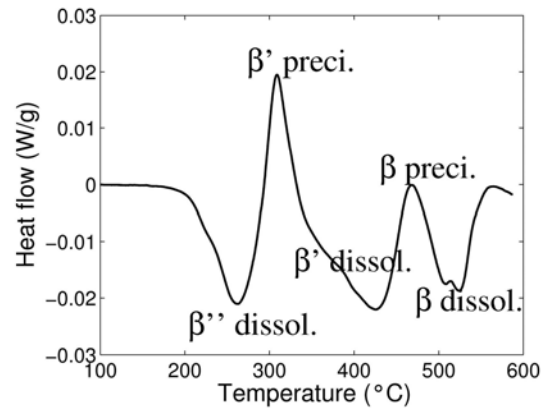


Figure 6.6. DSC curve of base alloy number 1.

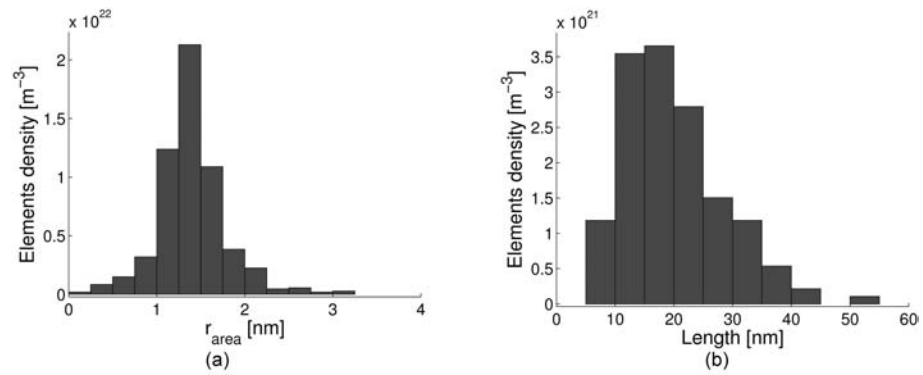


Figure 6.7. β'' precipitates size distribution in the base material. (a) Particles area radius distribution; (b) particles length distribution.

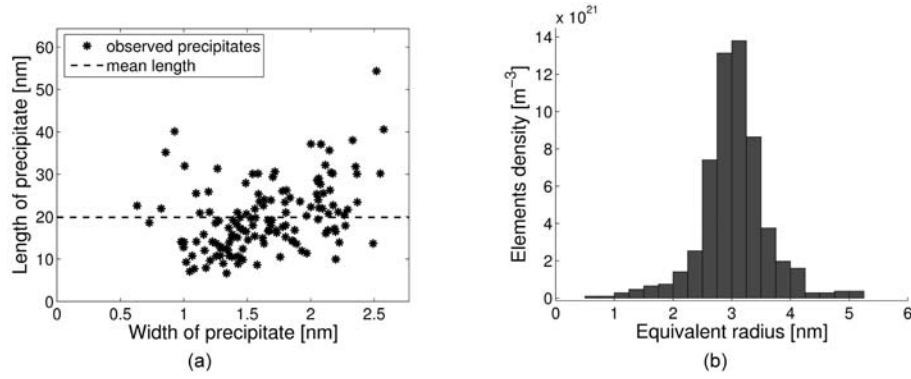


Figure 6.8. β'' precipitates size distribution in the base material: (a) length as a function of width for the precipitates with the long axis in the plane of observation; (b) equivalent radius distribution considering all the precipitates lengths are equal to the mean length.

Precipitate dimensions	Orientation of the precipitates	Mean value	Standard deviation
Total density	end-on (* 3)	$5.8 * 10^{22} m^{-3}$	/
Area radius	end-on	1.4 nm	0.4 nm
Feret radius	end-on	3.5 nm	1.1 nm
Length	long axis parallel to plane	20 nm	8 nm
Equivalent radius	end-on (+ mean length)	3.04 nm	0.6 nm

Table 6.2. Base material β'' precipitates analysis.

the base material β'' precipitates. Table 6.2 summarizes the results of the image analysis.

The total number of precipitates per unit volume (integration of the distribution in Figure 6.8(b)) has been compared to the maximum volume fraction considering all the magnesium had precipitated into $\beta(\text{Mg}_2\text{Si})$ compounds. This maximum volume fraction is equal to 0.75%. The volume fraction obtained by TEM images analysis is very close to 0.75%.

6.1.2 Natural aging

Alloy 6005A naturally ages at room temperature in less than one year depending very much on the alloy exact composition and in particular in

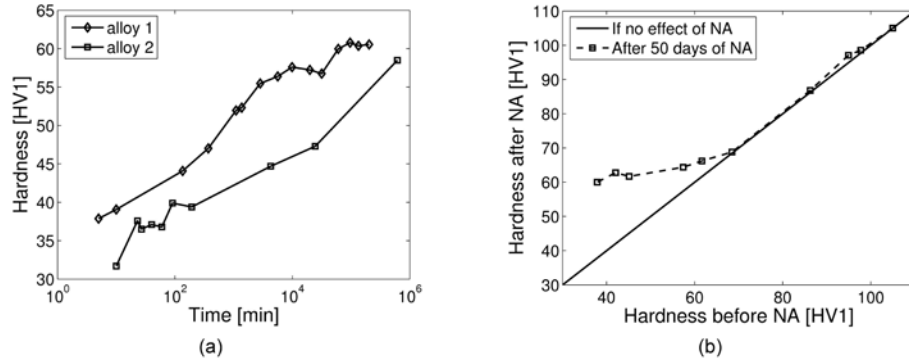


Figure 6.9. Natural aging (NA) effect on hardness: (a) evolution of hardness as a function of time after complete solution heat treatment (SHT); (b) comparison of the hardness before (less than 10 minutes of NA) and after natural aging (50 days) of partially dissolved samples.

Time of natural aging	< 10 min	35 days	100 days
Yield strength at 0.2%, R_p [MPa]	44.1	95.5	99.2
Tensile strength, R_m [MPa]	128.1	204.9	210.6
True strain at onset of necking, ε_u	0.173	0.209	0.215
Strain hardening exponent, n_u	0.326	0.200	0.213
Dislocation storage rate, θ [MPa]	1868	1934	1872
Recovery rate, β	16.9	11.2	10.4

Table 6.3. Natural aging effect on the tensile properties evolution after solution heat treatment in alloy number 1.

the copper content. The natural aging of the base materials was studied by solutionizing the material (530°C for 1 h, see[28]) and by measuring then the hardness evolution with time (see Figure 6.9(a)). The evolution of the tensile properties of base alloy number 1 at three different times of natural aging following a solution heat treatment are given in Table 6.3.

DSC is the only technique used in this study to quantify the extent of GP zones precipitation which can hardly be observed with classical TEM. Figure 6.10 compares the DSC curve after 3 weeks and 1.5 years of natural aging following solution heat treatment. Before the $\beta'' - \beta'$ precipitation peak observed at around 260°C – 310°C, an endothermic peak is detected around 220°C. This peak results from the dissolution of

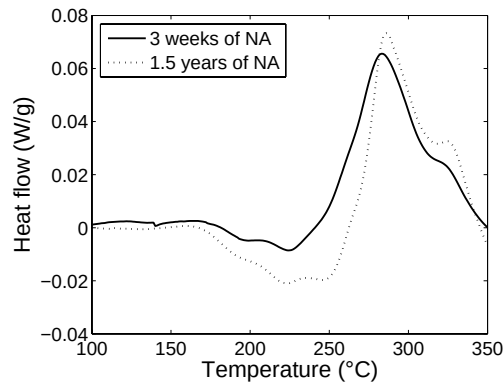


Figure 6.10. DSC curve of solution heat treated specimens having an intermediate natural aging level (NA = 3 weeks) and a completed natural aging level (NA = 1.5 years).

GP zones. The area of the GP dissolution peak after 3 weeks of natural aging is much smaller than after 1.5 years of natural aging confirming that the extent of GP zones precipitation significantly increases, even after 3 weeks of natural aging.

A low concentration in solid solution does not favor natural aging. A driving force is needed for natural aging. Figure 6.9(b) shows the evolution of the hardness of different samples heat treated for less than 10 seconds at various temperatures ranging from 275°C to 430°C². There seems to be a low hardness level of about 60 – 70 HV1 needed for the natural aging process to proceed. This low hardness level corresponds to a high solid solution concentration. The need for a high driving force (a high solid solution concentration) to precipitate GP zones could be due to the relatively low copper content of the 6005A aluminum alloy. Deschamps *et al.* [25] have shown that the copper content plays an important role on favoring precipitation, of GP zones in particular. Base alloy number 1 has a relatively low copper content. The slower GP zone precipitation kinetic of base alloy number 2 compared to base alloy number 1, which has a slightly higher copper content, confirms the tremendous effect of the copper content on natural aging (see Figure

²It is of no interest to indicate the exact times and the exact temperatures of the heat treatments since so short heat treatments are not reproducible and the samples do not reach directly the imposed temperature. The choice of working with very short heat treatment was made in order to limit the effect of coarsening that also has an impact on hardness.

6.9(a)).

6.1.3 Tensile properties of heat treated specimens

Figure 6.11 and 6.12 show the evolution of the tensile properties of alloy number 1 as a function of the duration of the heat treatment. The yield strength results are classically presented and commented in the scientific literature for other alloys of the 6xxx series [56, 57, 63, 90, 99, 100, 144], these will be the basis of the discussion of our results in this section.

Figure 6.11 shows the tensile properties of samples heat treated at 180°C, starting from a supersaturated solid solution comparing the effect of carrying out the heat treatment after 2-3 days of natural aging and after 2 weeks of natural aging. The 180°C heat treatment was not performed immediately after the solution heat treatment since it would then take a very long time for the material to harden. On the other hand, Figure 6.11 shows that there is no need to wait more than a few days for starting the hardening heat treatment since waiting for 2 weeks does not accelerate significantly the precipitation kinetics, small germs of GP zones are sufficient to favor precipitation of β'' . During this heat treatment, the evolution of the strengthening precipitates and the ensuing mechanical properties is the following:

- For time $< 6 - 10$ h, β'' precipitates grow to a size that insures optimal strength. The precipitates volume fraction is maximum after 6 to 10 h at 180°C [28]. The increase of the precipitates volume fraction causes an increase in strength and a decrease in strain at necking. The strain hardening parameter β seems to decrease only very slightly when precipitates grow to their optimal size. The strain hardening parameter θ is much more affected by this precipitation growth.
- For time $> 6 - 10$ h, β'' precipitates grow and eventually transform to β' and $\beta(\text{Mg}_2\text{Si})$ precipitates. The size of the precipitates being greater than a critical value, the precipitates are bypassed by the dislocations following the Orowan mechanism and this leads to a decrease in strength with increasing precipitate size. The strain at necking keeps decreasing. The strain hardening parameters θ and β increase drastically when the precipitate size increases.

Figure 6.12 shows the tensile properties of samples heat treated at 300°C and 350°C from the T6 state, i.e. base alloy number 1. Here

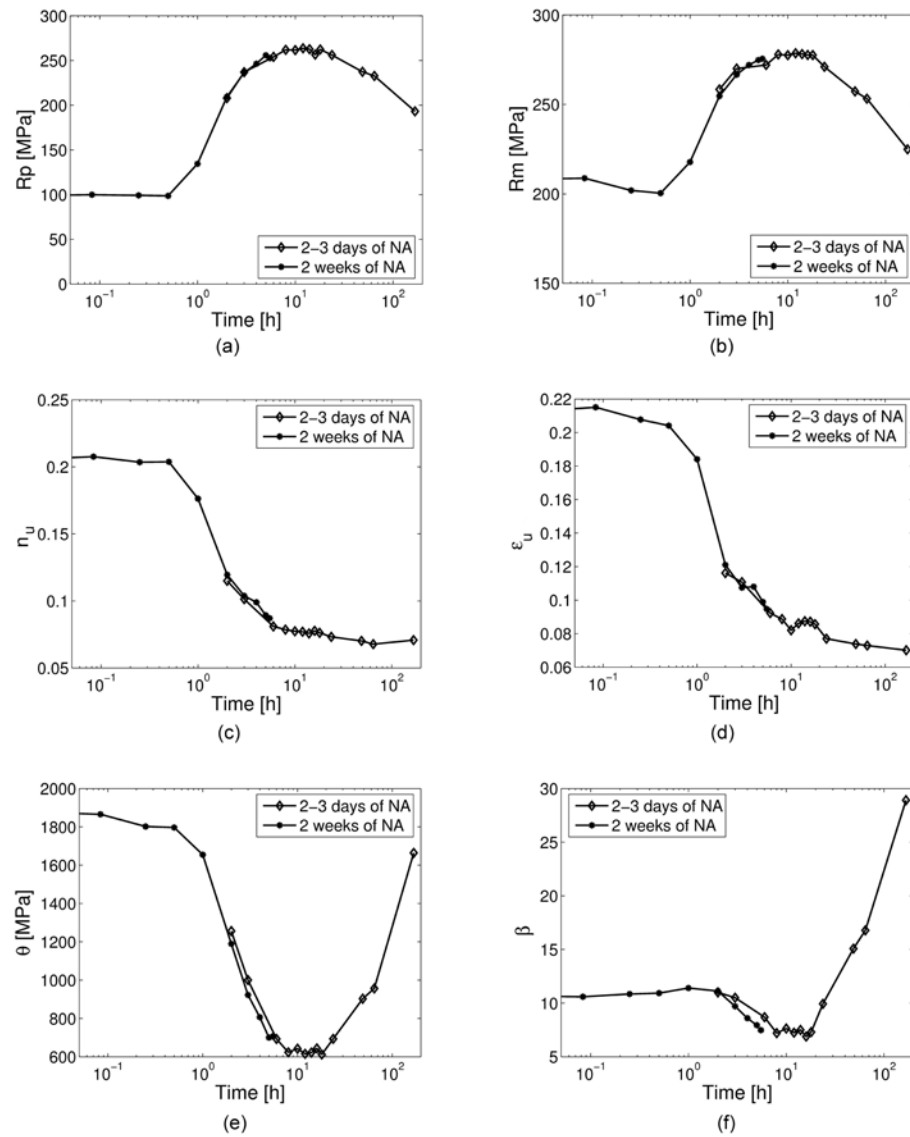


Figure 6.11. Evolution of the tensile properties as a function of the duration of heat treated at 180°C for alloy number 1. The heat treatment is performed after solutionizing and natural aging. Two times of natural aging (NA) after solutionizing and before the heat treatment are compared. (a) Yield strength R_p ; (b) ultimate tensile strength R_m ; (c) strain hardening exponent at necking n_u ; (d) true strain at the onset of necking ϵ_u ; (e) θ strain hardening parameter; (f) β strain hardening parameter.

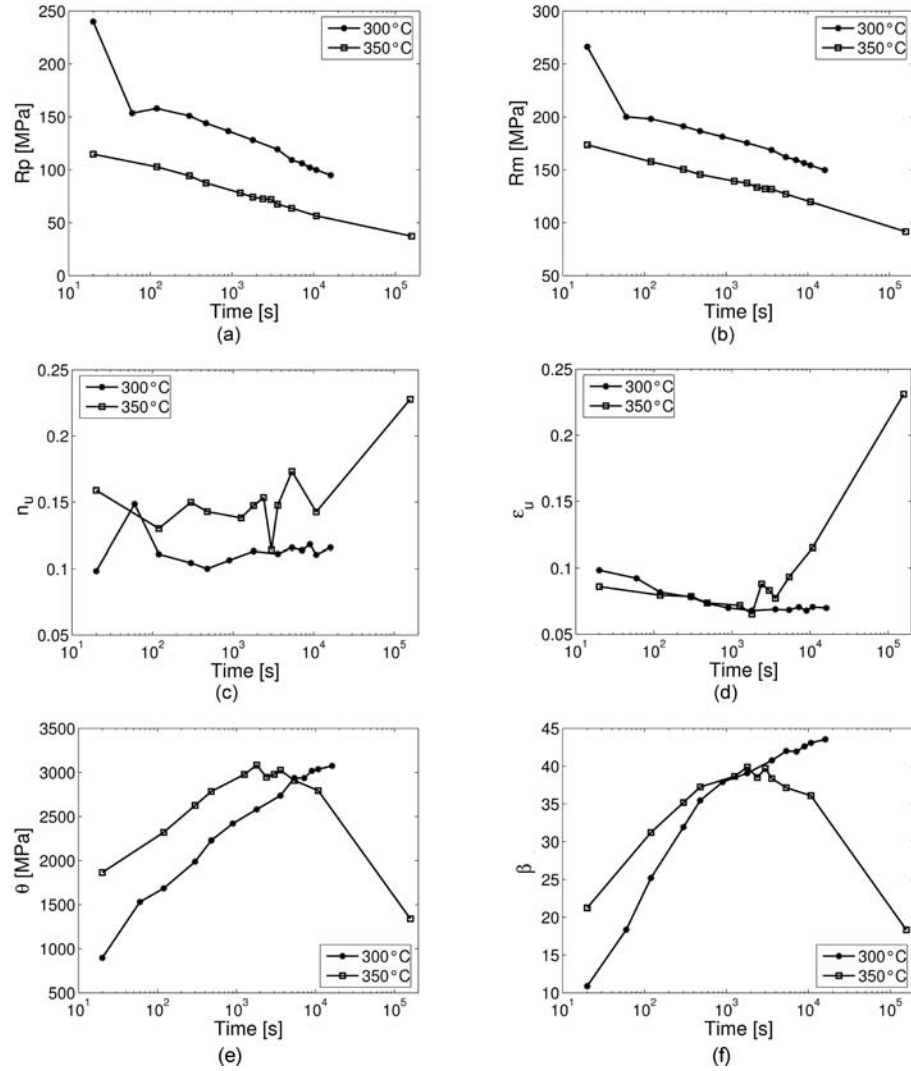


Figure 6.12. Evolution of the tensile properties as a function of the duration of heat treated 300°C and 350°C from T6 state for alloy number 1. (a) Yield strength R_p ; (b) ultimate tensile strength R_m ; (c) strain hardening exponent at necking n_u ; (d) true strain at the onset of necking ε_u ; (e) θ strain hardening parameter; (f) β strain hardening parameter.

two effects play a role: the dissolution of precipitates which takes place very rapidly and the slow coarsening of the precipitates. After only a few minutes of heat treatment, the strength drastically drops comparing to the initial strength of base alloy number 1 that is equal to 255 MPa. This is due to the partial dissolution of the fine hardening precipitates typical of the T6 state. After a longer heat treatment time, the strength decreases slowly for the 300°C and 350°C heat treated samples. This slower decrease in strength is due to the precipitates coarsening. A transition in the evolution of the strain hardening parameters θ and β occurs after some time of heat treatment. This time depends on the heat treatment temperature and is of about 30 minutes for the 350°C heat treated sample. After that transition, the strain hardening parameters θ and β drop while the strain hardening exponent n_u and the deformation at necking ε_u increase. This transition will be attributed in chapter 8 to the loss of coherency for very large precipitates.

Hardness measurements have been performed on samples of base alloy number 2 heat treated at 200°C, 250°C, 300°C, 350°C and 400°C. The trends are similar to those shown in Figure 6.12 for the yield strength. The results on alloy number 2 will only be used for the validation of the microstructural and yield strength models and will be presented in the corresponding chapter (see Figure 7.6).

6.2 Microstructural characterization of the welds

6.2.1 Grain size evolution

Figure 6.13 shows the evolution of the grain size in a friction stir weld on a transverse section from the base material to the nugget trough the heat affected zone. Image analysis was not systematically performed since the size of the grains in the base material vary a lot (see Figure 6.13(a)). Nevertheless, some trends are worth commenting:

- The heat affected zone seems to involve a slight grain growth (see Figure 6.13(b)). The effect of this small grain growth on the strength in this zone is certainly much smaller than the effect of the fine precipitates dissolution and coarsening.
- The grains in the TMAZ are elongated in the deformation direction (see Figure 6.13(c)). The size of these grains does not appear affected by the deformation process.

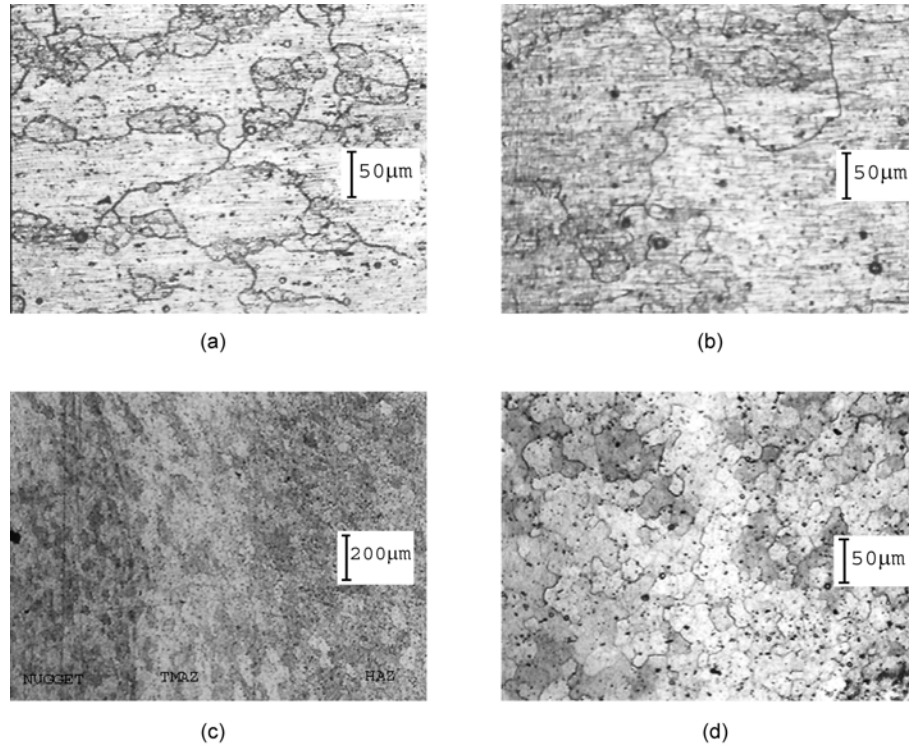


Figure 6.13. Observation of the grains by optical microscopy in a weld with welding parameters 500 mm/min and 2000 rpm: (a) base material; (b) heat affected zone (HAZ); (c) nugget-TMAZ-HAZ transition; (d) nugget.

- The weld nugget has very fine and equiaxed grains (see Figure 6.13(d)). There is thus a clear evidence of recrystallization in that zone. The strength of the weld nugget could be slightly increased by this grain size reduction but this effect is certainly smaller than the effect of the fine precipitates dissolution. It will be shown further that the yield strength and the hardness of the nugget are similar to that of the TMAZ which confirm the negligible effect of the grain size on the yield strength and hardness of the material.

6.2.2 Precipitates only affected by large strains in the weld center

As mentioned in chapter 2, the $\alpha(\text{AlFeMnSi})$ precipitates do not dissolve during welding since their temperature of dissolution is far above the temperature reached during the friction stir welding process. These

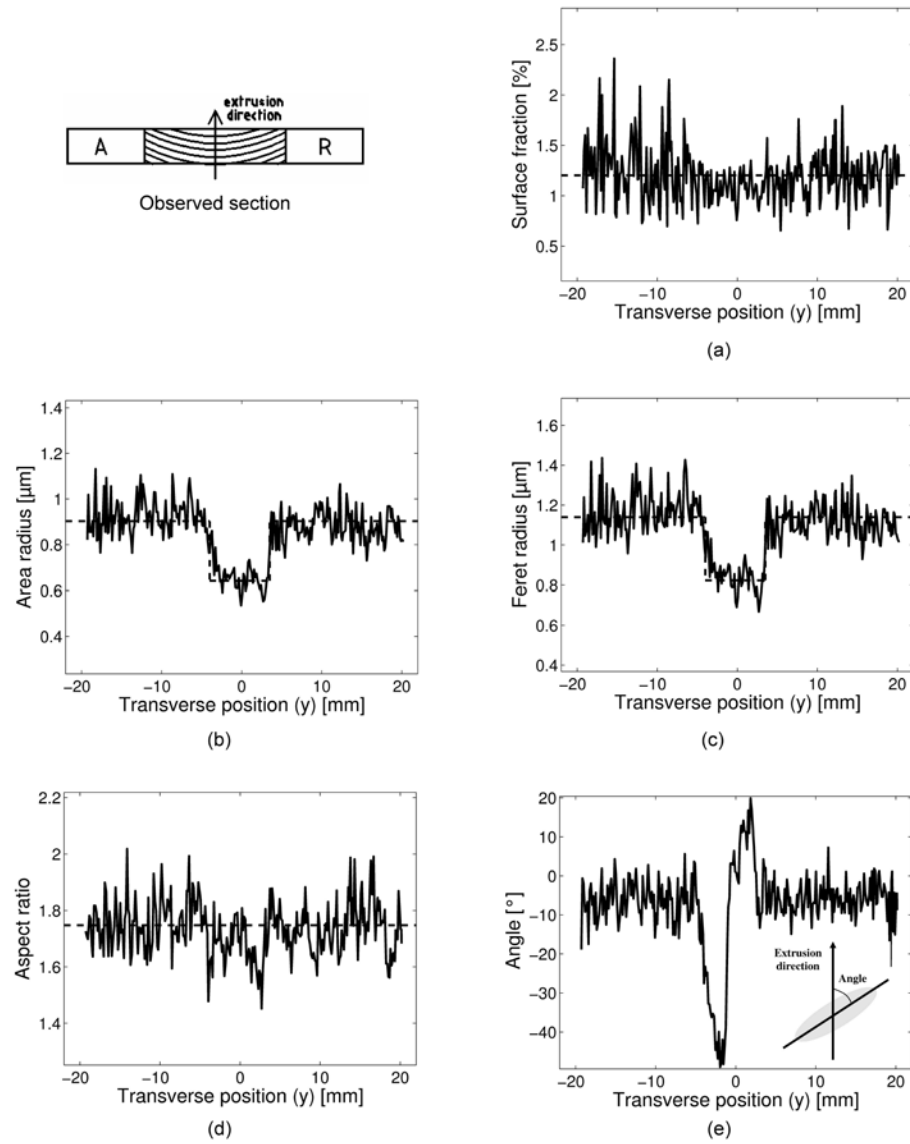


Figure 6.14. Evolution of the size and proportion of α precipitates in the mid-thickness plate section for a weld with welding parameters 1000 mm/min and 1000 rpm: (a) surface fraction; (b) area radius; (c) Feret radius; (d) aspect ratio; (e) angle with the extrusion direction (which is also the welding direction) of the ellipse that best fits a given precipitate. Scheme on top of Figure: marks left by the tool on the weld top surface. This distribution is the result of the analysis of 283 SEM images.

phases are fragmented in smaller particles in the central zone of the weld due to the high local deformation.

Figure 6.14 shows the results of the analysis of 283 SEM images of the α (AlFeMnSi) precipitates in the mid-thickness plate section (see scheme in Figure 3.4) for weld number "5" (1000 mm/min and 1000 rpm). Table 6.4 summarizes these results. The following observation can be done:

- The size of the precipitates is drastically reduced in the weld center for $-4 \text{ mm} < y < 3.5 \text{ mm}$ corresponding to the TMAZ width at mid-thickness (see Figure 6.14(b,c)). The particle size seems to be slightly lower on the retreating side than on the advancing side of the weld.
- The surface fraction of precipitates (see Figure 6.14(a)) is almost not influenced by the process since these phases do not dissolve at the temperatures attained during the welding. The slight decrease of the surface fraction in the central zone of the weld can be explained by the lower size of the precipitates, hence some very small particles could be under the detection limit.
- The shape ratio is almost unaffected by the welding process (see Figure 6.14(d)). A slight decrease of the precipitates aspect ratio in the TMAZ can be explained by the rotation of particles that could change the observed section of the precipitate.
- Interestingly the orientation of the precipitates with respect to the extrusion direction follows the marks left by the tool on the weld surface (see scheme on top of Figure 6.14 and Figure 6.14(e)). Hence the orientation angle is negative on the advancing side of the weld and positive on the retreating side of the weld.

6.2.3 Precipitates affected by the thermal cycles

(a) TEM study

Figure 6.15 shows the evolution of the fine hardening precipitates in friction stir welds observed by transmission electron microscopy (TEM). No TEM observations were performed in the TMAZ. As mentioned earlier the base material contains a high density of fine hardening precipitates. Comparing Figure 6.15(a) and (b), we see that the heat affected zone has larger and less precipitates than the base material. The section of the precipitates seem to be more rounded than rectangular which leads

Precipitate dimensions	Undeformed zone	Deformed zone
Surface fraction	1.2%	1.1%
Aspect ratio	1.75	1.67
Area radius	0.9 μm	0.6 μm
Feret radius	1.1 μm	0.8 μm
Largest axis of the closest ellipse	2.1 μm	1.5 μm
Smallest axis of the closest ellipse	1.2 μm	0.9 μm

Table 6.4. α phases analysis for a friction stir weld with welding parameters 1000 mm/min and 1000 rpm.

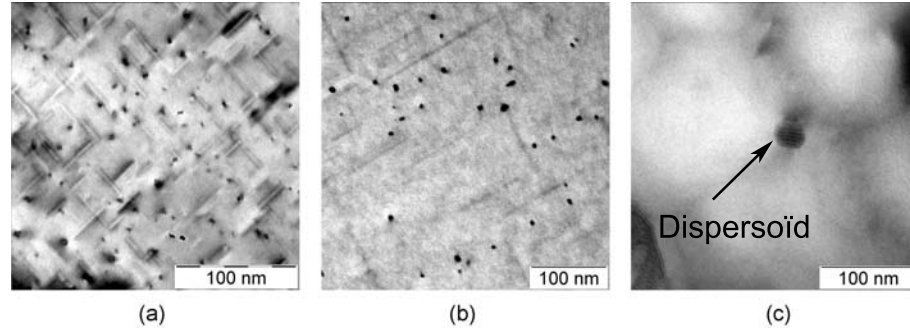


Figure 6.15. Fine precipitation evolution in friction stir welds: (a) base material; (b) heat affected zone (HAZ) of a weld with welding parameters 200 mm/min and 1000 rpm (large β' precipitates); (c) nugget of a weld with welding parameters 1000 mm/min and 1000 rpm (no β' precipitates observed, dispersoids are not dissolved). $[100]_{Al}$ zone axis.

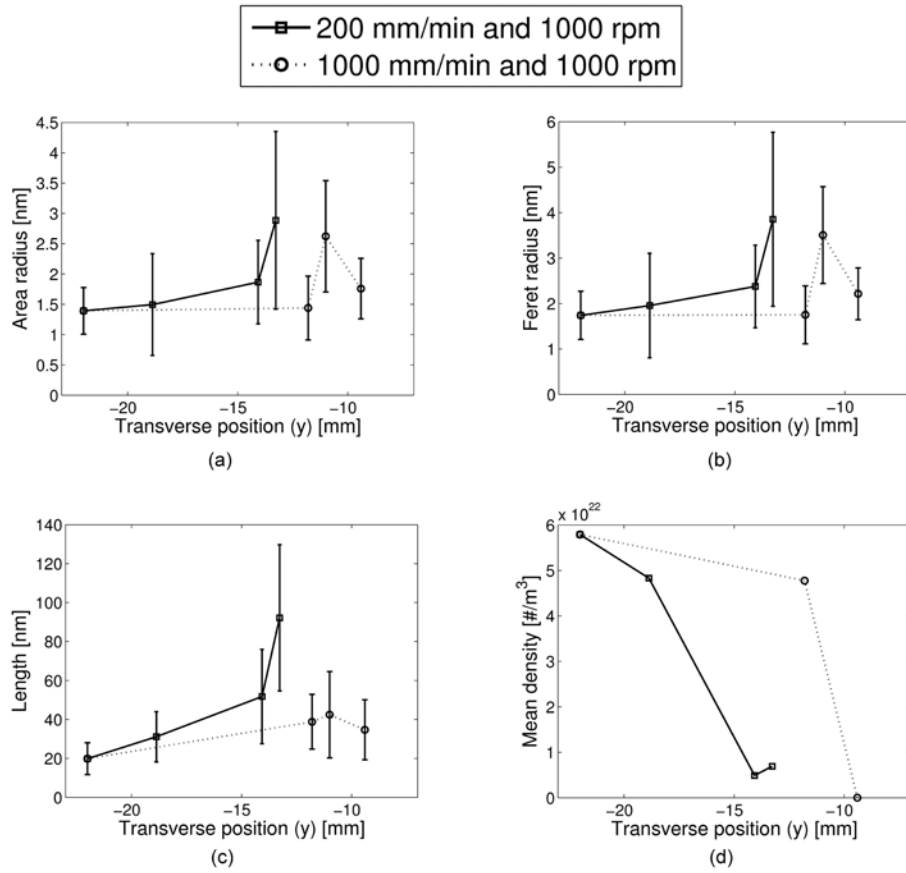


Figure 6.16. $\beta'' - \beta'$ precipitation size and density evolution in the heat affected zone (HAZ) on the advancing side of two friction stir welds: (a) area radius; (b) Feret radius; (c) length; (d) mean density (number of precipitates per unit volume). Error bars indicate the standard deviation on the mean values.

to the conclusion that these precipitates are β' precipitates rather than B' or Q' precipitates. For modeling purposes, it is interesting to note that the β' precipitation in the HAZ is homogeneous and seems thus not affected by the presence of the dispersoids or dislocations. Nevertheless, we can speculate that the fact that precipitation is not affected by the presence of dislocations might not be the case in the TMAZ where high deformations increase the dislocation density favoring heterogeneous precipitation. Fine precipitates are not observed in the nugget (see Figure 6.15(c)) and apparently the dislocation density is lower. The observation of a dispersoids, see Figure 6.15(c), lead to the conclusion that the dispersoids are not dissolved during welding.

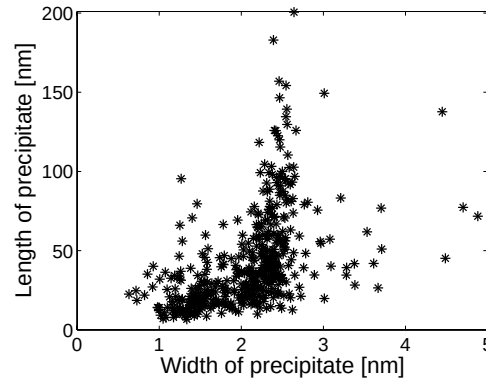


Figure 6.17. Length of all precipitates with the long axis in the plane of observation as a function of their width.

The evolution of the radii, length and density of the fine precipitates as a function of the position along the direction transverse to the welding line are presented in Figure 6.16 for a hot and a cold weld. Note that only one TEM sample is extracted for a given zone. The number of precipitates per unit volume is thus obtained from only one EELS (Electron Energy Loss Spectroscopy) thickness prediction. Considering also that the number of precipitates observed is very low, slight inhomogeneities in the precipitate distribution will significantly affect the measured number of precipitates per unit volume. Hence, the mean density results of Figure 6.16(d) should be treated with care.

The size and density evolutions of Figure 6.16 confirm the qualitative observations of Figure 6.15: the precipitates size increases in the heat affected zone (HAZ) and the precipitate density decreases. Note however that the precipitates size decreases nearer to the weld center due to the dissolution of the precipitates initially present in the base material. Indeed, the temperatures reached close to the weld center favor the dissolution of particles rather than their coarsening.

β' precipitates are semi-coherent while β'' precipitates are coherent with the aluminum matrix (see Table 2.1, page 10). The precipitates preferably grow in the direction of the rod axis due to the low coherency of the β' precipitates in this direction. Figure 6.17 shows that above a certain width (about 2.5 nm) the length of the precipitate grows much faster than the width. This makes us conclude that the β'' - β' transformation has been over-passed in these friction stir welds. Indeed, if the

length of some precipitates has more rapidly grown than their width it is a hint that the precipitates observed have most probably a preferable direction of growth and hence are β' rather than β'' .

Figure 6.16 shows that the precipitates length within the heat affected zone are larger in a hot weld (200 mm/min and 1000 rpm) than in a cold weld (1000 mm/min and 1000 rpm). The fact that the precipitates radius is relatively similar in the two welds even though the hot weld has much longer precipitates confirms that the heat affected zone is composed of β' precipitates in both welds. The transition between β'' and β' precipitates is identified at a width of the precipitates with the long axis in the plane of observation of about 2.5 nm in Figure 6.17. This transition width seems to be over-passed in both welds. Indeed, the width of the precipitates with the long axis in the plane of observation is in between the area and the Feret radius of the precipitates observed end-on. The maximum observed area and Feret radius of the precipitates observed end-on in the HAZ of both welds are respectively of about 3 and 4 nm.

(b) DSC study

β'' dissolution and β' precipitation. Figure 6.18(a) presents the typical DSC curves observed in the HAZ and weld nugget of a friction stir weld made of alloy 6005A-T6 in comparison to the base material DSC curve.

HAZ MID-LEVEL HARDNESS. *β'' precipitates have probably started to dissolve, some have been transformed in β' .* The surface of the β' DSC precipitation peak is unchanged when compared to the base material peak. The schematic curves in Figure 6.18(c) explains how we conclude that both β'' dissolution and β' precipitation occur in the "HAZ mid-level hardness" region of the weld.

HAZ LOW HARDNESS. *No β'' nor GP zones are present and β' have precipitated.* There is no dissolution peak at 260°C, which means that the β'' precipitates and GP zones were absent in that region of the weld. A precipitation peak at that temperature corresponding to the precipitation of magnesium and silicon in solid solution into β'' precipitates during the DSC cycle is observed which proves the existence of a solid solution in that region of the weld. This confirms the observation already made when presenting Figure 6.9(b) that no natural aging occurs if the solid solution concentration is not sufficient. Furthermore, a high supersaturation in vacancies favors the precipitation of GP zones and that supersaturation in vacancies is limited when the cooling is not fast

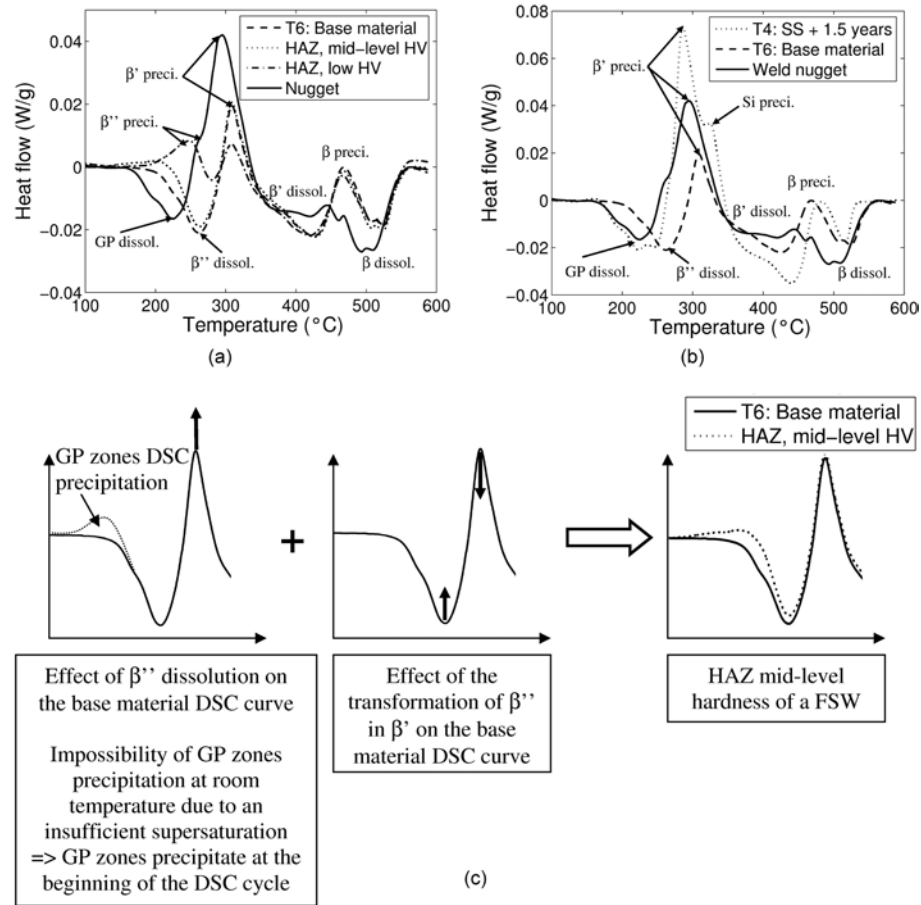


Figure 6.18. DSC curves in a friction stir weld for weld number 5 (1000 mm/min and 1000 rpm): (a) comparison of the DSC curves of the different zones of the weld (HV = Vickers hardness); (b) comparison of the DSC curve of a nugget sample with the DSC curves of a base material T6 sample and a T4 sample; (c) schematic explaining why the evolution of the DSC curve in the heat affected zone (mid-level hardness) can be explained by the dissolution of β'' precipitates and the transformation of β'' in β' precipitates in that zone of the weld.

enough since the material has more time to reach its thermodynamic equilibrium. The second peak in the DSC curve corresponds to the β' precipitation. The area of that peak being lower than in the base material, it means that β' have already precipitated. An estimation of the relative volume fraction of β' present in that region can be obtained by comparing the area of the β' DSC precipitation in the T6 base material to the area of the β' peak in the HAZ low hardness sample: 81% of the β' that can precipitate has precipitated in that region of the weld. The remaining 19% precipitate during the DSC heating. This percentage can be seen as the amount of solid solution which is the origin of the first precipitation peak during a DSC cycle.

NUGGET. GP zones formation. The DSC curve associated to sample extracted from the weld nugget shown in Figure 6.18(a) presents a large first dissolution peak attributed to GP zones dissolution. GP zone have thus precipitated in the weld nugget during natural aging. The rest of the DSC curve is very different than the other curves and will be commented here below.

$\beta(\text{Mg}_2\text{Si})$ precipitation in the weld center. Figure 6.18(b) compares the DSC curves of a T4 sample (solution heat treated and naturally aged for 1.5 years), a T6 sample (base alloy number 1) and a sample extracted from the nugget of a friction stir weld (naturally aged 1 year). The following analysis of these curves aims at showing that $\beta(\text{Mg}_2\text{Si})$ precipitation might have taken place in the weld nugget.

FIRST DISSOLUTION PEAK. The first dissolution peak in the T4 sample and in the weld nugget correspond to the dissolution of the GP zones nucleated during natural aging. The peak area of the T4 sample is larger than the peak area of the weld nugget. This is a first hint that extra precipitation has occurred in the weld nugget (probably of small $\beta(\text{Mg}_2\text{Si})$ precipitates), capturing part of the magnesium and silicon that would normally precipitate in GP zones. Note that the difference could be slightly exaggerated due to the potential dissolution of the coarse $\beta(\text{Mg}_2\text{Si})$ precipitates in the T4 sample during the solution heat treatment which would artificially increase the Mg and Si available for natural aging.

FIRST PRECIPITATION PEAK. The first precipitation peak in the T4 sample and in the weld nugget correspond to the precipitation of β'' and β' precipitates. These precipitates can transform from one to the other.

The shoulder in the weld nugget sample at 260°C corresponds to the transition between β'' and β' precipitation while the peak at 290°C – 300°C corresponds to β' precipitation. The T4 sample has a shoulder at about 320°C which can be attributed to the precipitation of Si phases that have probably been dissolved when solution heat treating the alloy. Actually, these precipitates dissolve during a DSC treatment at 520°C (at 10°C/min) while a solution heat treatment implies to hold the sample at 530°C for 1 h. Nevertheless, a clear difference is observed between the β'' - β' precipitation peak area of the T4 sample and the weld nugget sample. This is the second hint of $\beta(\text{Mg}_2\text{Si})$ precipitation in the weld nugget.

$\beta(\text{Mg}_2\text{Si})$ PRECIPITATION AND DISSOLUTION PEAKS. The weld nugget samples exhibits a lower $\beta(\text{Mg}_2\text{Si})$ precipitation "peak" (hidden by the peak convolution) than the T4 and T6 samples. Furthermore, the final dissolution peak, attributed to $\beta(\text{Mg}_2\text{Si})$ dissolution, exhibits a lower dissolution temperature in the weld nugget compared to the T4 sample. Moving in the quasi-binary diagram Al-Mg₂Si toward a higher aluminum content causes a decrease of the dissolution temperature. Hence, considering that no alloying element has been lost during the friction stir welding process, this means that precipitation has occurred in the weld nugget. The earlier hints as well as the surface of the $\beta(\text{Mg}_2\text{Si})$ dissolution peak suggest that this precipitation is most probably a precipitation of $\beta(\text{Mg}_2\text{Si})$.

Figure 6.19 shows squared-section platelets of small $\beta(\text{Mg}_2\text{Si})$ are present in the weld nugget. Nevertheless, a more systematic observation of these precipitates is needed to completely confirm the presence of $\beta(\text{Mg}_2\text{Si})$ precipitates in the weld nugget. Gallais [50] also found indications of the presence of $\beta(\text{Mg}_2\text{Si})$ precipitates in weld nuggets of AA6056 friction stir welds. Figure 6.18(a) shows that $\beta(\text{Mg}_2\text{Si})$ precipitation does not occur in the heat affected zone (HAZ) since there the end of the DSC curve is identical to the base material curve.

Comment. The possibility of $\beta(\text{Mg}_2\text{Si})$ precipitation in the weld center is a reasonable explanation of the shape of the weld nugget DSC curve. Nevertheless, the temperatures attained during welding might also cause the precipitation or the growth of Si rich particles in the weld center. The only definitive conclusions from our experimental results are that precipitation occurs in the weld center and that this precipitation decreases the amount of solute available for post-aging (natural aging or post-weld heat treatment).

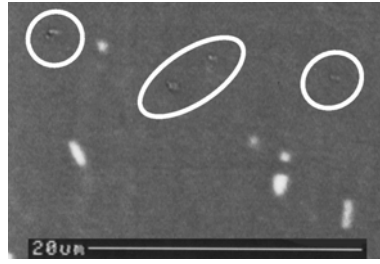


Figure 6.19. $\beta(\text{Mg}_2\text{Si})$ squared-section platelets in a friction stir weld nugget.

6.2.4 Conclusions of the microstructure characterization

Table 6.5 presents the conclusions of the microstructural characterization of friction stir welds. The DSC curve analysis conclusions corroborate the TEM observations of the fine precipitates. Our analysis of the microstructure evolution in friction stir welds is in agreement with the literature results presented in chapter 2. TEM observations have shown that the hotter weld (low advancing speed) show much larger β' precipitates in the heat affected zone compared to the colder welds (higher advancing speed).

6.3 Local mechanical properties of the welds

In this section, the mechanical characterization of the friction stir welds will essentially focus on the local tensile properties and hardness within the joints. The overall transverse tensile properties of the welds will be presented in chapter 9. The discussions presented in this chapter are only the ones that can be made based on the experimental results. Model development presented in chapters 7 and 8 will help further understand some aspects of the local mechanical properties distribution.

6.3.1 Results

(a) Hardness and yield strength of friction stir welds

Hardness evolution. The hardness distribution is the easiest local mechanical property to measure in a welded sample. It provides also very local measurements, more local than the tensile properties obtained by micro-tensile tests which give an average value over the thickness. A hardness map in a transverse section gives the shape of the thermally affected zone as shown in Figure 6.20(a,b) for different welding condi-

Weld zone	HV1 after NA	Microstructure
Base material (BM)	105 HV1	- high density of β'' - no Mg and Si in SS
Heat affected zone (HAZ)	80 – 90 HV1	- coarse α unaffected - less β'' than in BM - some Mg and Si in SS - some β'
Heat affected zone (HAZ)	60 – 70 HV1	- coarse α unaffected - no β'' - lot of β' - some Mg and Si in SS - no GP zones after NA
Weld center (nugget and TMAZ)	70 HV1	- small grains - low dislocation density - dispersoids not dissolved - coarse α broken up - no β'' nor β' - GP zones after NA - some $\beta(\text{Mg}_2\text{Si})$

Table 6.5. Summary of the conclusions of the microstructural characterization of friction stir welds (HV1=Vickers hardness 1 kg, SS=solid solution, NA=natural aging).

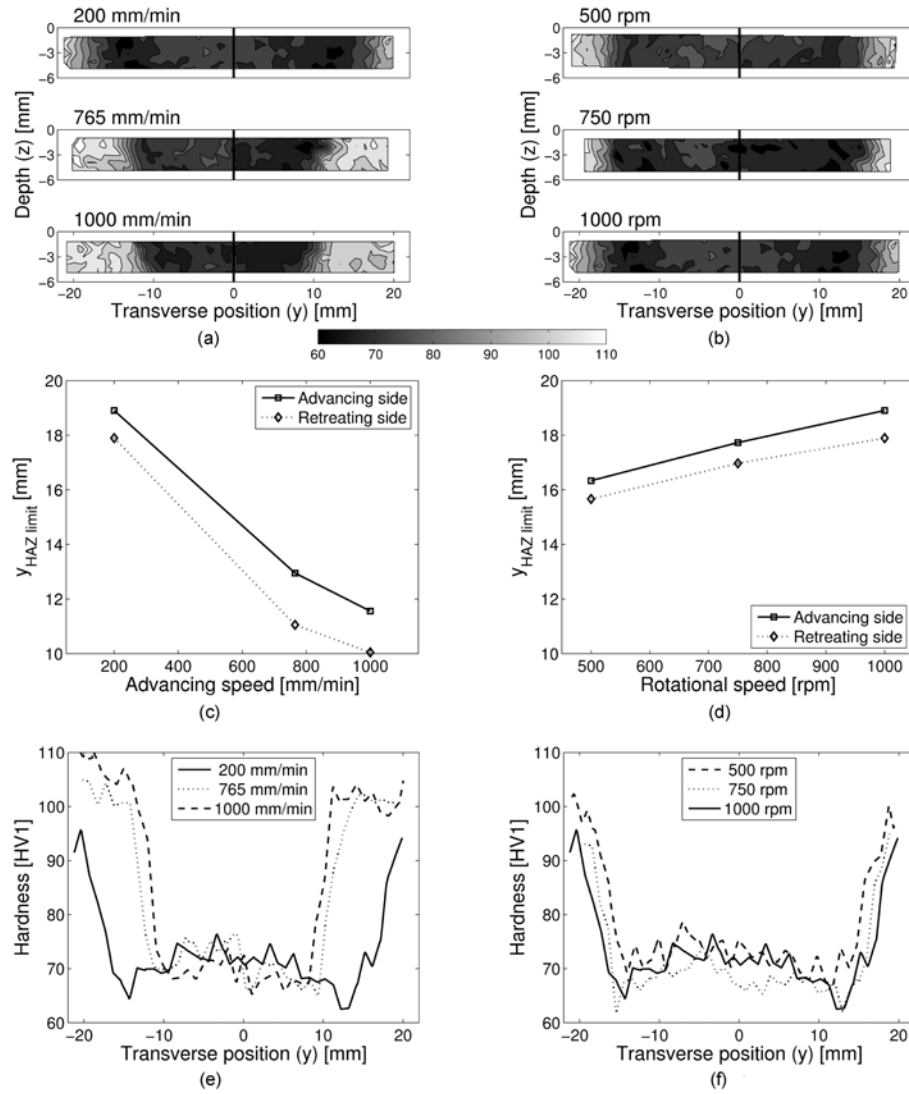


Figure 6.20. Hardness variation in transverse sections of friction stir welds at (a,c,e) constant rotational speed equal to 1000 rpm and (b,d,f) constant advancing speed equal to 1000 mm/min: (a,b) maps along transverse sections; (c,d) distances from the weld centerline of the minimum hardness zones (taken at 85 HV); (e,f) hardness profile at mid-depth.

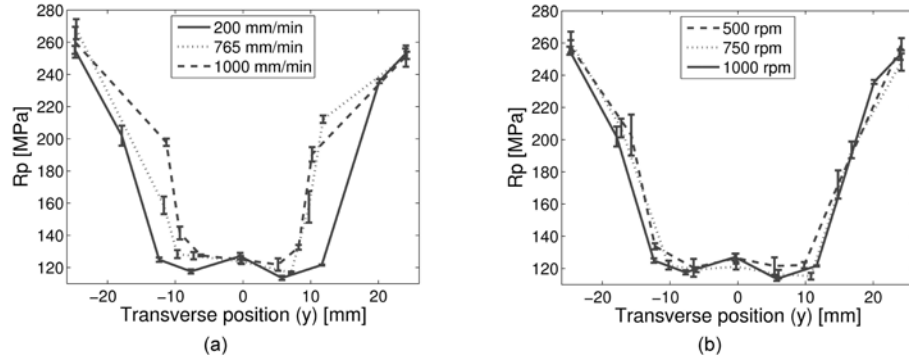


Figure 6.21. Evolution of the yield strength defined at 0.2% offset from linearity as a function of the position along a transverse section of the welds: (a) effect of the advancing speed for a rotational speed equal to 1000 rpm ;(b) effect of the rotational speed for an advancing speed equal to 200 mm/min. Errorbars indicate the standard deviation on the mean yield strength value from the measurements of 3 samples for each specific weld zone.

tions. When the advancing speed increases the inclination of the HAZ boundary increases. Figure 6.20(c,d) and (e,f) show that the width of the low hardness zone decreases with increasing advancing speed and decreasing rotational speeds. A clear asymmetry is observed with the position of the hardness drop which is closer to the weld centerline on the retreating side of the welds than on the advancing side of the welds. The difference increases with decreasing advancing speed and increasing rotational speed. The hardness of the weld is slightly lower on the retreating side of the weld. Figure 6.20(e,f) shows that the hottest welds (200 mm/min) present a hardness minimum in the heat affected zone lower than in the weld center. This difference is not observed in the cold welds.

Yield strength evolution The yield strength distribution throughout the weld zones has been measured with micro-tensile tests machined within the different zones of the welds as explained in chapter 3, section 3.3.2. Results are presented in Figure 6.21. Figure 6.22 shows that the minimum yield strength (always located in the heat affected zone or very close to the thermo-mechanically affected zone) increases with increasing advancing speed and decreasing rotational speed. The advancing side of the welds have generally a lower minimum yield strength than the retreating side.

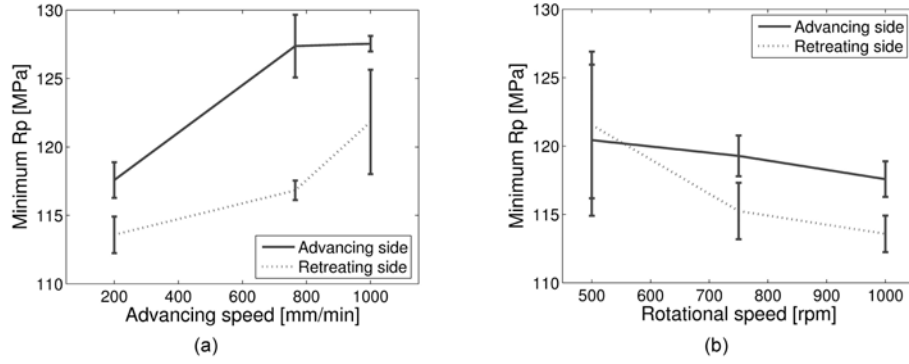


Figure 6.22. Comparison between the minimum yield strength of the micro-tensile specimens on the advancing and on the retreating side of the weld: (a) effect of the advancing speed for a rotational speed equal to 1000 mm/min; (b) effect of the rotational speed for an advancing speed equal to 200 mm/min.

(b) Local strain hardening capacity in friction stir welds

Figures 6.23 and 6.24 show the evolution of various index associated to the strain hardening capacity as a function of the position along the transverse section of the weld for different welding conditions.

The strain hardening exponent at the onset of necking n_u and the true strain at the onset of necking ε_u should theoretically be equal in order to fulfill the Considere criterion. Figures 6.23(b,c) and 6.24(b,c) show significant differences for the samples extracted from the heat affected zone and from the thermo-mechanically affected zone. The reason is that the Considere criterion is valid only for homogeneous materials. Hutchinson *et al.* [64] showed that tiny geometrical or material inhomogeneities causes premature necking. Figure 6.25 shows that the ratio of the smallest edge thickness to the largest edge thickness l_S/l_L (defined in section 3.3.2), considered as being a good parameter to study the inhomogeneity of the samples, is much lower than 1 in the zones where the Considere criterion is clearly not verified. Furthermore, the very small section of these micro-tensile samples (0.8 mm x 4 mm) increases the machining imprecision. Hence, the error bar on ε_u has been enlarged when the Considere criterion is not verified. This was done as explained in Figure 6.26(b). Figure 6.26 shows two extreme situations. Figure 6.26(a) shows a sample for which the Considere criterion is properly fulfilled. In that case no correction is made to the error bar. Figure 6.26(b) shows a case where premature necking occurs. In that case the value of ε_u could be higher than the 0.05 measured by up to a value equal to 0.09.

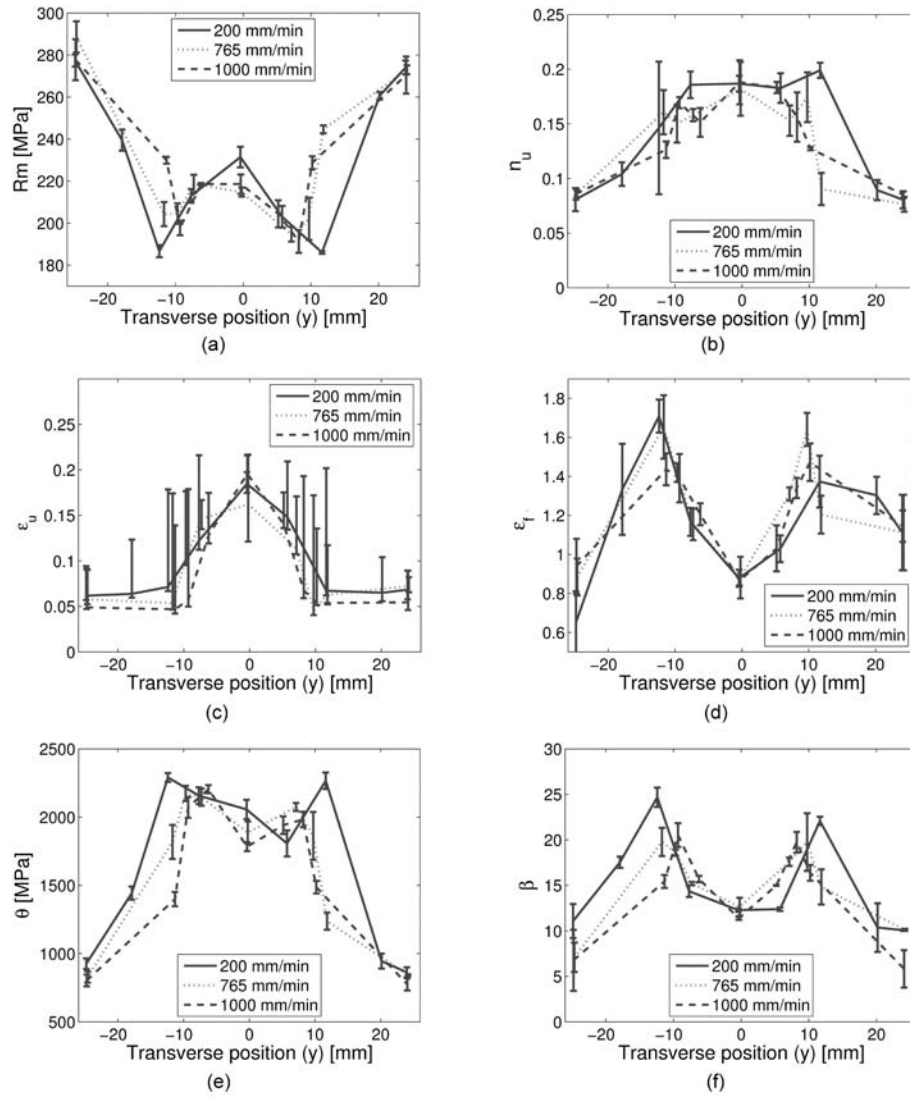


Figure 6.23. Variation of the tensile properties as a function of the position along a transverse section of the weld for different advancing speeds and a rotational speed equal to 1000 rpm: (a) Ultimate tensile strength R_m ; (b) strain hardening exponent at the onset of necking n_u ; (c) true strain at the onset of necking ϵ_u ; (d) true fracture strain ϵ_f ; (e) θ strain hardening parameter; (f) β strain hardening parameter.

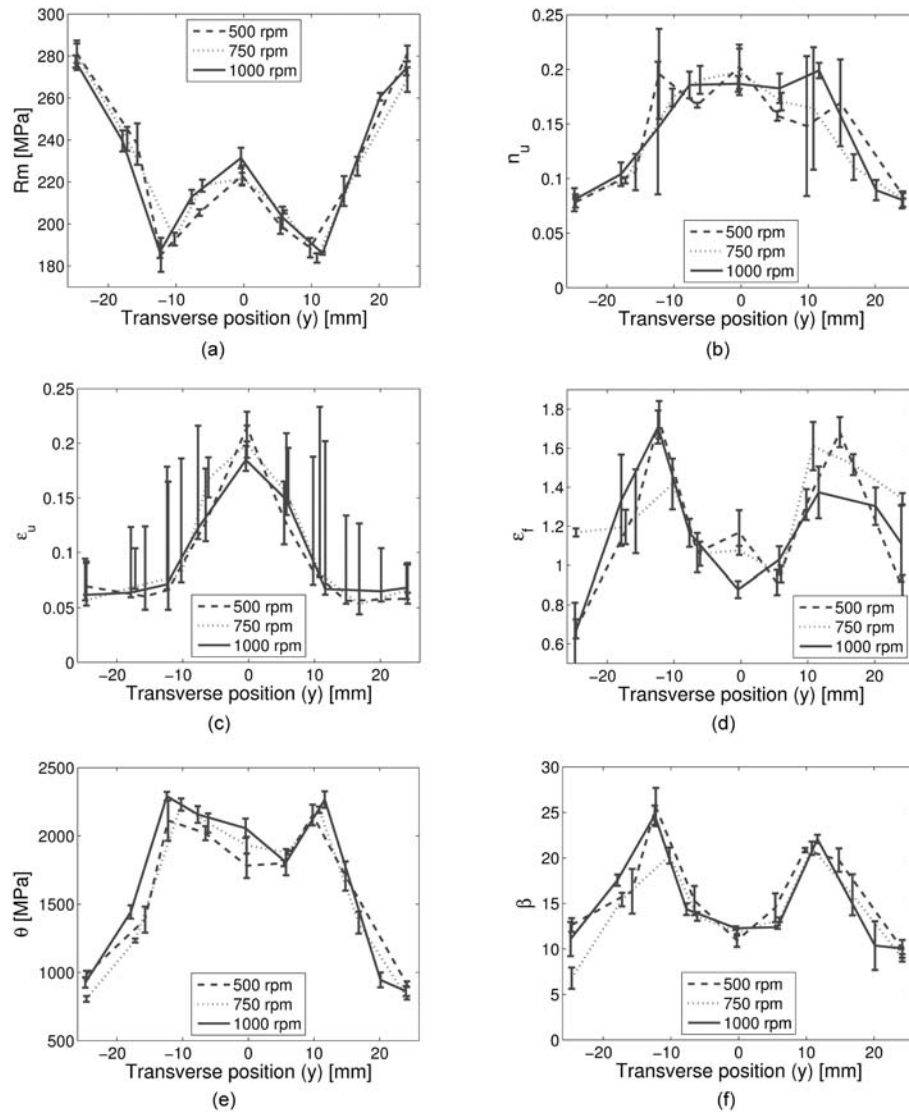


Figure 6.24. Variation of the tensile properties as a function of the position along a transverse section of the weld for different rotational speeds and an advancing speed equal to 200 mm/min: (a) Ultimate tensile strength R_m ; (b) strain hardening exponent at the onset of necking n_u ; (c) true strain at the onset of necking ϵ_u ; (d) true fracture strain ϵ_f ; (e) θ strain hardening parameter; (f) β strain hardening parameter.

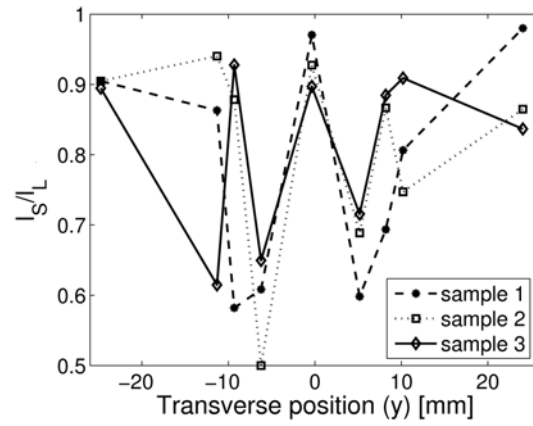


Figure 6.25. Ratio describing the inhomogeneities in the micro-tensile samples (ratio of the smallest edge thickness to the largest edge thickness) of a weld with welding parameters 1000 mm/min and 1000 rpm.

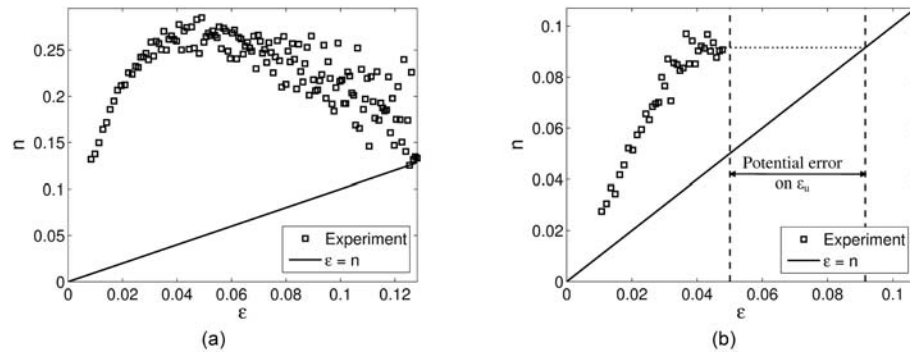


Figure 6.26. Evolution of the strain hardening exponent n as a function of strain ε : (a) sample for which the Considere criterion is verified; (b) sample showing premature necking.

The ultimate tensile strength in the weld center (see Figures 6.23(a) and 6.24(a)) is higher than the ultimate tensile strength in the HAZ while the magnitude of the yield strength are similar. One can notice that the effect of the welding parameters on the ultimate tensile strength follows the evolutions of the strain hardening parameters θ and β with the welding conditions.

The variation of θ is not symmetric with respect to the weld centerline close to the weld center. The retreating side quite systematically exhibits a lower θ . This trend is more marked for extreme advancing speeds. The maximum of both θ and β is located in the heat affected zone.

Figure 6.23 shows no significant effect of the advancing speed on the strain hardening exponent at the onset of necking. The HAZ has a slightly higher true strain at the onset of necking when the advancing speed decreases. Figure 6.24 shows that the rotational speed only affect the nugget ($y = 0$) resistance of material to necking. A higher rotational speed leads to a lower true strain at the onset of necking.

(c) Local strain to fracture of friction stir welds

Figures 6.23(d) and 6.24(d) show the evolution of the local fracture strain for different welding conditions. The true fracture strain is the highest in the heat affected zone. This is interesting since it is the weakest zone of the weld and hence the region where the deformations will localize when loading a welded sample. The rotational speed has a clear effect on the true fracture strain in the nugget center ($y = 0$). The true fracture strain of the weld nugget decreases when the rotational speed increases. The asymmetry in the HAZ true fracture strain seems to depend on the welding parameters. No definitive comparison can be given with the samples extracted from the welds with different rotational speeds but the effect of the advancing speed is more obvious. The heat affected zone on the advancing side of the weld has generally the lowest true fracture strain.

(d) Effect of a post-welding heat treatment

The post-welding heat treatment (PWHT) is a way for estimating the solid solution content of a region of the weld before the PWHT. Like DSC cycles, PWHT response are the signature of the microstructure. We are not interested here in finding ways to optimize the properties of the weld. The post-welding heat treatment will help discuss some of the

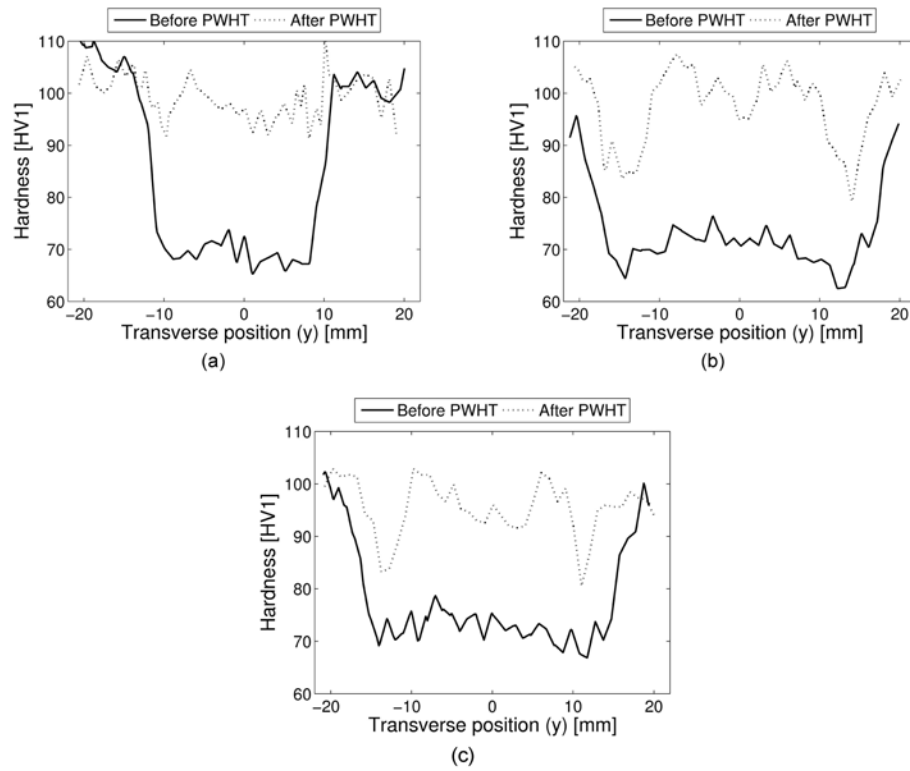


Figure 6.27. Evolution of the hardness profiles resulting from post-welding heat treatment at 185°C for 6 h for welds with welding parameters: (a) 1000 mm/min and 1000 rpm; (b) 200 mm/min and 1000 rpm; (c) 200 mm/min and 500 rpm.

results established by microstructural characterization. A post-welding heat treatment at 185°C for 6 h [28] has been applied to our friction stir welds. Figure 6.27 shows the hardness evolution caused by the post-welding heat treatment of three friction stir welds with extreme welding conditions.

The following trends can be observed:

- The base material is unaffected by the PWHT.
- The hardness at the outer side of the HAZ is recovered after a PWHT. This is not the case at the hardness minimum.
- The hardness of the weld nugget is nearly recovered.
- The zone of low hardness remain larger on the advancing side of the welds than on the retreating side of the welds after a PWHT.

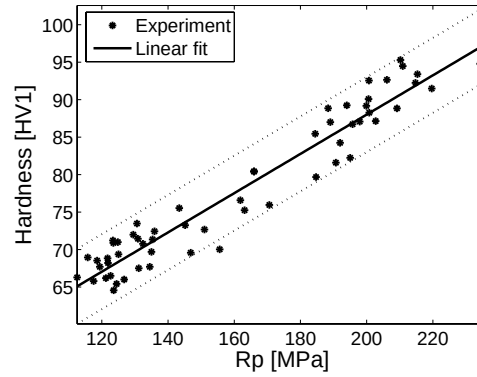


Figure 6.28. Variation of hardness as a function of yield strength in the heat affected zone. Linear fit according to expression (6.1).

- When the advancing speed decreases, the capacity to recover hardness after a PWHT at the hardness minimum decreases.

6.3.2 Discussion

(a) Linking the hardness and the yield strength

There is no apparent difference between the yield strength distribution shown in Figure 6.21 and the hardness distribution shown in Figure 6.20(e,f). The hardness and the yield strength are related by the following least square linear fit established on the hardness measurements of the micro-tensile specimens of the HAZ³ prior to the tensile test (see Figure 6.28):

$$HV = 35.6 + 0.26 * R_p \quad (6.1)$$

Note that the hardness is influenced by the degree of strain hardening. Hence, expression (6.1) is less accurate in the weld center where the strain hardening capacity is larger than in the base material.

(b) Linking the microstructure evolution with the hardness and local yield strength distribution

Yield strength of the weld center. The yield strength in the weld center is similar to the yield strength of a naturally aged sample (see

³It has been chosen to relate the hardness and the yield strength only in the HAZ since that zone of the welds exhibits the highest variations in hardness and yield strength.

Table 6.3, page 94) even though $\beta(\text{Mg}_2\text{Si})$ might have precipitated in the weld nugget, decreasing the amount of alloying elements available to generate GP zones. The grain size in the nugget is very low compared to that of the base material and the time of natural aging corresponding to the results of Table 6.3 (page 94) was smaller than the time left between the welding and the hardness and yield strength measurements reported in Figure 6.20 and 6.21. These effects probably explain the apparent contradiction of the $\beta(\text{Mg}_2\text{Si})$ precipitation in the weld center while the yield strength level in the weld nugget is equal to the level of a naturally aged sample.

Correlation with the DSC results. The marked drop in hardness and yield strength in the heat affected zone (HAZ) is the consequence of the partial dissolution and transformation of the hardening precipitates β'' into coarser β' precipitates. Figure 6.29 shows the evolution of the area of the first DSC dissolution peak as a function of the position along a transverse section of weld number "5" (1000 mm/min and 1000 rpm). The correlation with the hardness evolution is perfect: when the β'' precipitates dissolve in the weld, both the area of the first DSC dissolution peak and the hardness drop drastically. Note that this first dissolution peak should theoretically combine the dissolution of the GP zones and of the β'' precipitates but, in practice, very few GP zones will form in the weld center due to the fact that the supersaturation is not sufficient for GP zone precipitation when dissolution is partial. Hence the two dissolutions (GP zones and β'') can be considered as uncoupled in Figure 6.29. In the weld center, GP zone precipitation leads to an increase of the first DSC dissolution peak. The DSC first dissolution peak shows a minimum when only β' precipitates harden the material, since in that region no GP zones and no β'' are found (corresponds to plot "HAZ, low HV" in Figure 6.18(a)), hence no DSC first dissolution peak is observed).

General trends of the hardness evolution due to a PWHT. The evolution of the hardness with a post-welding heat treatment (PWHT) can be correlated with the microstructural evolution in the welds summarized in Table 6.5 (page 111). The trends of Figure 6.27 can be explained as follows.

- **Base material.** The base material is basically unaffected by the post-welding heat treatment. Figure 6.11 showed that the yield strength remains at a maximum for heat treatment lasting 6 hours

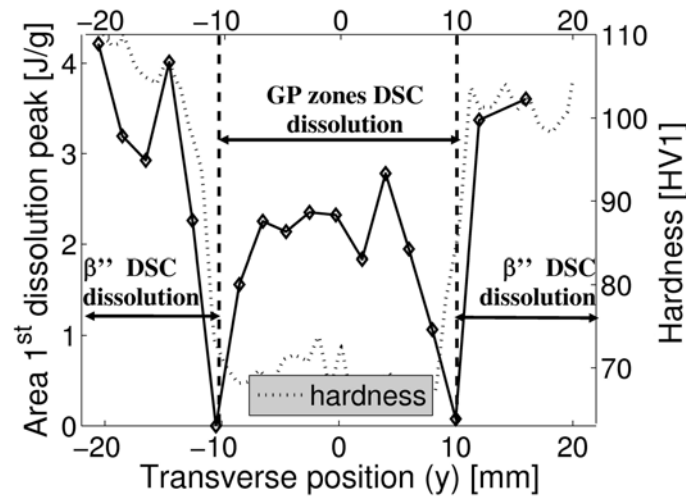


Figure 6.29. Area of the first DSC dissolution peak (β'' dissolution peak or GP zones dissolution peak) throughout weld number 5 (1000 mm/min and 1000 rpm), comparison with the hardness evolution.

at 180°C. Hence it is not surprising that the base material hardness is not sensitive to a post-welding heat treatment.

- Heat affected zone.** If the beginning of the hardness drop zone corresponds to a zone where the β'' precipitates have been partially dissolved, the hardness should be recovered by the PWHT. Hence the hardness level of that zone after PWHT should be higher than just after welding. This is what is observed in the three welds in Figure 6.27 which confirms the conclusions drawn from the analysis of the DSC results that some dissolution has occurred in the "HAZ, mid-level hardness". The β' precipitation leads to a decrease of the solid solution available for precipitation of β'' during the post-welding heat treatment, causing the drop of hardness after PWHT in the HAZ close to the weld center.
- Weld nugget.** The central zone of the weld has a high concentration in solid solution elements which lead to the formation of GP zones by natural aging. The GP zones constitute nucleation sites for the precipitation of hardening β'' precipitates during the PWHT increasing the PWHT hardness in the weld center. Nevertheless the hardness is not completely recovered. This confirms that another precipitation presumably of $\beta(\text{Mg}_2\text{Si})$ as explained earlier in section 6.2.3(b) has occurred leading to a decrease of the

magnesium and silicon in solid solution.

Effect of the welding parameters on the hardness evolution due to a PWHT. Figure 6.27(b) showed that the PWHT hardness of the HAZ in welds obtained with low advancing speed is much lower than the hardness of a weld with a higher advancing speed (see Figure 6.27(a)). Two effects can be invoked to explain this observation.

- TEM observations of the heat affected zones (HAZ) have shown that the β' precipitates are much larger in welds corresponding to a low advancing speed (see Figure 6.16). Large precipitates lead to lower hardening capacity than smaller ones. Hence, the mean radius after a PWHT (combination of the increased radius of the large precipitates and the radius of the precipitates that have nucleated during the PWHT) will be higher in the HAZ ("low hardness") for a weld with a lower advancing speed than for a weld with a high advancing speed.
- If the volume fraction of β' precipitates in the heat affected zone of a hot weld is higher than in a cold weld, then less solid solution is available for the precipitation of β'' during the post-welding heat treatment.

The rotational speed, for the low advancing speed in Figure 6.27(b) and (d) (200 mm/min), has no clear effect on the minimum PWHT hardness in the HAZ. Hence, the size and volume fraction of the β' precipitates in that zone should not be markedly affected by the rotational speed at least for an advancing speed equal to 200 mm/min.

Hardness drop in the HAZ zone. The drop in hardness or in yield strength in the heat affected zone is more important in the hottest weld. Two effects can cause this difference:

- Hot welds involve β' precipitates in the HAZ, see result of TEM measurements shown in Figure 6.16. These precipitates have even less hardening capacities if their size increases.
- If the volume fraction of β' precipitates is well higher in the hot weld (a conclusion of the PWHT results) then less solid solution is left for GP zones formation by natural aging.

(c) Strain hardening capacity of the weld zones

The ultimate tensile strength in the weld center is higher than the ultimate tensile strength in the HAZ while the magnitude of the yield strength are similar. This is due to the much larger strain hardening capacity of the weld center (see Figures 6.23(b) and 6.24(b)). This result is important because it will significantly affect the overall flow behavior of the welded sample. The weakest zone of the weld is the heat affected zone where not only the yield strength is minimum but also where the strain hardening capacity is the lowest. The importance of the strain hardening capacity in the global behavior of friction stir welds in 6005A-T6 justify the interest for developing a better understanding of the strain hardening mechanisms which is the purpose of chapter 8.

(d) True fracture strain evolution

True fracture strain of micro-tensile samples of the base material. The true fracture strain of the base material micro-tensile specimens is much higher than the value given in Table 3.4 (page 33) for macro-specimens of the base material. A closer look at the fracture mode shows that a shear band is present in the macro-specimens while a high level of necking is observed in the micro-tensile specimens. Hence, values of true fracture strain have to be taken comparatively to one another and not in absolute value. The geometry of tensile test samples does indeed affect the geometry of the neck and the propensity toward shear banding.

Evolution of the true fracture strain. The evolution of the true fracture strain is a complex interaction between the yield strength, the strain hardening capacity of the various regions of the weld and the evolution of the larger precipitates responsible for the nucleation of voids. Further study is needed to understand the true fracture strain distribution throughout friction stir welds. The effect of the rotational speed on the true fracture strain in the nugget can probably be attributed to the precipitation of $\beta(\text{Mg}_2\text{Si})$ in the weld center. This precipitation might depend more on the rotational speed than on the advancing speed. Indeed, the thermal model has shown that the maximum temperature reached in the weld at hottest point is mainly affected by the rotational speed (see Figure 5.12(d)). The $\beta(\text{Mg}_2\text{Si})$ precipitates are preferential sites for the cavities that will grow and coalesce to specimen fracture.

(f) Asymmetry

The thermocouple measurements presented in chapter 4 and the thermal modeling in chapter 5 brought the conclusion that the advancing side of the weld is generally hotter than the retreating side due to the convection of hot material toward the advancing side. This temperature difference increases with increasing advancing speed (see Figure 4.9). Some of the results of the local mechanical properties characterization also show asymmetries.

- **Width of the thermally affected zone.** Figure 6.20(a) showed that the width of the zone thermally affected is larger on the advancing side than on the retreating side. The difference increases with decreasing advancing speed and increasing rotational speed. The zone of low hardness after a PWHT is larger on the advancing side than on the retreating side for all the welding parameters of Figure 6.27. Both these observations speak for a hotter advancing side.
- **Minimum tensile properties.** Figure 6.22 shows that the minimum yield strength is generally higher on the advancing side than on the retreating side. A similar effect was observed on the hardness distributions of Figure 6.20. The tendency of the strain hardening capacity results of Figure 6.23 and 6.24 is also to have an advancing side stronger than the retreating side. Model results presented in chapter 10 will help understand why the hottest side of the weld (i.e. generally the advancing side of the weld) is not the weakest side of the weld.

6.3.3 Conclusions of the mechanical characterization

Table 6.6 summarizes the evolution of the tensile properties in the different zones of a friction stir weld.

The distribution of hardness and local tensile properties of five friction stir welds corresponding to different advancing and rotational speeds have been measured. The yield strength and hardness variations are obviously highly correlated through a linear relationship.

On the one hand, the variation of the strength along a transverse line through the weld are directly connected to the fine precipitates evolution in the different zones of friction stir welds. On the other hand, the relationship between the microstructure evolution and the evolution of the

Weld zone	Tensile properties
Base material (BM)	- R_p (and HV) maximum - θ and β minimum - R_m high
Heat affected zone (HAZ)	- R_p (and HV) low - θ and β high - R_m low
Weld center (nugget and TMAZ)	- R_p (and HV) low (same level as HAZ for cold welds) - θ high - β low - R_m medium level

Table 6.6. Summary of the local tensile properties of friction stir welds. R_p is the yield strength; HV is the Vickers hardness; θ and β are the strain hardening parameters; R_m is the ultimate tensile strength.

local strain hardening capacity is not obvious. Chapter 8 will discuss the effect of the microstructural evolution on the local strain hardening abilities. Chapter 9 will discuss the effect of microstructure on the damage mechanism.

The hardness evolution after the post-welding heat treatment has allowed confirming some of the conclusions of the microstructure characterization of friction stir welds. In particular:

- The base material β'' precipitates dissolve in the zone where hardness starts to drop.
- A phase, probably $\beta(\text{Mg}_2\text{Si})$, which captures some of the magnesium and/or silicon precipitates is present in the weld center.
- The size and volume fraction of β' precipitates is higher in the heat affected zone of the weld having the lowest advancing speed. No marked effect of the rotational speed has been identified.

The following effects of the welding parameters have been identified:

- The width of the zone affected thermally increases with decreasing advancing speed and increasing rotational speed.
- The yield strength of very hot welds (low advancing speed) is lower in the heat affected zone than in the weld nugget while it is not the case in the cold welds.

- The true fracture strain of the weld center region increases with decreasing rotational speed.

Evidences of a difference in mechanical properties between the advancing side and the retreating side have been found: the width of the zone thermally affected are different, the hardness and yield strength minimum and the strain hardening parameters θ and β are also different (and the ultimate tensile strength also as a consequence). The minimum of hardness after the post-welding heat treatment seems also higher on the advancing side than on the retreating side of the weld. These differences have been attributed to the temperature difference found in chapter 4 between the advancing and the retreating side of the friction stir welds.

Chapter 7

Microstructure evolution and yield strength modeling

The microstructural evolution model presented in this chapter is similar to the initial version of the "size class model" by Myhr, Grong and co-workers [98]. Studies by Shercliff and Ashby [121, 122] as well as by Deschamps, Brechet and co-workers [23] will be used to predict the yield strength based on the microstructural evolution in the spirit of the work by Myhr *et al.* [99]. The thermal cycles measured by thermocouples (see chapter 4) are used as input data in the model. Since equivalent radius measurements of the precipitates are only available for base alloy number 1 and two friction stir welds, both models will be calibrated using the yield strength measurements on heat treated specimens at 180°C from solid solution (see Figure 6.11(a)) and on the T6 base alloy number 1 mean equivalent radius (see Table 6.2, page 93). The models will then be validated by comparing predicted and measured yield strength evolutions for tensile tests at 300°C and 350°C from the T6 state. Finally the model will be applied to friction stir welds comparing the predictions with the measured yield strength of the friction stir welds (from the micro-tensile tests: see Figure 6.12(a)) and precipitate size evolutions in friction stir welds from TEM observations (see Figure 6.16).

7.1 Models description

The "size class model" is well adapted to study microstructural evolutions during non-isothermal transformations which is particularly the case during a welding process. This model takes into account an initial particles size distribution typical of the T6 state of our base material. The choice of using a "size class model", rather than the very first model

developed by Myhr *et al.* [96] often adapted to predict the hardness evolution in welds (e.g. [97, 46]), is made in order to predict the microstructure explicitly and use it as input to the strain hardening model of chapter 8.

Myhr *et al.* [99, 100, 101] developed a more evolved version of the "size class model" to distinguish between the β'' and β' precipitation rather than to consider β' precipitates as a coarse version of β'' precipitates as will be assumed here. M. Nicolas and A. Deschamps [104] have applied the "size class model" to the modeling of the microstructural evolution in the arc welding of an Al-Zn-Mg alloy in the T6 and T7 state. Gallais *et al.* [49, 50] and Kamp *et al.* [66] have adapted the "size class model" to predict the microstructural and yield strength evolution during the friction stir welding process in 6056 and 7449 aluminum alloys.

7.1.1 Description of the microstructural model

The model consists of three parts:

- a nucleation law predicting the number of stable precipitate nuclei that form at each time step;
- a rate law calculating the dissolution or coarsening of the precipitates;
- a continuity equation which keeps the record of the amount of solute being involved in precipitates.

The composition of all the precipitates will be assumed to be Mg_2Si . Literature has shown that β'' , β' and $\beta(\text{Mg}_2\text{Si})$ precipitates do not have exactly the same composition (see Table 2.1, page 10). Nevertheless, the assumption of a constant composition allows the treatment of the complex precipitation sequence of the $\beta(\text{Mg}_2\text{Si})$ phase as the nucleation, dissolution and coarsening of only one type of precipitate. The precipitates will be assumed spherical with an equivalent radius (denoted r) and having the same volume as the elongated precipitates.

Nucleation law. If the incubation period is neglected, the steady state nucleation rate j [$\#/\text{m}^3\text{s}$] can be expressed as

$$j = j_0 \exp\left(-\frac{\Delta G_{het}^*}{R_g T}\right) \exp\left(-\frac{Q_d}{R_g T}\right) \quad (7.1)$$

where j_0 [$\#/\text{m}^3\text{s}$] is a pre-exponential term to the nucleation rate j , ΔG_{het}^* [J/mol] is the energy barrier for heterogeneous nucleation, R_g

(8.314 J/K mol) is the universal gas constant, T [K] is the temperature and Q_d [J/mol] is the activation energy for diffusion. The energy barrier for heterogeneous nucleation can be expressed as

$$\Delta G_{het}^* = \frac{(A_0)^3}{(R_g T)^2 [\ln(C_{Mg}/C_e)]^2} \quad (7.2)$$

where A_0 [J/mol] is a parameter related to the energy barrier for nucleation, C_{Mg} [wt %] is the mean solute magnesium content in the matrix and C_e [wt %] is the equilibrium solute content at the precipitate/matrix interface at temperature T . Hence, the nucleation process will eventually stop when $C_{Mg} = C_e$. For the sake of simplicity, C_e can be expressed as

$$C_e = C_{e0} \exp\left(-\frac{Q_e}{R_g T}\right) \quad (7.3)$$

where C_{e0} [wt %] is a pre-exponential term to the equilibrium solute content at the precipitate/matrix interface and Q_e [J/mol] is the apparent solvus boundary enthalpy (energy per mole needed for the dissolution of precipitates if expression (7.3) is verified). C_{e0} and Q_e are given by the equilibrium diagram of the Mg₂Si phase.

Rate law. When a precipitate, having a radius r and a magnesium concentration C_p [wt %], is present in a supersaturated solid solution with a mean concentration C_{Mg} [wt %]:

- if $C_{Mg} > C_i$, the precipitate will dissolve (r decreases),
- if $C_{Mg} < C_i$, the precipitate will grow (r increases).

where C_i is the precipitate/matrix interface concentration and is given by the Gibbs-Thomson equation:

$$C_i = C_e \exp\left(\frac{2\gamma_i V_m}{r R_g T}\right) \quad (7.4)$$

where γ_i [J/m²] is the precipitate/matrix interface energy and V_m [m³/mol] is the molar volume of the precipitate, in our case, Mg₂Si. The rate $v_{d/c}$ [m/s] at which dissolution or coarsening occurs is given by:

$$v_{d/c} = \frac{dr}{dt} = \frac{C_{Mg} - C_i}{C_p - C_i} \frac{D}{r} \quad (7.5)$$

where D [m²/s] is the diffusion coefficient which can be written

$$D = D_0 \exp\left(-\frac{Q_d}{R_g T}\right) \quad (7.6)$$

where D_0 [m²/s] is a pre-exponential term to the diffusion coefficient D and Q_d [J/mol], defined earlier, is the activation energy for diffusion. Based on equation (7.4) and (7.5), it is possible to define a critical equivalent radius r_c of a precipitate that will neither grow nor dissolve:

$$r_c = \frac{2\gamma_i V_m}{R_g T} \left(\ln \left(\frac{C_{Mg}}{C_e} \right) \right)^{-1}. \quad (7.7)$$

Continuity equation. If N [# / m³] is the density of precipitates then $Nv_{d/c}$ is the particles flux from one class to the other and a mass balance gives:

$$\frac{\partial N}{\partial t} = - \frac{\partial(Nv_{d/c})}{\partial r} + j \quad (7.8)$$

Numerical discretization. The equivalent radius distribution is divided into small classes of size Δr having a density of precipitates N_i [# / m³] and a mean equivalent radius r_i . These size classes are treated as control volumes to which the continuity equation is applied. As boundary conditions, an initial size distribution needs to be defined (if no precipitates are present, i.e. after a solid solution heat treatment, it will be a null distribution) and a very small minimum radius for newly nucleated precipitates needs to be chosen. The precipitates volume fraction f_v is calculated by

$$f_v = \sum_i \frac{4}{3} \pi r_i^3 N_i \quad (7.9)$$

and the mean magnesium solute concentration in the matrix C_{Mg} [wt %] is calculated using

$$C_{Mg} = \frac{C_0 - C_p f_v}{1 - f_v} \quad (7.10)$$

where C_0 [wt %] is the initial magnesium content in the alloy assuming that the total amount of magnesium will precipitate as fine Mg₂Si precipitates. This assumption is valid if silicon is in excess in the alloy and if the initial coarse β (Mg₂Si) volume fraction is negligible. Both these hypothesis are valid in the 6005A aluminium alloy used in this study.

7.1.2 Description of the yield strength model

The main contributions to the strengthening mechanism of an age hardenable aluminum alloy are:

- σ_d [MPa] is the grain size contribution to the flow stress. It is considered to be negligible in age hardening aluminum alloys.

- σ_{ss} [MPa] is the solid solution contribution to the flow stress. It can be expressed as [60]:

$$\sigma_{ss} = k_{Mg}(C_{Mg})^{2/3} + k_{Si}(C_{Si})^{2/3} + k_{Cu}(C_{Cu})^{2/3} \quad (7.11)$$

where k_{Mg} , k_{Si} and k_{Cu} [MPa/wt%^{2/3}] are constants and C_{Si} and C_{Cu} [wt %] are respectively the silicon and copper solid solution concentration. Copper will be assumed to stay in solid solution since the θ precipitates with copper have very low dissolution temperatures (result of a PANDAT simulation of the alloy 6005A). This assumption is only valid if the initial copper content in the alloy $C_{Cu_{init}}$ [wt %] is very low otherwise Q phases might be formed, changing thus the precipitation sequence. The silicon solid solution concentration C_{Si} is assumed to vary proportionally to the magnesium solid solution concentration C_{Mg} . This is equivalent to assuming that the excess silicon has precipitated in non-hardening coarse Si precipitates and will not dissolve during the various transformations.

Equation (7.11) becomes

$$\sigma_{ss} = k(Mg, Si)_{eq}(C_{Mg})^{2/3} + k_{Cu}(C_{Cu_{init}})^{2/3} \quad (7.12)$$

where $k(Mg, Si)_{eq}$ is another constant which can be linked to k_{Mg} and k_{Si} if we assume that the magnesium and the silicon (that does not make Si rich phases) only enter Mg_2Si precipitates

$$k(Mg, Si)_{eq} = k_{Mg} + k_{Si} \left(\frac{100 - C_p}{C_p} \right). \quad (7.13)$$

- σ_{preci} [MPa] is the precipitation contribution to the flow stress. It can be expressed as

$$\sigma_{preci} = \frac{M\bar{F}}{bl} \quad (7.14)$$

where M is the Taylor factor, $\bar{F}$ [N] is the mean obstacle strength, b [m] is the magnitude of the burger vector and l [m] is the particles distance in the glide plane of the dislocations. l can be expressed in terms of the mean precipitates equivalent radius r using Friedel's formalism [52]

$$l = \sqrt{\frac{2\pi}{3}} \frac{r}{\sqrt{f_v}} \quad (7.15)$$

which, in the case of a size distribution, gives

$$\sigma_{preci} = \frac{M}{b} (2\Gamma)^{-1/2} \sqrt{\frac{3f_v}{2\pi}} \frac{\bar{F}^{3/2}}{r} \quad (7.16)$$

where $\Gamma = \beta_\Gamma G b^2$ [N] is the dislocation line tension, β_Γ is a constant close to 0.5 and G [GPa] is the shear modulus. The average obstacle strength $\bar{F}$ is calculated

$$\bar{F} = \frac{\sum_i N_i F_i}{\sum_i N_i} \quad (7.17)$$

where F_i [N] is the obstacle strength for the size class i . The obstacle strength F_i depends on the interaction between the dislocation and the precipitate.

- If the equivalent radius r_i is smaller than an equivalent transition radius r_{trans} , then the precipitates are sheared by the dislocations and $F_i = 2\beta_\Gamma G b^2 \left(\frac{r_i}{r_{trans}} \right)$. Note that if we assume that all the precipitates have a radius equal to the mean radius r we find the classical expression:

$$\sigma_{preci} = K \sqrt{f_v r} \quad (7.18)$$

where $K = K'/(r_{trans})^{3/2}$ is a proportionality constant and K' can be calculated as

$$K' = \frac{M}{b} (2\Gamma) \sqrt{\frac{3}{2\pi}} \quad (7.19)$$

- If the equivalent radius r_i is larger than an equivalent transition radius r_{trans} , then the precipitates are bypassed by the dislocations following the Orowan mechanism (see schematic on Figure 7.1) and $F_i = 2\beta_\Gamma G b^2$. Note that if we assume that all the precipitates have a radius equal to the mean radius r we find the classical expression:

$$\sigma_{preci} = K' \frac{\sqrt{f_v}}{r} \quad (7.20)$$

- $\sigma_{pl} = M\alpha G b \sqrt{\rho}$ is the dislocation density contribution to the flow stress, where ρ is the dislocation density and α is a material dependent constant taken typically between 0.2 and 0.5. Concerning the calculation of the yield strength, we will assume that this last term is negligible which corresponds to assuming that $\sigma_{preci} \gg \sigma_{pl}$ ($\rho = \rho_{init}$). Note that this term will have to be accounted for when modeling strain hardening.

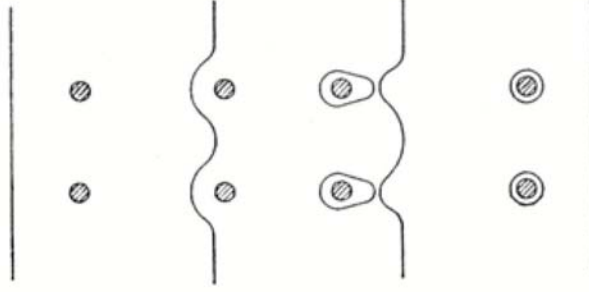


Figure 7.1. Dislocation that bypasses precipitates following the Orowan mechanism; Orowan loops formation (from [67]).

The important contributions of the yield strength are added up

$$\sigma_y = \sigma_0 + \sigma_{ss} + \sigma_{preci} \quad (7.21)$$

where σ_0 is the friction stress of a pure aluminum (about 10 MPa).

7.1.3 Description of the natural aging model

Natural aging induces an increase of the yield strength due to the precipitation of GP zones. Esmaili *et al.* [39] proposed a model to describe the yield strength evolution during natural aging. We are only interested in the final yield strength when the maximum possible amount of GP zones has precipitated. As explained earlier, a low concentration in magnesium and copper does not favor the precipitation of GP zones, hence a certain amount C_{lim} [wt %] of alloying elements will remain in solid solution. The yield strength after natural ageing will therefore be given by equation (7.22) following the formalism of Esmaili *et al.* [39].

$$\sigma_{yNA} = \sigma_y + K_{NA} \sqrt{\frac{C_{Mg} - C_{lim}}{C_0 - C_{lim}}} - k(Mg, Si)_{eq}(C_{Mg})^{2/3} + k(Mg, Si)_{eq}(C_{lim})^{2/3} \quad (7.22)$$

where K_{NA} [MPa] is a constant. The last two terms of expression (7.22) account for the loss in the solid solution concentration (term σ_{ss} of equation (7.21)). K_{NA} is calculated from the values of the yield strength just after a solution heat treatment σ_{ySHT}^{init} and after the natural aging process is over σ_{ySHT}^{NA} :

$$K_{NA} = \sigma_{ySHT}^{init} - \sigma_{ySHT}^{NA} + k(Mg, Si)_{eq}(C_0)^{2/3} - k(Mg, Si)_{eq}(C_{lim})^{2/3}. \quad (7.23)$$

Param.	Significance	Value	Origin
r for BM	BM equivalent radius distribution	Figure 6.8(b)	TEM on BM
C_0	Mg content of alloy 1	0.48 wt %	Table 3.3
C_p	Mg content in Mg_2Si	63.4 wt %	[98]
A_0	Parameter of the energy barrier for nucleation	18.6 kJ/mol	DSC and $\approx$ [99, 100, 101]
V_m	Molar volume of Mg_2Si	$3.95 \cdot 10^{-5} \text{ m}^3/\text{mol}$	[98]
C_{e0}	Pre-exponential term to C_e	290 wt %	[101]
Q_e	Apparent solvus boundary enthalpy	41,000 J/mol	[101]
D_0	Pre-exponential term to the diffusion coefficient	$10^{-4} \text{ m}^2/\text{s}$	Optimized
Q_d	Activation energy for diffusion	130,000 J/mol	[98, 99] [100, 101]
j_0	Pre-exponential term to the nucleation rate j	$3 \cdot 10^{37} \text{ \#}/\text{m}^3$	Optimized
γ_i	Precipitate/matrix interface energy	0.26 J/m ²	Optimized + [99, 100, 101]
M	Taylor factor	2	Textured alloy
G	Shear modulus	$2.7 \cdot 10^{10} \text{ Pa}$	[99, 100, 101]
b	Magnitude of Burger's vector	$2.84 \cdot 10^{-10} \text{ m}$	[99, 100, 101]
σ_0	Yield strength of pure aluminum	10 MPa	[99, 100, 101]
k_{Cu}	Constant for effect of Cu SS content on σ_{ss}	$46.4 \text{ MPa} \cdot \text{wt}\%^{2/3}$	[99, 100, 101]
$k(\text{Mg,Si})_{eq}$	Constant for effect of Mg and Si SS content on σ_{ss}	$39.7 \text{ MPa} \cdot \text{wt}\%^{2/3}$	σ_y in SS Table 6.3
r_{trans}	Transition radius	3.04 nm	Table 6.2
β_Γ	Line tension constant	0.45	Maximum σ_y
C_{lim}	Limit C_{Mg} for NA	0.39 wt%	DSC, model and welds
K_{NA}	Constant for NA model	58.1 MPa	Table 6.3 and eq. 7.23

Table 7.1. Summary of the parameters chosen for the application of the microstructural model, the yield strength model and the natural aging model to alloy 6005A (SS=solid solution, BM=base material, NA=natural aging)

7.2 Parameters identification

Table 7.1 summarizes the parameters chosen for the application of the microstructural and yield strength models to the aluminum alloy 6005A. Myhr *et al.* [101] have studied the precipitation in a 6005 alloy (0.55% Mg and 0.82% Si) having nearly the same composition as to the 6005A alloy studied in this work. The values of the parameters are similar to those selected in the latest version of the model by Myhr *et al.* [101] (when applicable) since their identification has been refined over the years up to a very high degree of confidence and physical relevance. Myhr *et al.* [101] had to calibrate a few parameters on experimental data of the equivalent radius and yield strength evolution with time of heat treatment. We will also consider these parameters as tuning parameters.

7.2.1 Parameters of the yield strength model

M . The Taylor factor is generally equal to about 3.1 in isotropic polycrystals. Nevertheless our base alloys are highly textured as confirmed by the anisotropy of the mechanical properties shown in Table 3.4 (page 33). Hence, a lower value of the Taylor factor, $M = 2$, is chosen.

β_{Γ} . The yield strength model has been applied to the base material equivalent radius distribution in order to get the value of the β_{Γ} constant that gives a good prediction of the base material yield strength. This yield strength is given in Table 3.4 (page 33): a mean value between the transverse long and extrusion direction equal to 260 MPa was chosen for the optimization of β_{Γ} . A value of β_{Γ} equal to 0.45 was identified in agreement with the theoretical value of 0.5 confirming also that the choice of the Taylor factor $M = 2$ is reasonable.

r_{trans} . The radius of transition from dislocations shearing precipitates to dislocations bypassing precipitates by the Orowan mechanism, corresponds almost to the radius at the peak yield strength. The T6 state is the peak yield strength state. Hence, the transition radius can be estimated using the mean equivalent radius of the base material 6005A in the state T6 given in Table 6.2 (page 93).

7.2.2 Parameters of the microstructural model

A_0 . DSC measurements from the solid solution state (with and without naturally aging) (see figure 6.10) show a peak of β'' precipitation at around 260°C. This value will certainly slightly depend on the heating

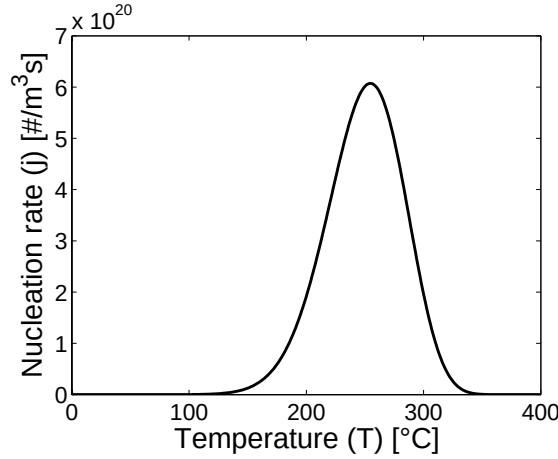


Figure 7.2. Evolution of the nucleation rate with temperature with $A_0 = 18.6$ kJ/mol.

rate (if the heating rate increases from 10°C/min to 20°C/min the β'' peak is shifted by about 20°C [56]) but it will be considered to provide a good estimation of the precipitation temperature. The nucleation rate (equation (7.1)) is maximum at 260°C when A_0 is equal to 18.6 kJ/mol for the chosen values of C_{e0} , Q_e , C_0 and Q_d ¹ (see Table 7.1, page 136).

$$A_0^3 = \frac{Q_d(RT)^2}{2(Q_e/RT)[\ln(C_0/C_{e0})]^{-3} - 3[\ln(C_0/C_{e0})]^{-2}} \quad (7.24)$$

Figure 7.2 shows the evolution of the nucleation rate with temperature, with a maximum at 260°C when A_0 is equal to 18.6 kJ/mol.

The parameters D_0 , j_0 and γ_i are calibrated based on the yield strength values of solution heat treated samples aged at 180°C (see Figure 6.11, $R_p \approx \sigma_y$). Figure 7.3(a) shows that for optimal values of γ_i and j_0 , a pre-exponential term to the diffusion coefficient D_0 equal to 10^{-4} m²/s better predicts the yield strength variation than $D_0 = 2.2 \cdot 10^{-4}$ m²/s as proposed by Myhr *et al.* [98, 99, 100, 101]. Our value of D_0 is though similar to the value found in the work by Van Horn [141]. This correction on the diffusion coefficient is acceptable since its measurement is approximate. Figure 7.3(b) shows that optimal values of the pre-exponential term j_0 and the precipitate/matrix interface energy γ_i (for $D_0 = 10^{-4}$ m²/s) are respectively of 0.26 J/m² and $3 \cdot 10^{37}$ #/m³ s giving a standard

¹A known typing error of reference [99] is corrected here.

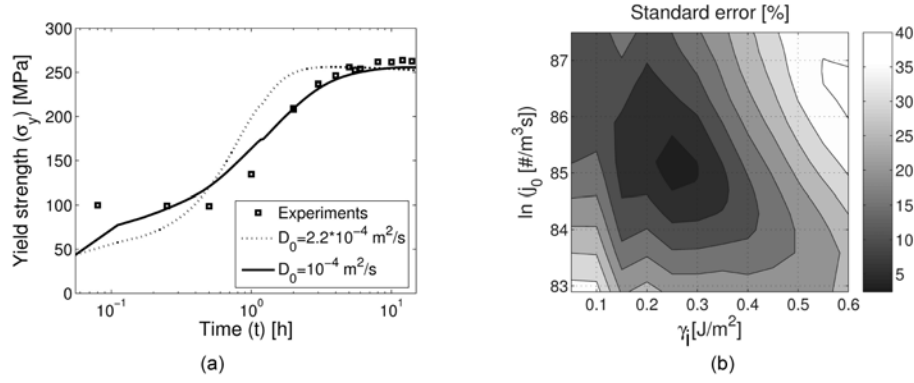


Figure 7.3. Microstructural model parameters optimization on the yield strength of solution heat treated samples aged at 180°C: (a) optimization of the pre-exponential term to the diffusion coefficient D_0 ; (b) optimization of the pre-exponential term to the nucleation rate j_0 and the precipitate/matrix interface energy γ_i for D_0 equal to $10^{-4} \text{ m}^2/\text{s}$.

error of only a few % on the yield strength prediction for solution heat treated samples aged at 180°C. The standard error is defined similarly to a standard deviation but on the difference between the predicted and measured value. The value of γ_i obtained by this optimization procedure is a good compromise between a case where all the precipitates are coherent with the aluminum matrix ($\gamma_i = 0.02 \text{ J/m}^2$) and a case where all precipitates are incoherent with the aluminum matrix ($\gamma_i = 0.6 \text{ J/m}^2$) [50]. Indeed, we have to model, with a unique value of γ_i , coherent β'' precipitates and larger semi-coherent β' precipitates. Interestingly, Myhr *et al.* [101] use the same value of γ_i in their model. Their value of γ_i has been calibrated based on a large amount of heat treatment data. In a more evolved version of the model which distinguishes between β'' and β' precipitation, Myhr *et al.* [99, 100] give a value of the precipitate/matrix interface energy γ_i equal to 0.26 J/m^2 for β'' precipitates. The value of j_0 proposed here is slightly higher than the different ones proposed by Myhr *et al.* [98, 99, 100, 101]. Nevertheless, many physical parameters, in particular the initial concentration in vacancies, may affect the nucleation rate.

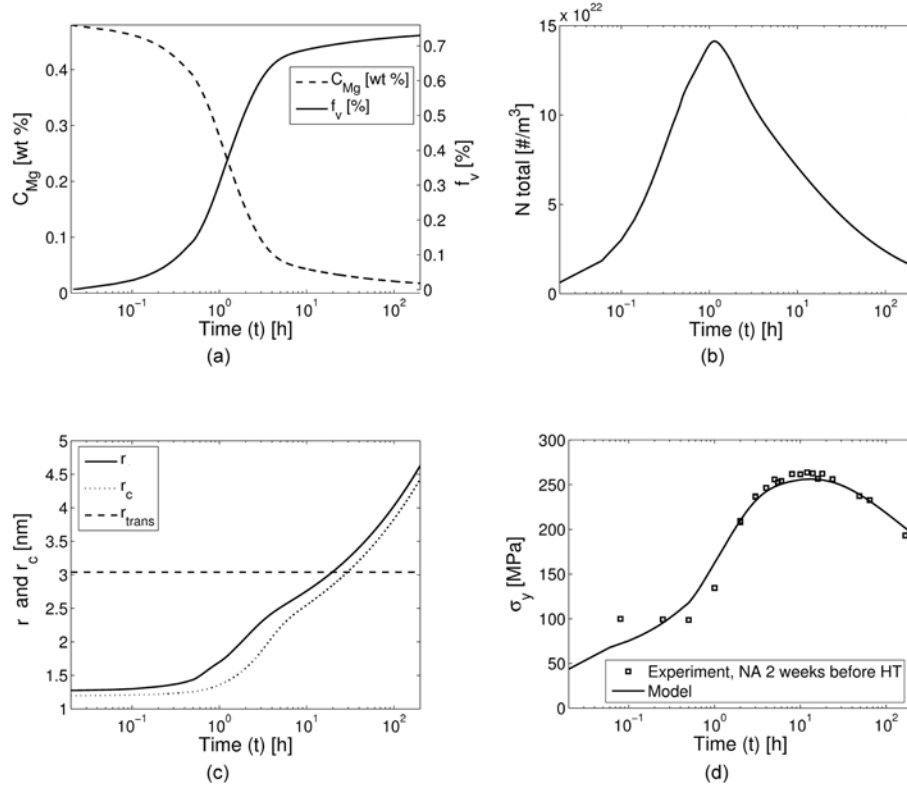


Figure 7.4. Predictions of the microstructure and yield strength model and comparison to the experimental results for samples solution heat treated and aged at 180°C: (a) magnesium concentration in solid solution C_{Mg} and volume fraction f_v ; (b) total number of precipitates N total; (c) equivalent radius r and critical radius r_c ; (d) yield strength σ_y (NA = natural aging and HT = heat treatment).

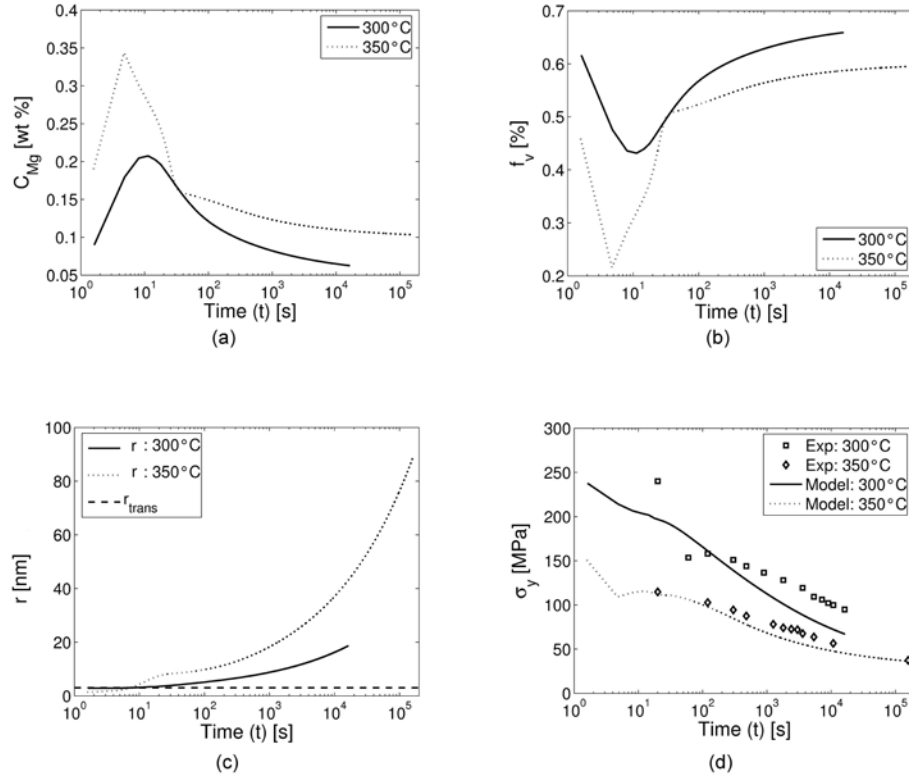


Figure 7.5. Predictions of the microstructure and yield strength model and comparison to the experimental results for samples heat treated at 300°C and 350°C from the T6 state: (a) magnesium concentration in solid solution C_{Mg} ; (b) volume fraction f_v ; (c) equivalent radius r ; (d) yield strength σ_y .

7.3 Model assessment

7.3.1 Application of the model to solution heat treated samples

The predictions of the yield strength model for samples solution heat treated and aged at 180°C are obviously very good since the parameters D_0 , j_0 and γ_i have been calibrated from these data. Figure 7.4 shows the predictions of the microstructural and yield strength model for these samples. Note that the effect of natural aging before a heat treatment at 180°C is not taken into account by the model which explains the low quality of the predictions for short heat treatment times. The natural aging model cannot be applied in this case since it applies to natural aging after a heat treatment and not before.

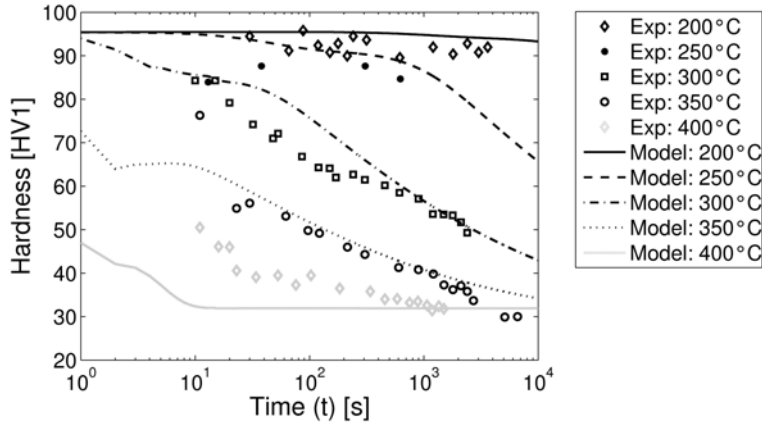


Figure 7.6. Hardness predictions for heat treated samples of base alloy number 2

The predictions of the yield strength model for samples heat treated at 300°C and 350°C from the T6 state (see Figure 7.5) provide a conspicuous validation of the microstructural and yield strength model. The model slightly underestimates the yield strength for long heat treatment times. The dissolution of the precipitates is very rapid at such high heat treatment temperatures. The fraction dissolved at 350°C is higher than the fraction dissolved at 300°C.

A few modifications had to be done in order to apply the model to the base alloy number 2. Firstly, the copper content of the alloy had to be changed since it is lower in base alloy number 2. Secondly, the value of β_T had to be adapted in order to ensure an accurate prediction of the base alloy yield strength. A value of $\beta_T = 0.47$ has been found. Finally, equation (6.1) relating the hardness and the yield strength is not applicable to an alloy in solid solution since it would drastically overestimate the solid solution hardness. Hence, a new law for the hardness evolution has been identified based on the two available data for base alloy number 2: the hardness and yield strength of base alloy number 2 at T6 state and in solid solution (the solid solution yield strength being a result of the model):

$$HV = 21.3 + 0.28 * R_p. \quad (7.25)$$

The other parameters have been assumed constant. Figure 7.6 shows that the application of the model to heat treatments on the base alloy number 2 give very satisfactory results. Nevertheless, the predicted hardness decreases too rapidly for the 400°C heat treatments.

7.3.2 Application of the model to welds and discussion

(a) Microstructure model applied to welds

The microstructure evolution model was applied to the two friction stir welds for which the precipitates radius was measured by TEM analysis. The input thermal cycles are the ones measured by thermocouples inserted in the workpiece during friction stir welding². Note that some thermocouples were missing on the retreating side of the welds and it has been assumed that the temperature difference between both sides was negligible at 14 mm and more from the weld centerline.

Figure 7.7 shows the variation of the microstructural parameters with the position along a transverse section of the weld for two welds with different advancing speeds. The radii predictions are quite good considering the large dispersion of the experimental measurements. The hypothesis of a constant length for the equivalent radius calculation is clearly a wide approximation (see Figure 6.8(a) for the base material) but it still gives satisfactory results in the heat affected zone of these friction stir welds.

On the outer region of the heat affected zone, due to the high kinetics of dissolution the volume fraction drops very rapidly while the solid solution concentration in magnesium increases. In the mean time, precipitates coarsening takes place in the heat affected zone. Globally, in these friction stir welds, total dissolution of the β'' - β' precipitates occurs close to the weld center.

The hot weld (200 mm/min) presents a plateau of the volume fraction f_v and the solid solution concentration C_{Mg} at about 15 mm from the weld centerline. This corresponds to the sudden change between growing precipitates and massive dissolution which are two antagonistic effects on the evolution of the volume fraction and solid solution concentration. The reason why this plateau is not observed in the cold weld (1000 mm/min) is probably due to the lower precipitate radii reached in that weld.

(b) Effect of the heating and cooling rate

Figure 7.8 compares the evolution of the equivalent radius with time for the thermal cycles in the heat affected zone with the same maximum

²The predictions of the microstructure evolution in the weld center will be addressed in chapter 10 when bridging all the models.

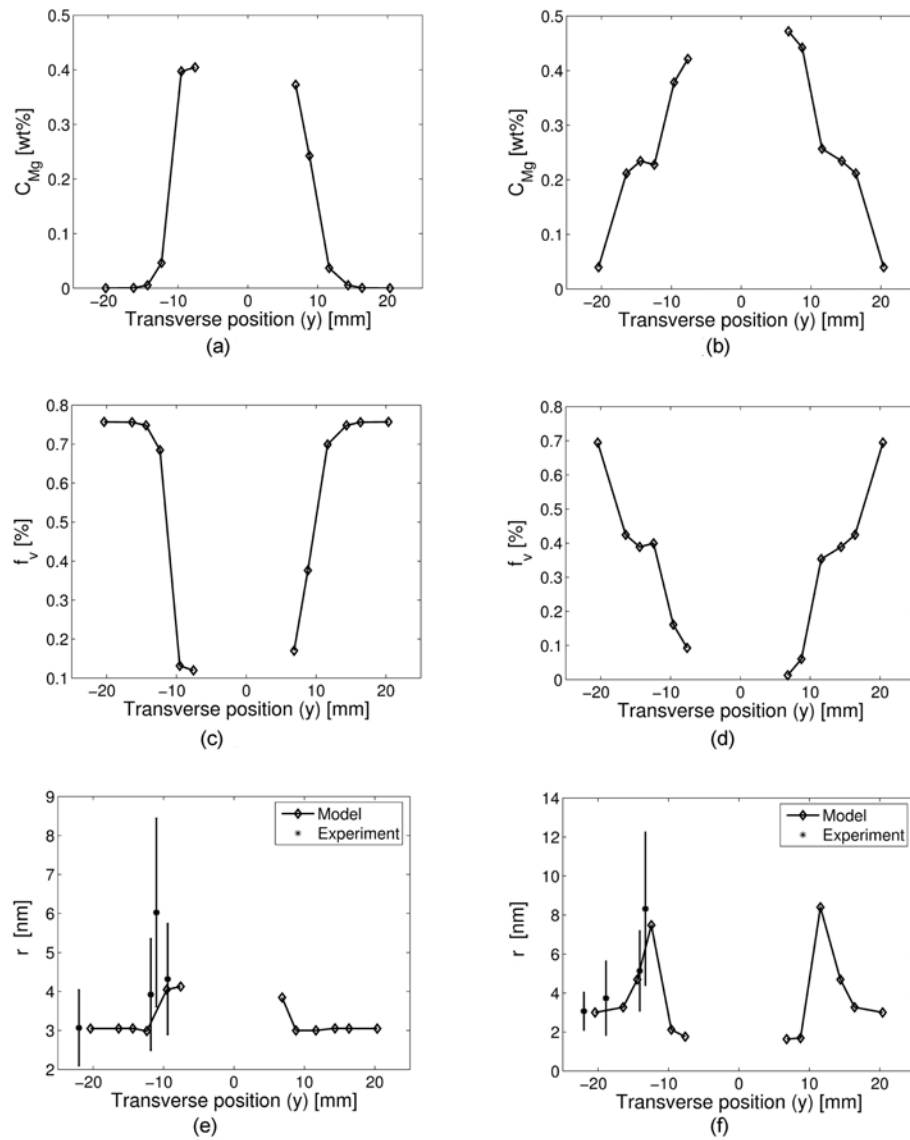


Figure 7.7. Results of the microstructure model for two friction stir welds with welding conditions 1000 rpm with (a,c,e) 1000 mm/min; (b,d,f) 200 mm/min: (a,b) magnesium concentration in solid solution C_{Mg} ; (c,d) volume fraction f_v ; (e,f) equivalent radius r

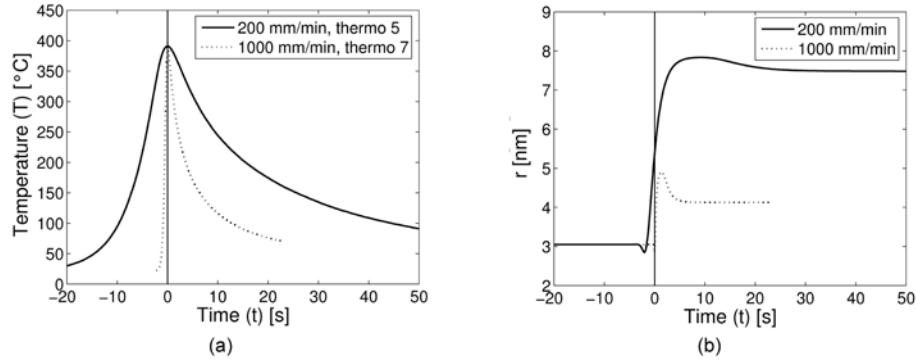


Figure 7.8. Evolution of the radius during a thermal cycle. Comparison of a hot (200 mm/min and 1000 rpm) and a cold weld (1000 mm/min and 1000 rpm): (a) thermal cycle for respectively thermocouple 5 and 7; (b) consequent evolution of the equivalent radius. The maximum temperature is reached at the time 0 s and is indicated by a vertical line.

temperature comparing cold and hot welds. Figure 4.4 has already shown that the heating and cooling rates of hot and cold welds are very different. Figure 7.8(b) shows that this difference in heating and cooling rate has a tremendous impact on the coarsening of the precipitates: coarsening starts earlier and finishes later in the hot weld compared to the cold weld.

(c) Parameters for the natural aging model

A natural aging model is needed to predict the yield strength in the welds contrarily to the heat treated samples for which very little time was left for natural aging between the heat treatment and the tensile test. The natural aging model presented in the preceding section necessitates the identification of two parameters C_{lim} and K_{NA} . Once C_{lim} is identified, K_{NA} can simply be calculated by expression (7.23) and the yield strength results of Table 6.3 (page 94).

The value of C_{lim} was identified on the friction stir weld with welding parameters 1000 mm/min and 1000 rpm. Figure 7.7(a) shows that at about 10 mm from the weld centerline on the advancing side of the weld, the solid solution concentration is of 0.39 wt %. The DSC results of Figure 6.29 shows that at 10 mm from the centerline on the advancing side of the weld no GP zones have precipitated in the friction stir weld. The hardness of that weld at 10 mm from the weld centerline on the advancing side of the weld is equal to about 70 HV1 which corresponds

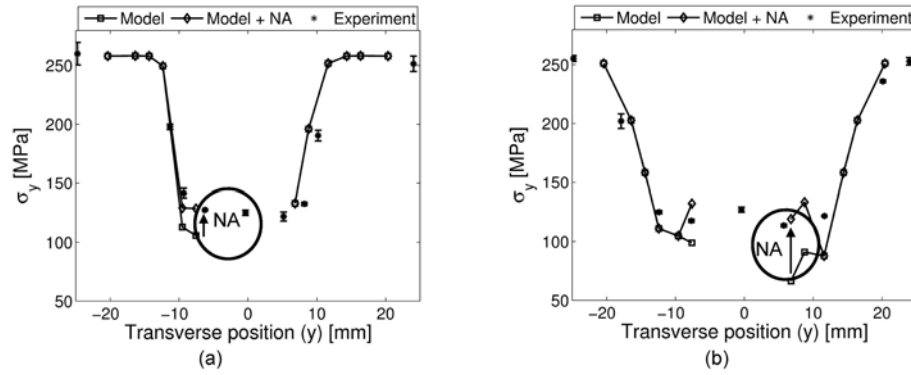


Figure 7.9. Results of the yield strength model for two friction stir welds with welding parameters 1000 rpm and (a) 1000 mm/min; (b) 200 mm/min. (NA = natural aging)

to the hardness at which natural aging starts to play an important role for partially dissolved samples as shown previously in Figure 6.9(b). Hence, we will consider that the precipitation of GP zones starts only at a very high level of solid solution concentration of $C_{lim} = 0.39$ wt %.

(d) Yield strength and hardness predictions

Figure 7.9 shows the evolution of the predicted and measured yield strength with the transverse position. The effect of natural aging is to increase the yield strength in the weld center. Note that the effect of natural aging is not tremendously marked in the zones of the welds where thermocouple measurements were possible. The effect of natural aging will be much clearer in the weld center when using the thermal cycles predicted by the thermal model as input to the microstructure evolution model (see Chapter 10). The yield strength predictions are of remarkably good quality in both friction stir welds.

Figure 7.10 shows the variation of the predicted and measured hardness with the transverse position. The hardness predictions are based on the yield strength predicted after natural aging linking them by the linear fit identified on the micro-tensile samples (equation (6.1)). Indeed, expression (6.1) gives better predictions of the hardness variation with the yield strength compared to expression (7.25) when long times are left for natural aging.

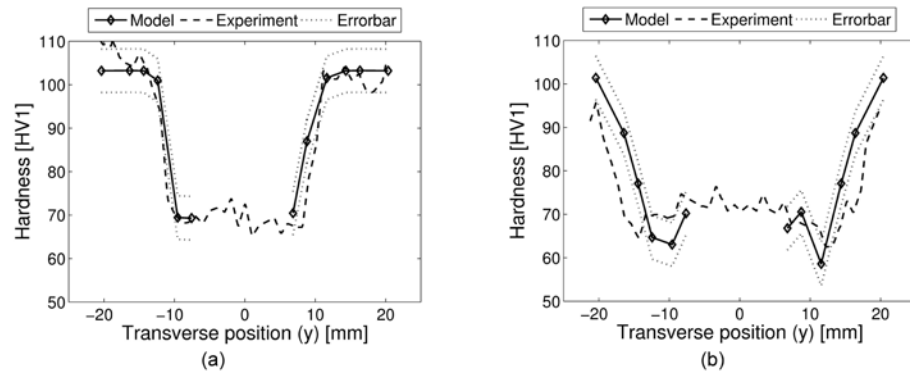


Figure 7.10. Predictions of the hardness HV1 based on the yield strength model for two friction stir welds with welding parameters 1000 rpm and (a) 1000 mm/min; (b) 200 mm/min. The ± 5 HV1 error bar is due to the linear fit error relating the yield strength and the hardness. (NA = natural aging)

The prediction of the hardness is a bit less satisfactory in the hot weld than in the cold weld. Hardness slightly depends on the strain hardening capacity which is not taken into account for in equation (6.1). It will be shown in chapter 8 that the strain hardening capacities of hot and cold welds in the HAZ are very different due to the marked influence of the size of the β' precipitates on strain hardening mechanisms. Anyway, hardness predictions are presented here only because they are practical measurements in welding characterization but hardness is not essential to the building of the complete modeling chain.

(e) Effect of a post-welding heat treatment

The model can be applied to post-welding heat treatments at 185°C for 6 h by introducing as input the distribution of precipitates radii predicted by the microstructure evolution model applied to the welds thermal cycles. Figure 7.11 compares the results of the model with the experimental measurements of the hardness after a post-welding heat treatment for two friction stir welds obtained with different advancing speeds. Very good agreement is found between the model and the experiments. The effect of the change of advancing speed is also well reproduced.

(f) Comparison with the results of the weld characterization

The comparison of the results of the weld characterization presented in chapter 6 with the results of the microstructure and yield strength

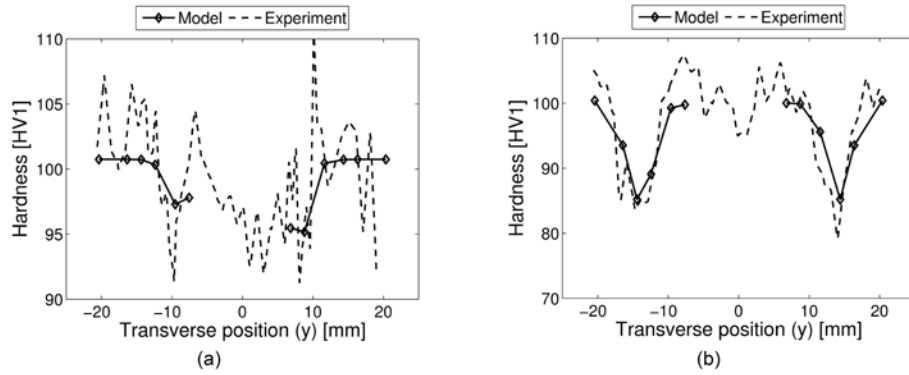


Figure 7.11. Predictions of the hardness HV1 after a post-welding heat treatment at 185°C for 6 h for two friction stir welds with welding conditions 1000 rpm at (a) 1000 mm/min and (b) 200 mm/min.

model lead to the following comments

- The model captures the fact that a higher precipitate mean equivalent radius is obtained in the heat affected zone of a hot weld, as observed with TEM.
- The lower yield strength in the heat affected zone, compared to that in the weld center, observed in the hot weld and not in the cold weld is predicted by the model (see Figure 7.9(b)).
- The heat affected zone is wider in the hot weld than in the cold weld. Hardness measurements showed the same trends (see Figure 6.20(c)).
- The results of the modeling of the yield strength variation and the volume fraction are shown together in Figure 7.12. These plots show that the volume fraction drops simultaneously with the yield strength (and the hardness) for the two welds. Hence, the precipitates start dissolving at the beginning of the zone where the yield strength start to drop. These results are in agreement with the dissolution of β'' precipitates in the "HAZ, mid-level hardness" determined by DSC. The post-welding heat treatments results also confirm the presence of a supersaturation of the solid solution at the beginning of the hardness drop zone. This explains the good results of the model after a post-welding heat treatment in that region of the weld (see Figure 7.11).

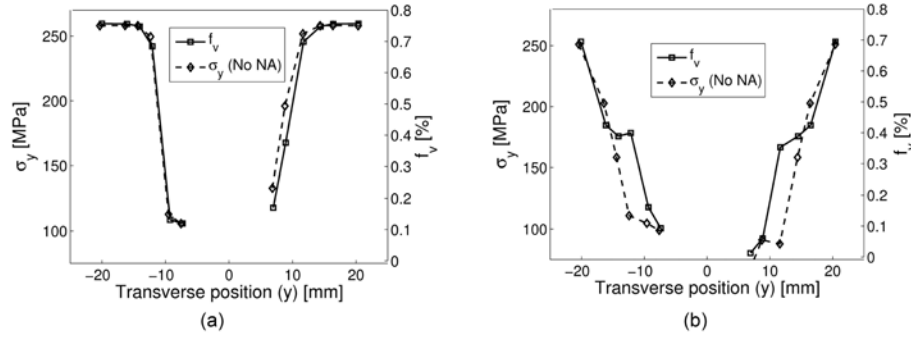


Figure 7.12. Comparison of the predicted yield strength σ_y and the volume fraction of precipitates f_v for two friction stir welds with welding conditions 1000 rpm at (a) 1000 mm/min and (b) 200 mm/min.

- As is shown in Figure 7.13, the mean equivalent radius after a post-welding heat treatment (PWHT) is lower in a cold weld than in a hot weld. Note that this mean equivalent radius corresponds to two populations of precipitates: the new ones created by the elements in solid solution that have precipitated during the PWHT and the precipitates already present before the PWHT that have grown during the PWHT.
- The post-welding heat treatments seems to indicate that the volume fraction of precipitates in the lowest hardness zone (i.e. the lowest yield strength zone in the model) could be higher in the hot weld than in the cold weld. This resulted of the observation of a lower minimum hardness after PWHT in the hot weld than in the cold weld. Figure 7.14 shows a comparison of the predicted hardness after a post-welding heat treatment and the volume fraction of precipitates before the post-welding heat treatment for two friction stir welds. The lowest hardness region after a post-welding heat treatment corresponds to a volume fraction equal to about 0.1% in the cold weld and equal to about 0.4% in the hot weld (corresponding to the mid-level plateau of the volume fraction f_v). This explains the tremendous difference in the minimum post-welding heat treatment hardness between a hot and a cold weld.

7.4 Conclusions

A model for predicting the microstructure variation and the yield strength and hardness variation for any thermal history and an initial

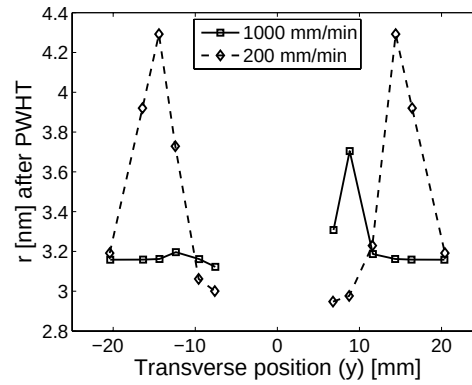


Figure 7.13. Comparison of the predicted mean radius after a post-welding heat treatment for two advancing speeds and a rotational speed of 1000 rpm.

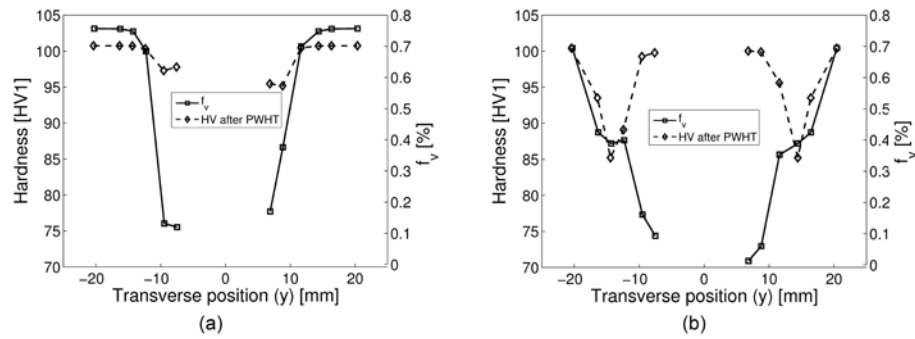


Figure 7.14. Comparison of the predicted hardness after a post-welding heat treatment and the volume fraction of precipitates before the post-welding heat treatment f_v for two friction stir welds with welding parameters 1000 rpm at (a) 1000 mm/min and (b) 200 mm/min.

precipitate size distribution (or eventually no size distribution if a solution heat treated sample is the start microstructure) has been presented, identified and validated in this chapter. The predictions of the yield strength and hardness evolutions of heat treated samples agree very well with experimental results. Furthermore, the model allows predicting the fine precipitates evolutions in friction stir welds before and after a post-welding heat treatment. The observations of the weld characterization presented in chapter 6 concerning the fine precipitation, the yield strength and hardness evolution have been shown to be well reproduced by the model. In particular, the model supports the conclusion made based on the hardness measurements after a post-welding heat treatment: the volume fraction of precipitates of the lowest hardness zone is higher in a hot weld than in a cold weld.

Chapter 8

Microstructure based strain hardening modeling

The θ and β strain hardening parameters have already been introduced in equation (3.5). Figure 8.1 recalls their definition. The first part of this chapter present a novel strain hardening model that allows to relate these parameters to the alloy microstructure. The second part is applying this model to the friction stir welds for which the microstructure was modeled in chapter 7.

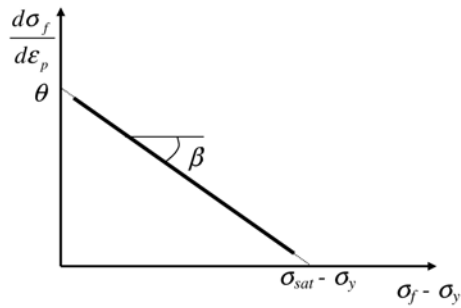


Figure 8.1. Definition of the strain hardening parameters θ and β . σ_f is the flow stress, σ_y is the true yield strength and σ_{sat} is the saturation stress.

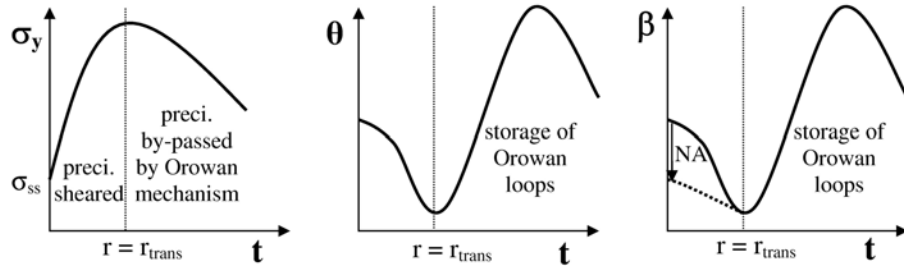


Figure 8.2. Schematic of the evolution of the yield strength σ_y and strain hardening parameters θ and β with time of heat treatment t starting from a supersaturated solid solution (NA = natural aging).

8.1 Position of the problem and limitations of the classical KME model

8.1.1 Literature survey and confrontation with the experimental results on the alloy 6005A

Using the parameters θ and β to describe strain hardening is classically done in the scientific literature for aluminum alloys [15, 21, 37, 44]. For example, Esmaili *et al.* [37] have applied it to samples of AA6111 aged at 180°C priorly solution heat treated. When the heat treatment time is such that maximum yield strength is not yet obtained, θ decreases with heat treatment time. The value of θ and β for massively over-aged samples (6 months) are even lower than for the maximum yield strength value. This is in agreement with our results already shown in Figure 6.11. Massive over-aging causes the drop observed in Figure 6.12. Cheng *et al.* [15] have also shown results for aluminum alloys AA6111 and AA7030 very similar to the ones shown in Figure 6.11. After a long time of aging, β tends to a maximum as shown in Figure 6.12.

The effects of precipitates on θ and β can be summarized as follows based on our experimental results presented in Figure 6.11(e,f) and 6.12(e,f) and on the short literature survey discussed before (see schematics on Figure 8.2):

- The supersaturated solid solution samples have a high value of θ while β is only slightly higher than its lowest level attained after natural aging (NA).
- When the precipitates radius is close to r_{trans} , θ and β show a local minimum.

- When aging proceeds (over-aging), precipitates are by-passed leaving Orowan loops around the precipitate. θ and β dramatically increase and reach a maximum at a certain level.
- When the samples are tremendously over-aged, θ and β drop.

8.1.2 Classical KME approach

(a) Pure materials

The θ and β strain hardening parameters are defined with reference to the Kocks-Mecking-Estrin (KME) model [41, 89] developed for pure materials. This section aims at explaining the theoretical basis of this model.

In a pure crystal, the shear flow stress τ_f [Pa] is related to the dislocation density ρ [# / m²] through relation (8.1).

$$\tau_f = \tau_y + \alpha G b \sqrt{\rho} \quad (8.1)$$

where τ_y [Pa] is the shear flow stress at the beginning of yielding and α is a material dependent constant taken typically between 0.2 and 0.5. The evolution of the dislocation density with the local plastic shear strain γ_p depends on two mechanisms:

$$\frac{d\rho}{d\gamma_p} = \left. \frac{d\rho}{d\gamma_p} \right|_h - \left. \frac{d\rho}{d\gamma_p} \right|_s \quad (8.2)$$

where the subscripts h and s represent the storage hardening mechanisms and the dynamic recovery which governs softening, respectively.

- **First term.** The storage term is assumed to be inversely proportional to the average spacing between obstacles L_0 [m]. For pure metals where the only storage sites are dislocations, L_0 is proportional to $1/\sqrt{\rho}$.
- **Second term.** The contribution to the time derivative ($d\rho/dt$) scales as ρ^2 . This could be justified by assuming that dislocation annihilation is a pair process. Making also use of Orowan's equation ($d\gamma_p/dt$) $\propto \rho$, we find that ($d\rho/d\gamma_p|_s$) is linear in the dislocation density ρ . The proportionality coefficient is then directly related to the critical annihilation distance below which two dislocations of opposite sign will spontaneously annihilate.

This leads to

$$\left. \frac{d\rho}{d\gamma_p} \right|_h = k_1 \sqrt{\rho}, \quad (8.3)$$

$$\left. \frac{d\rho}{d\gamma_p} \right|_s = k_2 \rho. \quad (8.4)$$

Hence,

$$\frac{d\rho}{d\gamma_p} = k_1 \sqrt{\rho} - k_2 \rho. \quad (8.5)$$

Combining equations (8.1) and (8.5) lead to

$$\frac{d\tau_f}{d\gamma_p} = \frac{d\tau_f}{d\rho} \cdot \frac{d\rho}{d\gamma_p}; \quad (8.6)$$

$$= \frac{\alpha G b}{2 \sqrt{\rho}} (k_1 \sqrt{\rho} - k_2 \rho); \quad (8.7)$$

$$= \frac{\alpha G b}{2} (k_1 - k_2 \sqrt{\rho}); \quad (8.8)$$

$$= \frac{\alpha G b k_1}{2} - \frac{k_2}{2} (\tau_f - \tau_y). \quad (8.9)$$

For polycrystals, the local shear flow stress τ_f and the plastic shear strain γ_p are related respectively to the flow stress σ_f and the plastic strain ε_p by the Taylor factor M (i.e. $M = \sigma_f / \tau_f = \gamma_p / \varepsilon_p$). Hence equation 8.9 becomes:

$$\frac{d\sigma_f}{d\varepsilon_p} = \theta_0 - \beta_0 (\sigma_f - \sigma_y). \quad (8.10)$$

where $\theta_0 = \alpha G b M^2 k_1 / 2$ and $\beta_0 = k_2 M / 2$ giving an expression of $(d\sigma_f / d\varepsilon_p)$ similar to the one sketched in Figure 8.2 with $\beta = \beta_0$ and $\theta = \theta_0$. θ_0 and β_0 are the values of β and θ for pure materials. After integration, expression (8.10) gives the expression of the flow stress σ_f during work hardening:

$$\sigma_f = \frac{\theta_0}{\beta_0} (1 - \exp(-\beta_0 \varepsilon_p)) + \sigma_y \quad (8.11)$$

This expression is called Voce law. Strictly speaking, the microscopic derivative of Voce law is valid for pure metals and solid solutions only. However, when such a linear description $d\sigma_f / d\varepsilon_p = \theta - \beta(\sigma_f - \sigma_y)$ is possible for alloys, it is reasonable to consider θ as resulting from dislocation storage and β as controlled by dynamic recovery mechanisms. This hypothesis keeps to a minimum the number of parameters fitting the flow stress during work hardening but the question becomes then to understand the influence of precipitation on θ and β .

(b) Effect of Orowan loops formation

In chapter 7, the formation of Orowan loops was already introduced and shown to be at the origin of the decrease of the yield strength when $r > r_{trans}$. When $r < r_{trans}$, the precipitates are sheared by the dislocations and therefore no extra storage of dislocations occurs during the deformation of the material compared to a material without precipitates. When $r > r_{trans}$, Orowan loops are stored around the precipitates. Estrin [42] and later Cheng *et al.* [15] and Poole *et al.* [108] take the storage of Orowan loops into account by introducing a new term in the evolution of the dislocation density with deformation (i.e. equations (8.5)). This term is inversely proportional to the particles spacing in the glide plane of the dislocations l [m] (given by equation (7.15)):

$$\frac{d\rho}{d\gamma_p} = k_1\sqrt{\rho} + \frac{1}{bl} - k_2\rho \quad (8.12)$$

Using equation (8.1), we find the expression of the work hardening rate for $r > r_{trans}$.

$$\frac{d\tau_f}{d\gamma_p} = \frac{d\tau_f}{d\rho} \cdot \frac{d\rho}{d\gamma_p} \quad (8.13)$$

$$= \frac{\alpha G b}{2\sqrt{\rho}} \left(k_1\sqrt{\rho} + \frac{1}{bl} - k_2\rho \right) \quad (8.14)$$

$$= \frac{\alpha G b k_1}{2} + \frac{\alpha^2 G^2 b}{2l(\tau_f - \tau_y)} - \frac{k_2}{2}(\tau_f - \tau_y). \quad (8.15)$$

Using the Taylor factor M and the expressions of θ_0 and β_0 , one gets

$$\frac{d\sigma_f}{d\gamma_p} = \theta_0 + \frac{\alpha^2 G^2 b M^3}{2l(\sigma_f - \sigma_y)} - \beta_0(\sigma_f - \sigma_y). \quad (8.16)$$

Hence, now the dislocation storage parameter θ expresses as:

$$\theta = \theta_0 + \frac{\alpha^2 G^2 b M^3}{2l(\sigma_f - \sigma_y)}. \quad (8.17)$$

For the sake of simplicity one can assume that the variation of $(\sigma_f - \sigma_y)$ between 0 and $(\sigma_{sat} - \sigma_y)$ (see Figure 8.1 for the definition of the saturation stress σ_{sat}) is small, hence we will take a mean value of $(\sigma_f - \sigma_y)$ to express the second term of equation (8.17): $(\sigma_{sat} - \sigma_y)/2 = \theta/(2\beta_0)$. Using this formalism, the assumption that the dislocation storage parameter θ is independent of the flow stress is an approximation which seems to be well validated by experimental results of tensile tests on

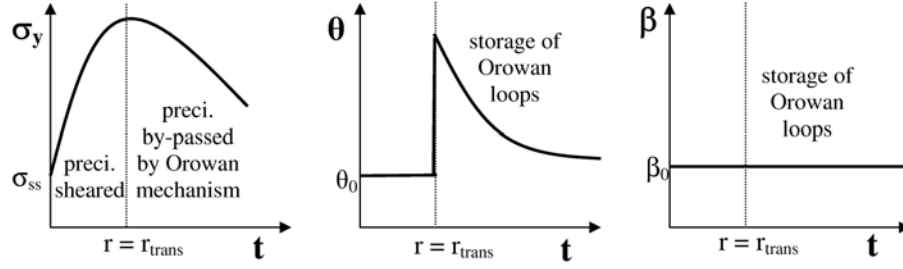


Figure 8.3. Schematic of the evolution of the yield strength σ_y , the dislocation storage parameter θ and the dynamic recovery rate β with time of heat treatment t starting from a supersaturated solid solution. Predictions of the dislocation storage parameter θ and the dynamic recovery rate β for the classical KME approach.

aluminum alloys found in the literature [15, 21, 37, 44]. Using the expression of l (equation (7.15)), we get the following implicit expression for the dislocation storage parameter θ :

$$\frac{\theta}{G} = \frac{\theta_0}{G} + \alpha^2 G M^3 \frac{\beta_0}{\theta} \sqrt{\frac{3f_v}{2\pi}} \left(\frac{b}{r}\right). \quad (8.18)$$

Hence,

$$\frac{\theta}{G} = \frac{\theta_0}{2G} + \sqrt{\left(\frac{\theta_0}{2G}\right)^2 + \alpha^2 M^3 \beta_0 \left(\frac{b}{r}\right) \sqrt{\frac{3f_v}{2\pi}}}. \quad (8.19)$$

(c) Limitations of the classical KME approach

Figure 8.3 shows the evolution of the strain hardening parameters predicted by this approach. By comparison with the experimental observation summarized in Figure 8.2, we see that the current approach is insufficient. Indeed it does not capture:

- the decrease of the dynamic recovery rate β with increasing precipitate size, as long as they are overcome by the shearing process;
- the increase of the dynamic recovery rate β with increasing precipitate size, when the precipitates are bypassed by the Orowan mechanism;
- the final drop of the dynamic recovery rate β for massively overaged samples;
- the drop of the the dislocation storage parameter θ parameter when precipitates are sheared by the dislocations (i.e. $r < r_{trans}$);

- for only slightly over-aged materials (i.e. $r > r_{trans}$), the increase of the dislocation storage parameter θ is progressive at $r = r_{trans}$. Orowan loops storage is probably more complex when the precipitates radius is slightly above the transition radius r_{trans} .

Some references in the literature model the dislocation storage rate in aluminum alloys [15, 53, 103, 108]. Nevertheless, these models are mainly phenomenological models: the parameters of these models are hardly related to the precipitates/dislocation interaction mechanisms. Furthermore, to the author's knowledge, no clear explanation nor model have been proposed for the effect of precipitation on dynamic recovery.

8.2 Enhanced physics based modeling of strain hardening by dislocation-precipitation interaction

8.2.1 Stability of the Orowan loops around coherent precipitates

The question of the stability of the Orowan loops is crucial to explain why the sudden theoretical increase of the dislocation storage parameter θ when $r = r_{trans}$ (see Figure 8.3) is not observed experimentally. Indeed, if the Orowan loops formed for $r > r_{trans}$ are unstable (i.e. annihilate themselves) then the dislocation storage parameter θ (and eventually the dynamic recovery rate β) will not be at its highest possible value for a given precipitate radius.

A sharp transition between precipitates shearing and precipitates bypassing (equation (7.14) and (7.16)) is somewhat simplistic when the material deforms plastically beyond the yield point. The flow stress increases due to work hardening. In practice, the precipitates with r slightly greater than r_{trans} will first be bypassed by the dislocation. When plastic straining proceeds, the Orowan loop undergoing an increasing stress due to the increasing pile up of other dislocation loops and the increasing overall stress will end-up shearing the precipitate if it is coherent with the matrix. Finally, the loop will annihilate itself.

Figure 8.4 sketches a precipitate of equivalent radius r being (a) surrounded by a circular Orowan loop and (b) partially sheared by a circular Orowan loop, depending on the distance x of the loop to the center of the precipitate. The variation of the free energy of the system

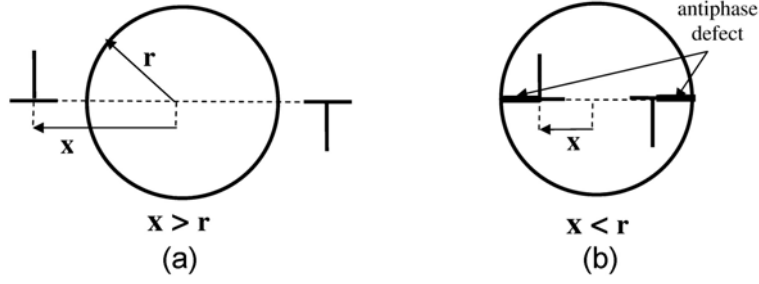


Figure 8.4. Precipitate of equivalent radius r being (a) surrounded by a circular Orowan loop or (b) sheared by a circular Orowan loop

(loop+precipitate) dW_{l+p} for a variation dx of the position of the loop is composed of three terms [7]:

$$dW_{l+p} = dW_{area} + dW_{PK} + dW_{chemical} = 2\pi\Gamma dx + \tau b(2\pi x)dx - E_a(2\pi x)dx \quad (8.20)$$

The first term, $dW_{area} = 2\pi\Gamma dx$ is the reduction of line energy, Γ [J/m] being the line tension of dislocations and $2\pi dx$ the reduction in perimeter due to the variation of the loop position. The second term $dW_{PK} = \tau b(2\pi x)dx$ is the work of the Peach Köhler force τb [N/m] [145] needed to shear a precipitate of perimeter $2\pi x$ (where τ is the applied shear stress). The third term $dW_{chemical} = E_a(2\pi x)dx$ is the increase of the antiphase boundary surface energy E_a [J/m²] due to the chemical defect caused by the shift of atoms due to the dislocation movement. By integrating equation (8.20), we find the expression of the free energy as a function of the position of the Orowan loop inside the precipitate [7]:

$$\int_r^x \frac{dW_{l+p}}{dx} dx = W_{l+p}(x) - W_{l+p}(r) = 2\pi\Gamma(x - r) - \pi(E_a - \tau b).(x^2 - r^2) \quad (8.21)$$

Equation (8.21) shows a maximum when $\tau < \tau_C$ (see Figure 8.5) corresponding to an energy barrier that prevents the loop to shear completely the precipitate and collapse itself. When the critical value of $\tau = \tau_C$ is reached there is no more energy barrier and the loop disappears. τ_C is given by the following expression [7]:

$$\tau_C = \frac{1}{b} \left(E_a - \frac{\Gamma}{r} \right). \quad (8.22)$$

For a polycrystal, the expression of the critical flow stress is given by

$$\sigma_C = \frac{M}{b} \left(E_a - \frac{\Gamma}{r} \right). \quad (8.23)$$

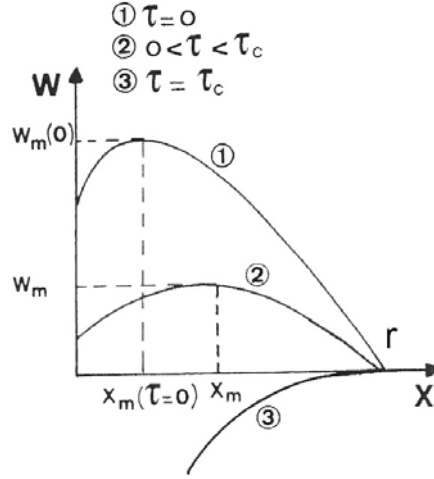


Figure 8.5. Free energy of the system (loop + precipitate) as a function of the position x of the loop (from [7]).

Figure 8.6 shows the effect of the radius of the precipitate on the strain hardening capacity. If $r < r_{trans}$, the precipitates are sheared by the dislocations. The constants K and K' (see expression (7.19) and preceding) depend on the mean obstacle force for precipitates shearing and bypassing. If $r > r_{trans}$, the precipitate is first bypassed and, then when the applied shear stress reaches $\sigma_y + \Delta\sigma_f$, it is sheared by the left Orowan loops. The expression of $\Delta\sigma_f$ is the following:

$$\Delta\sigma_f = \frac{ME_a}{b} - \frac{K'\sqrt{f_v} + (M/b)\Gamma}{r} \quad (8.24)$$

If the precipitates are incoherent (i.e. high equivalent radius) the Orowan loops can hardly enter the precipitate anymore due to the incoherent interface between the matrix and the precipitate. Hence, when the equivalent radius associated to the loss of coherency (noted r_{cl}) is reached the Orowan loop will remain around the precipitate.

8.2.2 Efficiency of dislocation storage φ

An efficiency factor φ for the storage of Orowan loops is introduced in order to take into account the stability of the Orowan loops. The factor φ is defined as the proportion of the Orowan loops that could potentially be formed and will not finish annihilating themselves when

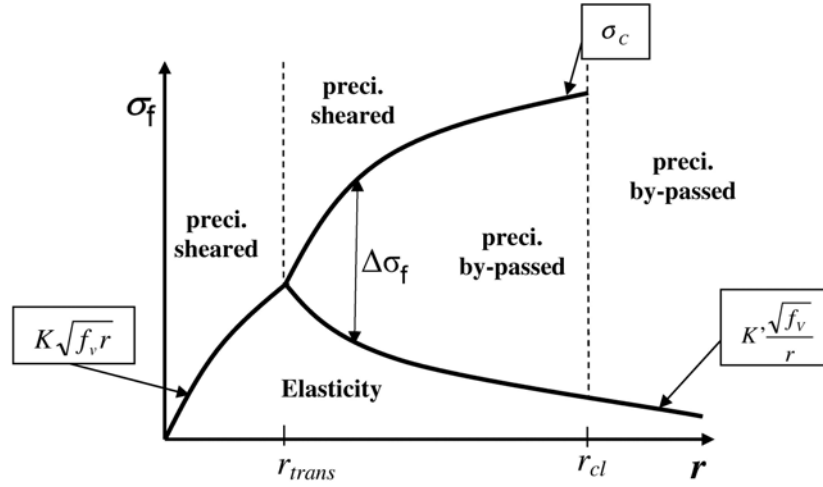


Figure 8.6. Flow stress evolution with the radius including the effect of the Orowan loop stability for coherent precipitates and definition of $\Delta\sigma_f$; r_{trans} is the transition radius for Orowan loops to be formed; r_{cl} is the radius at which the precipitates starts to be incoherent with the aluminum matrix.

the flow stress increases. These are the efficiently stored loops. Two extreme cases can be noticed (see Figure 8.6). These are

- When $r = r_{trans}$, no Orowan loops will be stored since the Orowan loops created will directly annihilate themselves after precipitate shearing with no need for excess plastic deformation. Hence, for $r = r_{trans}$ we have $\varphi = 0$.
- When $r = r_{cl}$, the radius of loss of coherency, all the Orowan loops will be stored. Hence, for $r = r_{cl}$ we have $\varphi = 1$.

Moreover, the efficiency of dislocation storage φ certainly increases with increasing radius. Indeed, the stress $\Delta\sigma_f$ increases with increasing radius (see expression (8.24) and Figure 8.6) which means that a higher stress needs to be attained by strain hardening before the Orowan loops can annihilate themselves by shearing the precipitates. Hence, the storage of Orowan loops becomes more efficient.

For the sake of simplicity, a linear variation will be assumed for φ as a function of r (see Figure 8.7):

- if $r < r_{trans}$ then $\varphi = 0$;

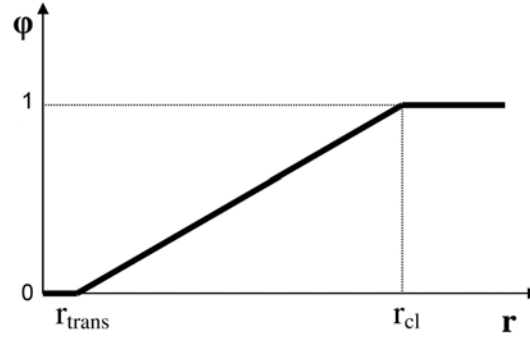


Figure 8.7. Evolution of the efficiency of dislocation storage φ as a function of the equivalent radius r .

- if $r_{trans} < r < r_{cl}$ then

$$\varphi = \frac{r - r_{trans}}{r_{cl} - r_{trans}}; \quad (8.25)$$

- if $r > r_{cl}$ then $\varphi = 1$.

8.2.3 Dynamic precipitation

Precipitation from a supersaturated solid solution (i.e. $C_{Mg} > C_e$) requires atomic transport. Usually this transport occurs by diffusion of chemical species. But, plastic deformation induces also atomic transport, and creates point defects enhancing transport phenomena. This suggests that deforming a supersaturated solid solution can sometimes trigger precipitation, and that the relevant parameter becomes strain accumulation rather than the time. This phenomenon is called "dynamic precipitation".

This dynamic precipitation induces an increase of flow stress while deforming the material since σ_{preci} increases with increasing radius and volume fraction of precipitates. These precipitates are small enough to be sheared by the dislocations. This precipitation and the associated increase in flow stress is expected to increase as the strain increases. The apparent effect of dynamic precipitation on strain hardening will therefore be an increase of the dislocation storage parameter θ by a quantity called θ_{dp} . After Deschamps *et al.* [26], the contributions of dislocation storage and of dynamic precipitation can be added linearly.

We propose a linear relationship between the excess solute and θ_{dp} , i.e.:

$$\theta = \theta_0 + \theta_{dp} \text{ where } \theta_{dp} = k_{\theta dp} \left(\frac{C_{Mg} - C_e}{C_0 - C_e} \right) \quad (8.26)$$

where C_e is the equilibrium concentration of solid solution at room temperature given by expression (7.3), $(C_{Mg} - C_e)/(C_0 - C_e)$ is therefore the relative remaining excess solute (C_{Mg} is the concentration of magnesium in solid solution and C_0 is its maximum value). Note that $C_e < C_{lim}$, where C_{lim} is the limit concentration for the precipitation of GP zones at room temperature. Deforming the material can be the accelerating factor which helps precipitating the excess solid solution ($C_{lim} - C_e$) that the driving force associated to natural aging is not sufficient to achieve.

The value of $k_{\theta dp}$ is calculated from the values of the dislocation storage parameter θ after a solution heat treatment θ_{SHT} and the minimum value of $\theta = \theta_0$:

$$k_{\theta dp} = \theta_{SHT} - \theta_0. \quad (8.27)$$

The experimental results of Table 6.3 (page 94) show that the dislocation storage parameter θ of a solution heat treated sample is unaffected by natural aging. This is apparently in contradiction with the fact that when natural aging has proceeded, the solid solution concentration decreases and hence the potential for dynamic precipitation. We suggest here an explanation to this phenomenon. The dislocation storage parameter θ is defined as the plastic slope ($d\sigma_f/d\varepsilon$) when the flow stress equals the yield strength ($\sigma_f = \sigma_y$), i.e. for a very small plastic strain. This could explain why the solid solution, that did not precipitate by natural aging, will precipitate when supported by a small plastic strain (i.e. σ_f slightly above σ_y), giving a value of the dislocation storage parameter θ similar to that of the supersaturated solid solution before natural aging.

8.2.4 Influence of precipitation on dislocation recovery

The increase of the dynamic recovery rate β when Orowan loops are stored is not fully understood in the scientific literature [15]. This section proposes a novel model that tends to explain and to quantify the drastic increase of the dynamic recovery rate β when Orowan loops are stored.

(a) Effect of the variation in yield strength and solid solution concentration

As extensively discussed in the microstructure and yield strength evolution models, the yield strength and the solid solution concentration are affected when precipitates form. Both have an effect on the recovery rate:

- The dynamic recovery rate β is proportional to the annihilation distance between two dislocations of opposite sign y_a . When the interaction stress between two dislocations (Gb/y_a) exceeds the yield strength, the dislocations will annihilate one another. Hence, if the yield strength increases, the dynamic recovery rate β should decrease. Furthermore, if dynamic precipitation occurs, the yield strength is increased during deformation leading to an additional decrease of the dynamic recovery rate β . It is observed in Figure 6.11 that the dynamic recovery rate β tends to evolve opposite to the yield strength evolution.
- Precipitation and dynamic precipitation causes a depletion of solid solution which can ease the intrinsic mobility of dislocations (increasing the dynamic recovery rate β) out of their plane by modifying the core properties of the dislocation (e.g. increasing the stacking fault energy, which favors cross slip).

The solid solution effect (possibility of dynamic precipitation and modification of the root properties of the dislocation) is difficult to quantify due to its coupling with the yield strength evolution. Indeed, when the solid solution concentration decreases, the yield strength increases (it is an indirect effect related to the precipitation) leading to a potential decrease of the dynamic recovery rate β , but, the decrease in solid solution concentration reduces dynamic precipitation leading thus to a potential increase of the dynamic recovery rate β . This effect will not be modeled here at this level of complexity.

The dynamic recovery rate β is assumed to be inversely proportional to the yield strength σ_y . The reference is the minimum value β_{min} for which the maximum yield strength σ_y^{max} is reached. The expression of the dynamic recovery rate β writes

$$\frac{1}{\beta_{min}} - \frac{1}{\beta} = k_\beta \left(\frac{\sigma_y^{max} - \sigma_y}{G} \right) \quad (8.28)$$

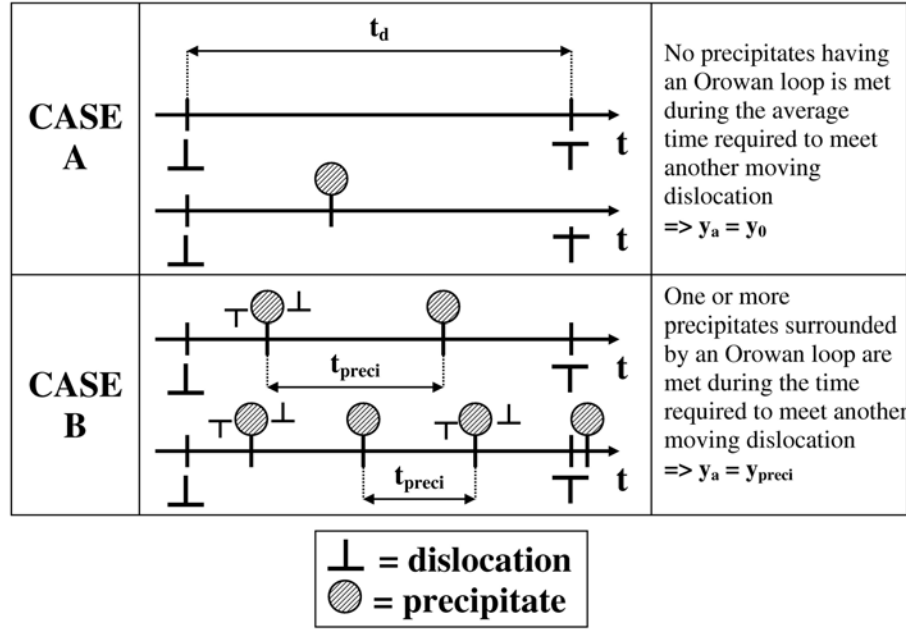


Figure 8.8. Effect of the presence of a precipitate surrounded by an Orowan dislocation loop on the annihilation distance between two moving dislocations (y_a); traveling time associated to the mean spacing between two opposite dislocations t_d [s]; $1/t_{preci}$ [s^{-1}] is the frequency of precipitates distribution.

where k_β is an adimensional constant which can be evaluated by knowing the value of the dynamic recovery rate β for a solution heat treated sample β_{SHT}^{init} having a yield strength equal to σ_{ySHT}^{init} :

$$k_\beta = \frac{G \left(\frac{1}{\beta_{min}} - \frac{1}{\beta_{SHT}^{init}} \right)}{\sigma_y^{max} - \sigma_{ySHT}^{init}} \quad (8.29)$$

(b) Effect of Orowan loops formation on the dislocation recovery rate

Physics of the problem. Figure 8.8 explains the physics of the model. Two cases are possible:

- **Case A.** The mean spacing between two opposite dislocations and associated traveling time t_d [s] is such that they annihilate before encountering a precipitate with an Orowan loop surrounding it. In this case, the annihilation distance with another mobile dislocation is $y_a = y_0$.

tion y_a ¹ is equal to y_0 which is the annihilation distance when no dislocations are stored around precipitates in the form of Orowan loops. In that case, the dynamic recovery rate β is equal to β_0 the value of the dynamic recovery rate β when no Orowan loops are stored.

- **Case B ("The dislocation lift").** At least one precipitate is met during the time t_d and that precipitate has an Orowan loop surrounding it. In that case, the dislocation will partially and locally annihilate with the Orowan loop favoring the change of the dislocation plane. Consequently, the dislocation glide is "de-localized" and the effective annihilation distance with another mobile dislocation y_a will be larger. The apparent recovery rate β will increase to β_{preci} . If the dynamic recovery rate β can be interpreted via the critical annihilation distance y_{preci} at the precipitate/matrix interface, $\beta_{preci} \approx 2y_{preci}/b$ [24] and is then expected to be independent of the radius.

Expression of the dynamic recovery rate β . Case A and B can be both present when deforming an alloy containing precipitates around which Orowan loops are stored. Let $p(0)$ be the probability to have zero precipitates between two dislocations that would potentially annihilate one another. The value of y_a then writes

$$y_a = y_0 p(0) + y_{preci}(1 - p(0)), \quad (8.30)$$

or equivalently, if β_{preci} can be interpreted via the critical annihilation distance y_{preci} :

$$\beta = \beta_0 p(0) + \beta_{preci}(1 - p(0)). \quad (8.31)$$

Estimation of $p(0)$. If we note that

$$t_d = L_0/v_r, \quad (8.32)$$

where L_0 [m] is the mean distance between two mobile dislocations of opposite sign and v_r [m/s] is the relative velocity of two moving dislocations, and

$$t_{preci} = l/v_r, \quad (8.33)$$

where l [m] is given by expression (7.15) and is the precipitates spacing in the glide plane of the dislocations. The mean number ν of precipitates

¹Distance under which two dislocations undergo a force field such that they annihilate.

encountered before two moving dislocations meet is

$$\nu = t_d/t_{preci}. \quad (8.34)$$

Hence the mean number ν_φ of precipitates having an Orowan loop efficiently stored around it encountered before two moving dislocations meet is

$$\nu_\varphi = \nu\varphi, \quad (8.35)$$

where φ is the efficiency of dislocation storage (i.e. the probability of an Orowan loop to survive the deformation process). Consequently we get the following expression for ν_φ :

$$\nu_\varphi = \varphi \frac{t_d}{t_{preci}} = \varphi \frac{L_0}{l} = \sqrt{\frac{3}{2\pi}} \frac{\sqrt{f_v} L_0 \varphi}{r}. \quad (8.36)$$

Using a Poisson probability distribution, we can calculate $p(m)$ as the mean probability to meet m precipitates having an Orowan loop surrounding it during the time t_d knowing the mean number ν_φ of precipitates having an Orowan loop efficiently stored around it and encountered before two moving dislocations meet:

$$p(m) = \frac{\nu_\varphi^m}{m!} \exp(-\nu_\varphi). \quad (8.37)$$

Hence, the probability $p(0)$ to meet no precipitate having an Orowan loop surrounding it during the time t_d is given by (using expression (7.15) to calculate l):

$$p(0) = \exp(-\nu_\varphi) = \exp\left(-\sqrt{\frac{3}{2\pi}} \frac{\sqrt{f_v} L_0 \varphi}{r}\right). \quad (8.38)$$

8.3 Extended KME model

8.3.1 Formulation

Formulation of the dislocation storage parameter θ . The extra term proposed by Estrin (expression (8.12)) is now multiplied by the efficiency of dislocations storage φ in order to take into account the stability of the Orowan loops.

$$\frac{d\rho}{d\gamma_p} = k_1 \sqrt{\rho} + \frac{\varphi}{bl} - k_2 \rho. \quad (8.39)$$

Expression (8.18) can be modified by

- taking the efficiency of dislocations storage φ (equation (8.26)) into account i.e. by multiplying the term in $(1/r)$ by φ ;
- taking dynamic precipitation into account, i.e. by adding θ_{dp} to the expression of the dislocation storage parameter θ ;
- and, evaluating the mean plastic stress by $\theta/(2\beta)$.

Hence, the hardening rate writes

$$\theta = \theta_0 + \theta_{dp} + \alpha^2 G b M^3 \left(\frac{\beta}{\theta} \right) \left(\frac{\varphi}{bl} \right). \quad (8.40)$$

The dislocation storage rate is then given by

$$\frac{\theta}{G} = \frac{\theta_0 + \theta_{dp}}{G} + \alpha^2 G M^3 \varphi \frac{\beta}{\theta} \sqrt{\frac{3f_v}{2\pi}} \left(\frac{b}{r} \right), \quad (8.41)$$

Hence,

$$\frac{\theta}{G} = \frac{\theta_0 + \theta_{dp}}{2G} + \sqrt{\left(\frac{\theta_0}{2G} \right)^2 + \alpha^2 M^3 \beta \varphi \left(\frac{b}{r} \right) \sqrt{\frac{3f_v}{2\pi}}}. \quad (8.42)$$

with θ_{dp} given by

$$\theta_{dp} = k_{\theta dp} \left(\frac{C_{Mg} - C_e}{C_0 - C_e} \right), \quad (8.43)$$

and φ given by the expression plotted in Figure 8.7

- if $r < r_{trans}$ then $\varphi = 0$;
- if $r_{trans} < r < r_{cl}$ then $\varphi = \frac{r - r_{trans}}{r_{cl} - r_{trans}}$;
- if $r > r_{cl}$ then $\varphi = 1$.

Formulation of the dynamic recovery rate β . The dynamic recovery rate is obtained by combining expressions (8.31) and (8.38):

$$\beta = \beta_0 \exp \left(-\sqrt{\frac{3}{2\pi}} \frac{\sqrt{f_v} L_0 \varphi}{r} \right) + \frac{2y_{preci}}{b} \left(1 - \exp \left(-\sqrt{\frac{3}{2\pi}} \frac{\sqrt{f_v} L_0 \varphi}{r} \right) \right) \quad (8.44)$$

with β_0 given by expression (8.28) and corresponding to the dynamic recovery rate β when no Orowan loops are stored

$$\frac{1}{\beta_{min}} - \frac{1}{\beta_0} = k_\beta \frac{\sigma_y^{max} - \sigma_y}{G} \quad (8.45)$$

and φ given by the expression plotted in Figure 8.7

- if $r < r_{trans}$ then $\varphi = 0$;
- if $r_{trans} < r < r_{cl}$ then $\varphi = \frac{r - r_{trans}}{r_{cl} - r_{trans}}$;
- if $r > r_{cl}$ then $\varphi = 1$.

8.3.2 Qualitative consequences

Figure 8.9 shows the differences between the classical KME model and the extended version proposed here, showing the effect of each individual contribution.

Under-aged conditions ($r < r_{trans}$). When a material is in an under-aged state, no Orowan loops are stored (i.e. $\varphi = 0$) since the dislocations are in a regime of precipitates shearing; the dislocation storage parameter θ is artificially increased due to dynamic precipitation; the dynamic recovery rate β varies proportionally to the inverse of the yield strength.

Over-aged conditions ($r > r_{trans}$). When a material is in an over-aged state, Orowan loops are stored (i.e. $\varphi > 0$). The efficiency of Orowan loops storage gradually increases with precipitate radius causing a smoother transition in the dislocation storage parameter θ from its lowest value (θ_0) to its maximum when the radius reaches the precipitate radius of loss of coherency r_{cl} . The evolution of the dynamic recovery rate β is similar due to the "dislocation lift" effect caused by the presence of Orowan loops on part of the precipitates.

8.3.3 Model parameters

Table 8.1 gives the values of the model parameters. This section discusses the identification procedure.

(a) Identified parameters

θ_0 . The dislocation storage parameter θ is only slightly different from its value in pure aluminum when all the alloying elements have precipitated in the form of coherent precipitates that are sheared by dislocations. Experiments found in the literature [37] confirm this. The value of 600 MPa used here (minimum in Figure 6.11) is only slightly lower than the theoretical value of the dislocation storage parameter for pure aluminum $G/30$ - $G/20$ (900 – 1350 MPa) [4] but the order of magnitude is similar.

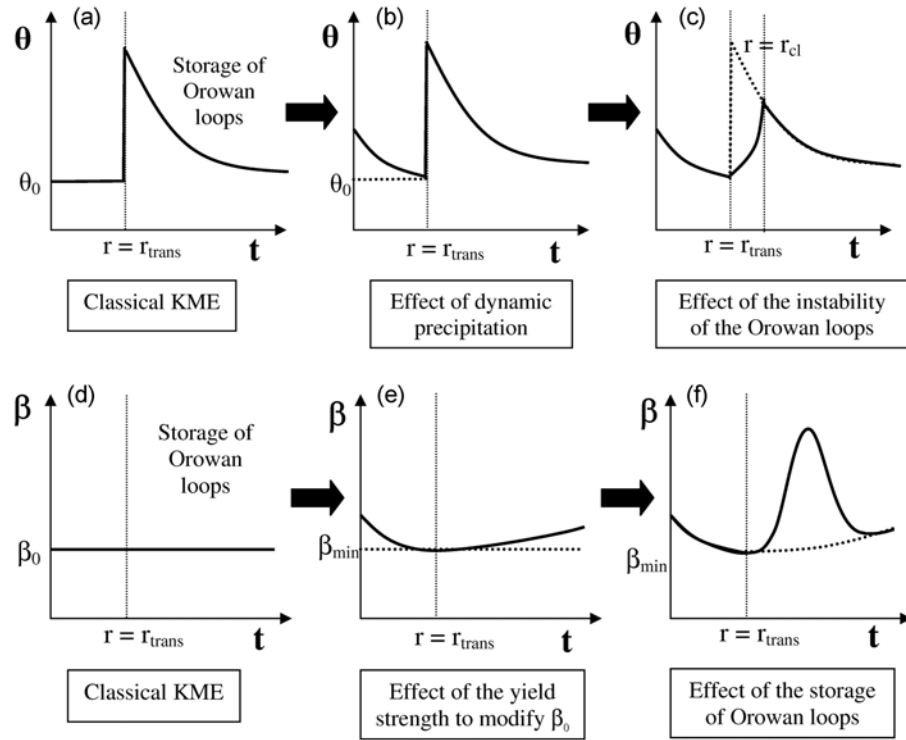


Figure 8.9. Schematic variation of the effect of the different changes in the extended KME model (c,f) with respect the classical KME model (a,d). **Dislocation storage parameter θ :** the extended KME model introduces the effect of dynamic precipitation (b) which increases the apparent dislocation storage parameter θ when the solid solution concentration exceeds the equilibrium concentration; the extended KME model also introduces the effect of the instability of Orowan loops (c) to reduce the dislocation storage rate for precipitate radii slightly larger than the transition radius r_{trans} . **Dynamic recovery rate β :** the extended KME model introduces the effect of the yield strength on the annihilation distance and hence on dynamic recovery (e); the extended KME model also introduces the "dislocation lift" effect if Orowan loops are efficiently stored around precipitates (f).

Param.	Significance	Value	Origin
θ_0	θ if no dynamic precipitation and no Orowan loops	600 MPa	θ minimum of Figure 6.11(e)
β_{min}	β when σ_y is minimum	7.5	β minimum of Figure 6.11(f)
$k_{\theta dp}$	Constant for the dynamic precipitation effect on θ	1270 MPa	eq. 8.27 Table 6.3 (pg 94)
k_β	Constant linking the yield strength to β	9.3	eq. 8.29 Table 6.3, pg 94
r_{cl}	Radius of loss of coherency	25 nm	r of θ and β transition
L_0	Distance between two moving dislocations of opposite sign	100 nm	eq. 8.46 and α , θ_0 and β_{min}
α	Constant linking the dislocation density to the shear stress	0.21	Optimized on θ of HT for $r > r_{trans}$
y_{preci}	Annihilation distance when at least one precipitate is between two dislocations of opposite sign	22 nm	Optimized on β of HT for $r > r_{trans}$

Table 8.1. Summary of the parameters for the strain hardening model (HT = heat treated samples)

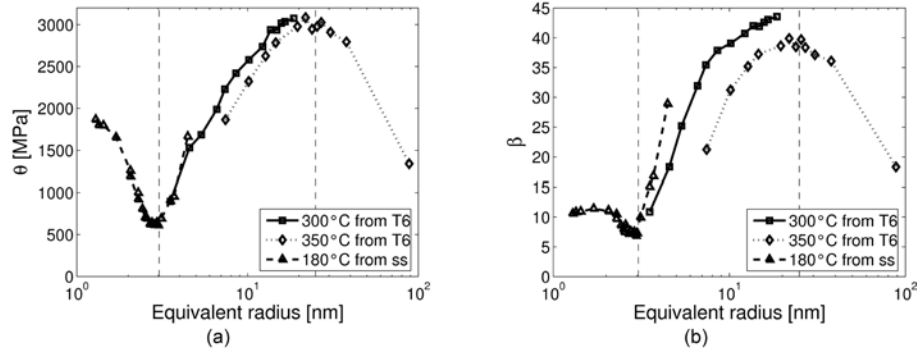


Figure 8.10. Evolution of the experimental strain hardening parameters with the modeled equivalent radius r for heat treated samples: (a) the dislocation storage parameter θ and (b) the dynamic recovery rate β (SS = supersaturated solid solution). The vertical lines indicate the position of the two transition radius r_{trans} and r_{cl} . The value for r_{cl} is identified based on these experimental results.

r_{cl} . This radius is identified from the tensile tests on heat treated specimens of base alloy number 1. Figure 8.10 shows that both the dislocation storage parameter θ and the dynamic recovery rate β reach a maximum for an equivalent radius $r_{cl} \approx 25$ nm. This value of the radius at which the precipitate loses its coherency with the matrix, is within the expected range.

L_0 . The mean characteristic distance between two dislocations of opposite sign can be estimated from the dislocation density ($L_0 = 1/\sqrt{\rho}$) when only mobile dislocations are present and no dynamic precipitation artificially changes the dislocation storage parameter θ and the dynamic recovery rate β . The dislocation density changes when deformation proceeds but, for the sake of simplicity, a mean value will be assumed. The dislocation density can be estimated by the increase of the plastic flow stress due to strain hardening as given by expression (8.1). The plastic flow stress varies between 0 and $(\sigma_{sat} - \sigma_y)$ giving a mean value of $((\sigma_{sat} - \sigma_y)/2)$. The values of the dislocation storage parameter θ and the dynamic recovery rate β required to calculate $(\sigma_{sat} - \sigma_y)$ are the values when no Orowan loops are stored and no apparent effect of dynamic precipitation changes the dislocation storage parameter θ and the dynamic recovery rate β , i.e. θ_0 and β_{min} .

$$L_0 = \alpha G b M \frac{2\beta_{min}}{\theta_0}. \quad (8.46)$$

This gives a value of L_0 of the order of magnitude of 100 nm for the classical range of α parameter (i.e. 0.2 - 0.5).

(b) Fitted parameter α and y_{preci}

The value of α was identified on the experimental values of the dislocation storage parameter θ for heat treated samples (see figure 6.11(e) and 6.12(e)) using the microstructure evolution model in order to estimate r , C_{Mg} and f_v and the experimental values of the dynamic recovery rate β . A value α equal to 0.21 gives a minimum error on the predictions of the dislocation storage parameter θ , equal to 16% for the heat treated samples. This value of α is in an acceptable range (0.2 - 0.5).

The value of y_{preci} was identified on the experimental values of the dynamic recovery rate β for heat treated samples (see figure 6.11(f) and 6.12(f)) using the microstructure evolution model in order to estimate r , C_{Mg} and f_v . A value y_{preci} equal to 22 nm gives a minimum error

on the predictions of the dynamic recovery rate β , equal to 14% for the heat treated samples. This value of y_{preci} seems reasonable since:

- y_{preci} is much higher than the critical distance for dislocation annihilation when no Orowan loops are present ($y_0 \approx \beta_{min} b/2 = 1$ nm [24]) which confirms the importance of the effect of precipitation on the recovery mechanism.
- y_{preci} is in the range of the β' precipitates length that can be reached when over-aging occurs, i.e. the dislocations have the capacity to de-localize a distance of the order of the precipitates length.

8.3.4 Modeling of isothermal heat treatments

Under-aged conditions. Figure 8.11 shows the evolution of the dislocation storage parameter θ and the dynamic recovery rate β for under-aged conditions ($r < r_{trans}$). The predictions of the dynamic recovery rate β have been obtained using the experimental values of the yield strength σ_y since no model for the predictions of the yield strength of samples naturally aged prior to artificial aging is available. The predictions of the dislocation storage parameter θ use the results of the microstructure evolution model presented in Figure 7.4. The predictions of the dislocation storage parameter θ are of good quality. Hence, this confirms that the effect of dynamic precipitation on the dislocation storage rate can be very simply taken into account through a linear relationship with the excess solute concentration. The predictions for the dynamic recovery rate β are slightly less satisfactory but the tendencies are well reproduced confirming the influence of the yield strength on the dynamic recovery rate. It has already been mentioned that the effect of the solid solution content is not yet clear and could influence the dynamic recovery rate. Indeed, the solid solution concentration is higher in a partially naturally aged sample (time = 0) than in a peak aged sample (time = 10 h) which could explain the approximative predictions.

Over-aged conditions. Figure 8.12 shows the evolution of the dislocation storage parameter θ and the dynamic recovery rate β for over-aged conditions ($r > r_{trans}$). The predictions for both the dislocation storage parameter θ and the dynamic recovery rate β are based on the results of the microstructure evolution model presented in Figure 7.4. The predictions are of relatively good quality. The slower increase with time of the dislocation storage parameter θ compared to the classical KME approach is correctly reproduced by the introduction of the efficiency of

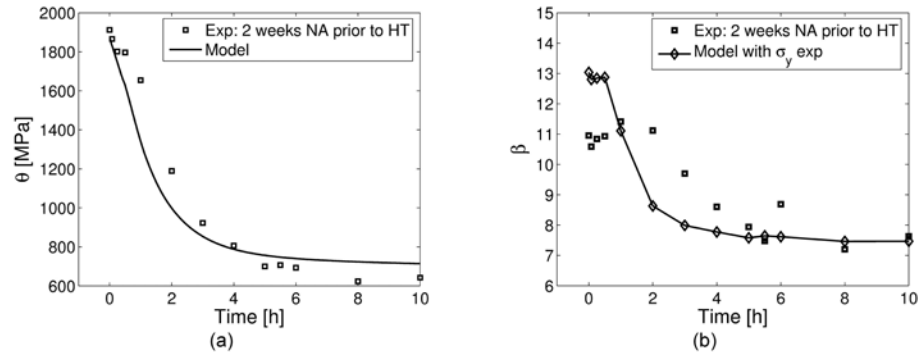


Figure 8.11. Assessment of the model predicting (a) the dislocation storage parameter θ and (b) the dynamic recovery rate β for samples heat treated at 180°C from a naturally aged state (2 weeks after solution heat treatment) for under-aged conditions. The predictions for the dynamic recovery rate β are based on the experimental measurements of the yield strength σ_y .

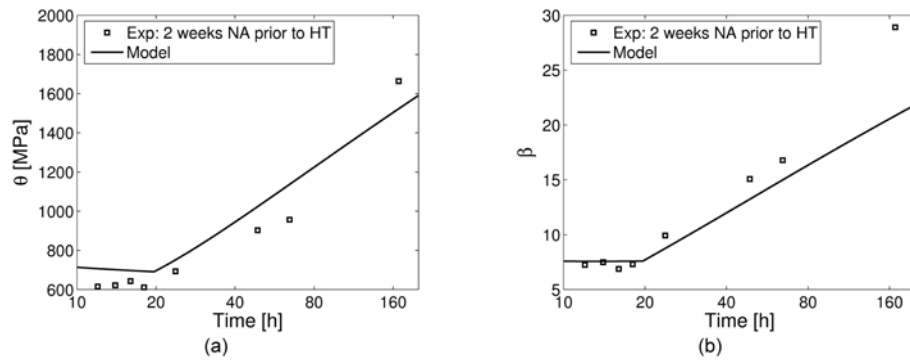


Figure 8.12. Assessment of the model predicting (a) the dislocation storage parameter θ and (b) the dynamic recovery rate β for samples heat treated at 180°C from a naturally aged state (2 weeks after solution heat treatment) for over-aged conditions.

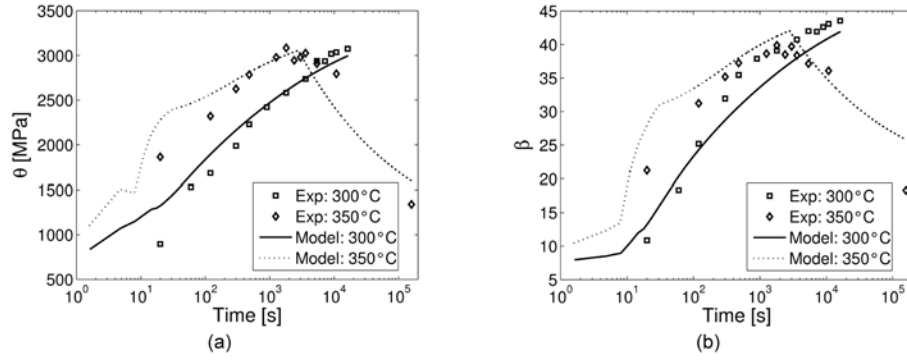


Figure 8.13. Assessment of the model predicting (a) the dislocation storage parameter θ and (b) the dynamic recovery rate β for samples heat treated at 300°C and 350°C from a T6 state.

dislocation storage reflecting the instability of Orowan loops around coherent precipitates. The experimental increase of the dynamic recovery rate β is greater than β_0 (β with only the term inversely proportional to the yield strength) and this is well reproduced by the model proposed here.

Massive over-aging. Figure 8.13 shows the evolution of the dislocation storage parameter θ and the dynamic recovery rate β for over-aged conditions with r evolving very rapidly since the heat treatment temperature is much higher (300°C and 350°C). The predictions for both the dislocation storage parameter θ and the dynamic recovery rate β are based on the results of the microstructure evolution model presented in Figure 7.5 in order to estimate the mean equivalent radius of the precipitates r , the solid solution concentration C_{Mg} and the precipitates volume fraction f_v . The conspicuous agreement between predictions and experimental results show that the strain hardening model correctly treats massive over-aging and in particular the drop of both strain hardening parameters when large precipitates no more coherent with the aluminum matrix (i.e. $r > r_{cl}$) are observed. Note that contrarily to the results of Figure 8.12, the predictions of the dislocation storage parameter θ in Figure 8.13(a) couple the stability of Orowan loops effect and the dynamic precipitation effect since, at these heat treatment temperatures, partial dissolution and coarsening occur simultaneously.

8.4 Application to the predictions of the flow properties

8.4.1 Considere criterion

The ultimate tensile strength R_m [MPa] and the strain at necking ε_u can be simply deduced from the predictions of the dislocation storage parameter θ and the dynamic recovery rate β . Indeed, using relationship (8.10) (which provides the plastic slope during deformation) and the Considere's necking condition (which states that, at necking, the plastic slope is equal to the flow stress) leads to

$$\frac{d\sigma_f}{d\varepsilon_p} = \sigma_f = \theta - \beta(\sigma_f - \sigma_y). \quad (8.47)$$

The true stress at necking σ_u is then simply related to the yield strength σ_y , the dislocation storage parameter θ and the dynamic recovery rate β by the following relationship

$$\sigma_u = \frac{\theta + \beta\sigma_y}{1 + \beta}. \quad (8.48)$$

Using the Voce law (equation (8.11) with β and θ replacing respectively β_0 and θ_0) and the above expression of the true stress at necking σ_u (equation (8.48)), the strain at necking ε_u is found to be:

$$\varepsilon_u = \frac{-1}{\beta} \ln \left(1 - \frac{\beta}{\theta} (\sigma_u - \sigma_y) \right). \quad (8.49)$$

The ultimate tensile strength is given by: $R_m = \sigma_u \exp(-\varepsilon_u)$ and can then be calculated from expression (8.48) and (8.49) as a function of the dislocation storage parameter θ and the dynamic recovery rate β .

8.4.2 Comparison between experiments and predictions

The predictions of the ultimate tensile strength R_m and the strain at necking ε_u are shown in Figure 8.14 for the heat treated samples. As for the predictions of the dynamic recovery rate β , the predictions of ε_u and R_m for the under-aged conditions use the experimental results for the yield strength σ_y .

Under-aged conditions. The predictions of the true strain at the onset of necking ε_u and the ultimate tensile strength R_m give the right tendencies for under-aged samples (see Figure 8.14(a,b)) but since the

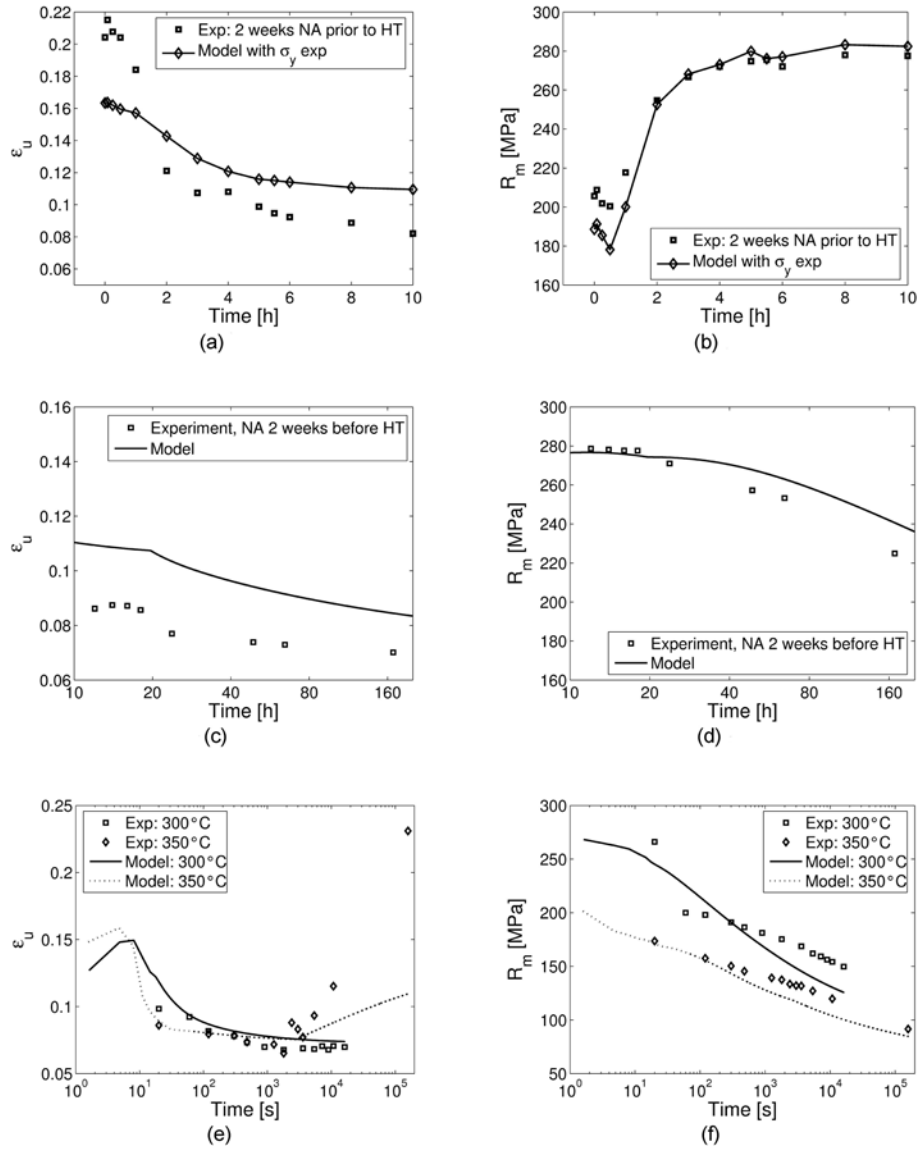


Figure 8.14. Assessment of the model for the predictions of (a,c,e) the true strain at the onset of necking ε_u and (b,d,f) the ultimate tensile strength R_m . (a,b) Under-aged conditions for samples heat treated at 180°C from naturally aged state (2 weeks); the model makes use of the experimental values of the yield strength σ_y ; (c,d) over-aged conditions for samples heat treated at 180°C from naturally aged state (2 weeks); (e,f) samples heat treated at 300°C and 350°C from T6 state.

dynamic recovery rate β is not perfectly predicted as shown in Figure 8.11(b) the predictions of R_m and ε_u cannot be perfect either. The true strain at the onset of necking ε_u seems to be much more affected by these inaccuracies than the ultimate tensile strength R_m . The main conclusion relative to the physical effects affecting the evolution of ε_u and R_m for under-aged conditions when aging proceeds are the followings.

- The yield strength increase with aging time affects ε_u and R_m directly and indirectly (through the dynamic recovery rate decrease with increasing yield strength).
- The solid solution concentration decreases with aging time decreases the potentiality for dynamic precipitation. Hence, the apparent dislocation storage rate decreases and the dynamic recovery rate is certainly affected but this effect is not included in the model.

Over-aged and massively over-aged conditions. The predictions of R_m for over-aged samples (see Figure 8.14(c,d,e,f)) are of the same quality as the predictions of the yield strength σ_y given in Figure 7.4(c) and Figure 7.5(c) which confirms the validity of the strain hardening model especially for over-aged conditions. In general, the strain at necking is slightly overestimated. The main conclusion relative to the physical effects affecting the evolution of ε_u and R_m for over-aged conditions when aging proceeds are the followings.

- The yield strength decrease with aging time affects ε_u and R_m directly and indirectly (through the dynamic recovery rate increases with decreasing yield strength).
- Orowan loops are stored with an increasing efficiency when the aging time increases (greater radii). Hence, ε_u and R_m are affected by the increase of the dislocation storage parameter θ and the decrease of the dynamic recovery rate β .
- The loss of coherency between the precipitates and the matrix and the ensuing perfect storage of Orowan loops cause an increase of ε_u but do not seem to affect very much the ultimate tensile strength R_m .
- For partially dissolved samples (over-aging at 300°C and 350°C from T6 state, Figure 8.14(e,f)), the evolution of solid solution concentration (see Figure 7.5(a)) affects the potential for dynamic precipitation.

8.5 Application to welds: local work hardening

8.5.1 General framework

The prediction of the strain hardening behavior of the different regions of a weld is fundamental for the understanding of the global behavior of friction stir welds. Indeed, the high level of the ultimate tensile strength observed in the center of the weld compared to the heat affected zone (HAZ) tremendously affects the global behavior and the plastic localization when submitting a welded sample to transverse loading. This has provided the impetus for the development of a model capable of taking the microstructural evolution predicted in chapter 7 into account. This has been done with success in the previous sections for homogeneous heat treated samples and will now be applied to friction stir welds.

8.5.2 Prediction of local work hardening

Figure 8.15(a,b) shows the evolution of the predicted strain hardening parameters compared to the measured ones (from Figure 6.23(e,f)) for a friction stir weld using the model presented in this chapter and the microstructure parameters predicted by the model developed in chapter 7 reproduced in Figure 8.15(c). Modeling of the weld center is not possible at this stage, due to the unknown experimental temperature history (no thermocouples can be placed in that region of the weld). The experimental trends are quite well reproduced by the model.

Looking at the dislocation storage parameter θ the trends can be explained as follows (see Figure 8.15(a,c)):

- The lowest level of the dislocation storage parameter θ is obtained in the base material, which is in a T6 state. This is consistent with the results of heat treated samples for which it was shown that if the solid solution concentration is low and no Orowan loops are stored (the case in a T6 state aluminum alloy where $r = r_{trans}$) then the dislocation storage parameter θ is equal to its minimum value θ_0 .
- When moving toward the heat affected zone (HAZ), the mean precipitates radius drastically increases as well as the solid solution content. Hence, some Orowan loops start being stored with an increasing efficiency and dynamic precipitation is made possible. Those two effects add up to increase the level of the dislocation storage parameter θ .

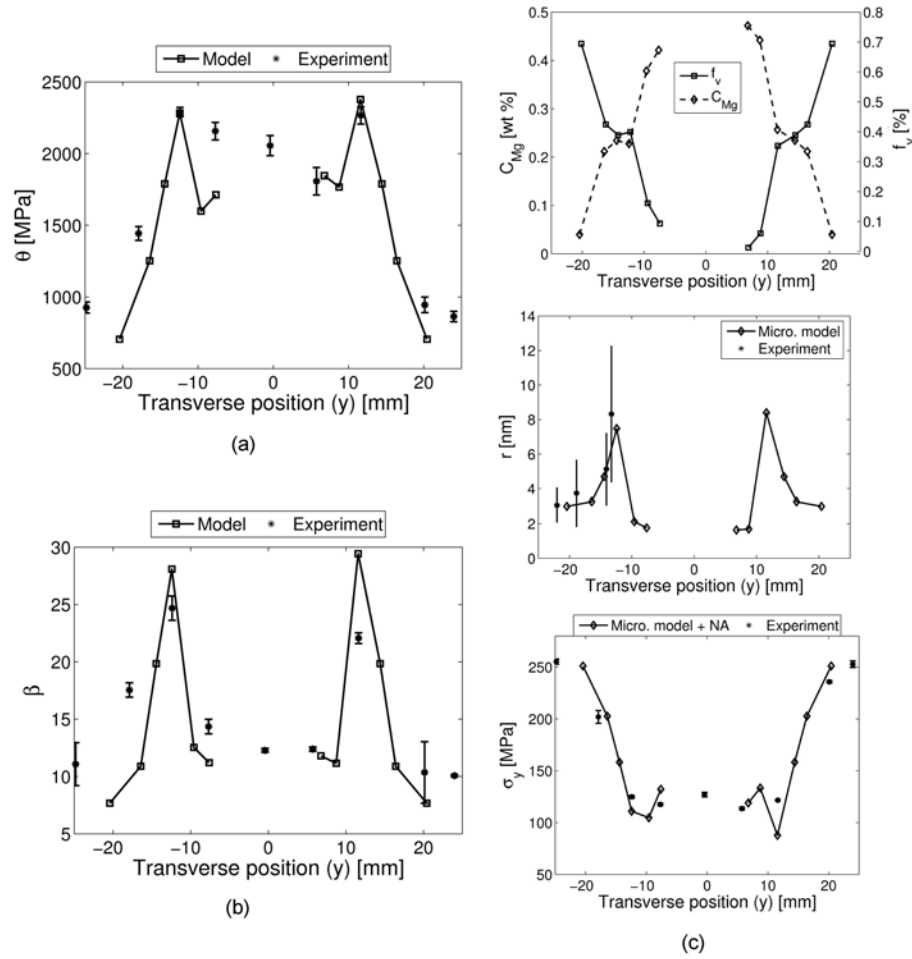


Figure 8.15. Prediction of (a) the dislocation storage parameter θ (dislocation storage rate) and (b) the dynamic recovery rate β for a friction stir weld with welding parameters 200 mm/min and 1000 rpm. (c) Microstructure and yield strength evolution results of chapter 7.

- When moving towards the weld center, the dislocation storage parameter θ decreases but in a relatively limited extent. In that region, all the elements are in solid solution (at least, that is what is predicted by the microstructure evolution model). So, even though no Orowan loops are stored, dynamic precipitation is maximum, leading to a high value of the dislocation storage parameter θ . Note that the presence of $\beta(\text{Mg}_2\text{Si})$ precipitates in the weld center might affect the value of the dislocation storage parameter θ in that region. Nevertheless, it has been shown that the value of the dislocation storage parameter θ drastically drops for large incoherent precipitates giving only a limited contribution of $\beta(\text{Mg}_2\text{Si})$ precipitates to the dislocation storage parameter θ in the weld center. Hence, the main effect would probably be to reduce dynamic precipitation due to a poorer solid solution. On that basis, the advancing side is probably the side of the weld with the larger content of $\beta(\text{Mg}_2\text{Si})$ precipitates (hence having the lowest remaining solid solution concentration). This confirms again the need for better understanding the precipitation of $\beta(\text{Mg}_2\text{Si})$ in the weld center.

Looking at the dynamic recovery rate β the trends can be explained as follows (see Figure 8.15(b,c)):

- The lowest level of the dynamic recovery rate β is obtained in the base material, which is in a T6 state. This is due to the fact that the yield strength is maximum in that state and no Orowan loops are stored around precipitates, so no "dislocation lift" effect is present.
- When moving towards the heat affected zone (HAZ) the mean precipitate radius drastically increases causing the storage of Orowan loops with a relatively high efficiency. The consequence is a drastic increase of the dynamic recovery rate β due to the "dislocation lift" effect. The decrease of the yield strength favors even more the increase of the dynamic recovery rate β .
- When moving towards the weld center, the dynamic recovery rate β decreases drastically. In that region, the mean precipitate radius is lower than the transition radius r_{trans} and hence no "dislocation lift" effect can occur. The level of the dynamic recovery rate β in that region is still slightly higher than in the base material due to the low yield strength level in that region of the weld.

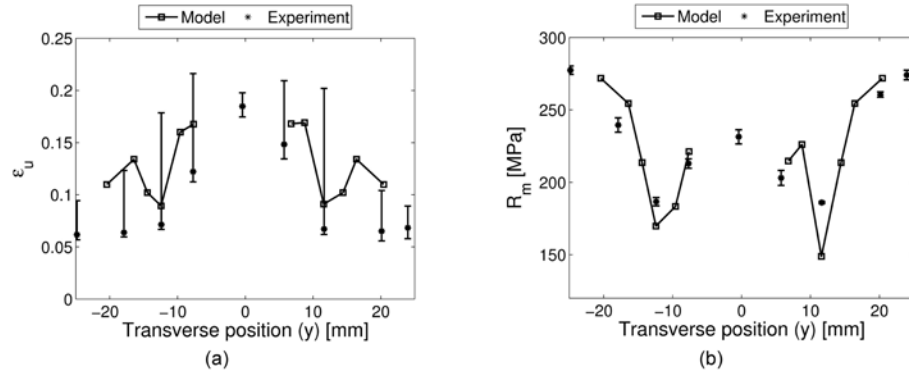


Figure 8.16. Prediction of (a) the strain at necking ε_u and (b) the ultimate tensile strength R_m for a friction stir welds with welding parameters 200 mm/min and 1000 rpm.

8.5.3 Prediction of local ductility

Figure 8.16(a) shows the evolution of the strain at necking ε_u for a friction stir weld comparing predicted and experimental results. We must remember here that premature necking occurred due to machining imprecisions. This effect was corrected in chapter 6 by enlarging the error bars up to values satisfying Considere's criterion. Consequently, the fact that the predictions of ε_u are generally in the upper limit of these error bars is justified. The variation of ε_u is relatively well captured, in particular the increase in true strain at the onset of necking in the weld center. It has been shown in Figure 8.14(a) that under-aged conditions (which is the case in the weld center) give values of ε_u higher than over-aged conditions (the case in the heat affected zone) and that peak aged conditions (the base material) lead to a similar level of ε_u than over-aged conditions (the case in the heat affected zone).

Figure 8.16(b) shows the evolution of the ultimate tensile strength R_m for a friction stir weld comparing predicted and experimental results. The trends are correctly reproduced, and, in particular, the measured increase in ultimate tensile strength in the weld center compared to the heat affected zone (HAZ). This effect can now be clearly understood and attributed to the high level of apparent dislocation storage rate θ due to dynamic precipitation and to the relatively low level of the dynamic recovery rate β in the weld center (in particular due to natural aging). The heat affected zone has a much lower level of ultimate tensile strength R_m due to a similar (only slightly higher) level of dislocation storage

parameter θ compared to the weld center but, contrarily to the weld center, a much higher level of dynamic recovery due to the "dislocation lift" effect which keeps the flow stress low during deformation.

8.5.4 Comparison between two welding conditions

Figure 8.17 shows the evolution of the dislocation storage parameter θ and the dynamic recovery rate β and the ultimate tensile strength comparing a cold (1000 mm/min) and a hot (200 mm/min) weld. The general effect of welding parameters are well reproduced by the strain hardening model. The values of the dislocation storage parameter θ and the dynamic recovery rate β are slightly underestimated in the heat affected zone (HAZ) of the coldest weld (see Figure 8.17(a)) due to the underestimation of the mean precipitate radius by the microstructure evolution model (see Figure 7.7(e)). The hottest weld did not present this underestimation of the mean precipitate radius and hence the prediction of the strain hardening parameters are much better, see Figure 8.17(b).

The difference in strain hardening variations between the cold weld compared to the hot weld heat affected zones (HAZ) is due to the tremendous difference in the variation of the mean precipitate radius for similar volume fractions of precipitates (see Figure 7.7(c-f)). The rate of dislocation storage parameter θ is very similar in hot and cold welds heat affected zones but still a small increase is observed in hot welds which have a greater particle radius in the HAZ. This increase of the mean precipitate radius for hot welds HAZ causes a larger increase of the dynamic recovery rate β . Hence, the heat affected zone of a hot weld has a lower ultimate tensile strength R_m . This effect is slightly exaggerated by the model due to the underestimation of the mean precipitate radius in the cold weld. This conclusion will certainly have a major impact on the predictions of the global tensile behavior of friction stir welds since deformation localizes in the heat affected zone of friction stir welds.

8.6 Conclusions

An extension of the classical KME model for predicting the strain hardening capacity of aluminum alloys (in particular the 6005A) has been proposed. The different experimentally observed effects have first been analyzed and then modeled using simple analytical expressions based on the physics of dislocation and precipitate/dislocation interactions. The following effects have been found to significantly affect the evolution of the strain hardening parameters:

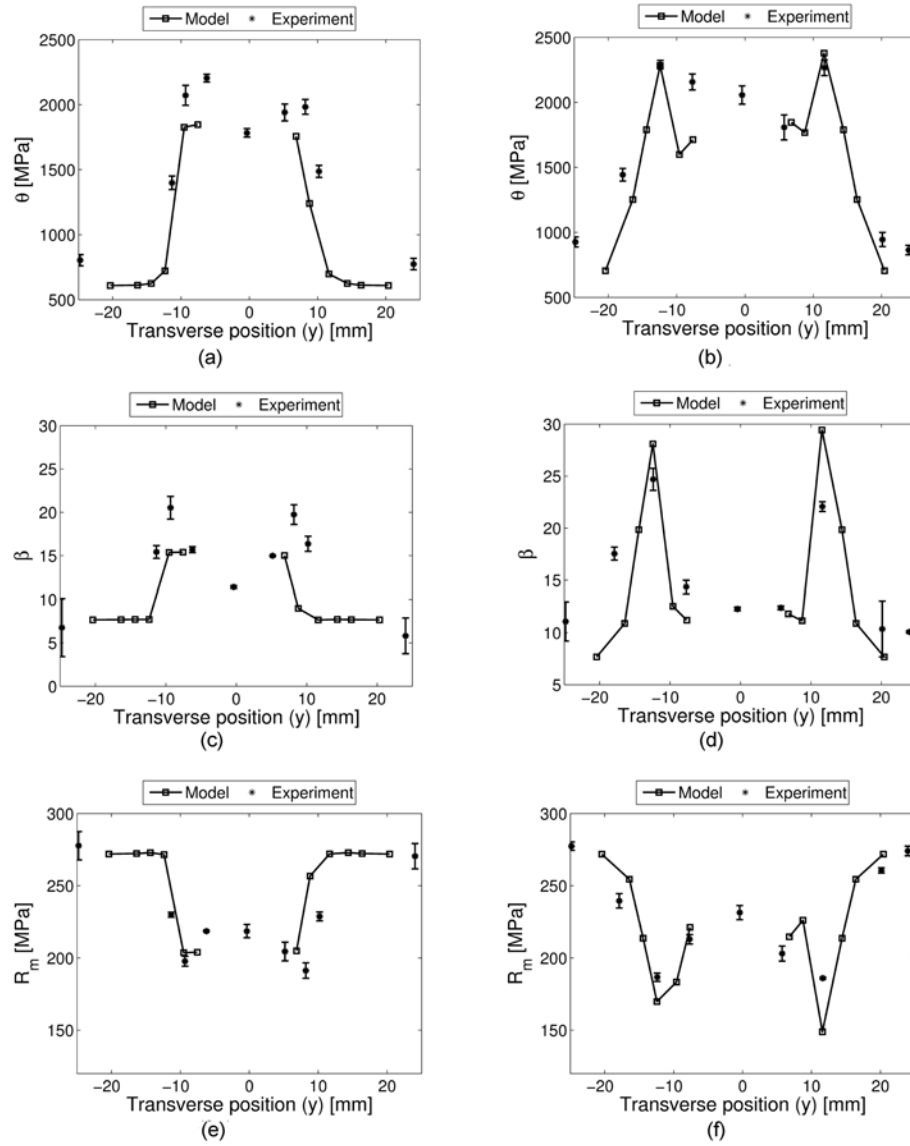


Figure 8.17. Prediction of (a,b) the dislocation storage parameter θ ; (c,d) the dynamic recovery rate β and (e,f) the ultimate tensile strength R_m for friction stir welds with welding conditions 1000 rpm and (a,c,e) 1000 mm/min (b,d,f) 200 mm/min.

- the instability of the Orowan loops for precipitates coherent with the matrix;
- dynamic precipitation;
- the effect of precipitation on dynamic recovery ("dislocation lift" effect and yield strength effect).

The α constant and the annihilation distance for two moving dislocations near precipitates had to be identified based on results obtained on heat treated tensile samples. The predictions have been compared to the resulting strain hardening properties of heat treated tensile samples and micro-tensile samples extracted from friction stir welds for which the microstructure model has been applied in chapter 7. Good agreement was found between predictions and the quite comprehensive set of experimental results.

Part IV

Integrated model and conclusions

ABSTRACT

The previous parts of this thesis have allowed understanding the influence of the thermal cycles on the microstructure, and how these microstructural evolutions affect the tensile properties of the various zones of a friction stir weld. The aim of this part of the thesis is to understand how the local tensile properties affect the global behavior of a friction stir weld, including damage, when subjected to transverse tensile testing. Chapter 10 aims at assembling all the models in chain in order to predict the transverse tensile properties of friction stir welds from the welding parameters. Hence, this model chain offers a process optimization tool and could be inserted in a model of the structure assessment for industrial applications.

The model of a transverse tensile sample uses the local tensile properties as input tensile properties of the finite element code. Chapter 9 uses as local tensile properties those measured in the various zones of a friction stir weld, while chapter 10 uses the predicted tensile properties as a function of the position along the transverse section of the welds. The micro-mechanically based damage model presented in chapter 9 is completely predictive considering that the volume fraction and shape of the coarse α phases of the base material are known and that these phases are unaffected by the welding process (see chapter 6).

The tensile strength of the welds increases with increasing advancing speed mainly due to the higher strength of the heat affected zone of a weld when the advancing speed increases. The elongation tends to decrease when the advancing speed increases mainly due to the decrease of the width of the heat affected zone when the advancing speed increases. Nevertheless, a maximum of the elongation can be observed for medium advancing speeds (500 mm/min and 750 rpm).

The slower deformation localization in a high advancing speed weld compared to a low advancing speed weld can be linked to the difference in strength between the heat affected zone and the weld center. Indeed, it has been shown in part III of this thesis that the strength mismatch between these zones decreases with increasing advancing speed.

No clear effect of the welding conditions has been found on the true fracture strain. This can be explained by the compensation of two antagonistic effects:

- When the advancing speed increases, as mentioned above, the strength mismatch between the weld center and the heat affected zone decreases and hence the maximum stress triaxiality decreases, increasing the true fracture strain.
- When the advancing speed increases, the width of the heat affected zone decreases and hence the maximum stress triaxiality increases, decreasing the true fracture strain.

The fracture of a transverse tensile sample occurs on the retreating side of the weld even if the difference between the advancing side and the retreating side is less marked early in the deformation process for low advancing speed welds. The measured minimum local yield strength is always found on the retreating side of the weld in the heat affected zone. Assembling the models helped understand why the retreating side is the weakest side of a weld. Indeed, the retreating side of the weld presents a higher precipitate radius and lower solid solution concentration (out of favor of natural aging) compared to the advancing side of the weld. Nevertheless, the volume fraction of precipitates is predicted to be higher on the advancing side of the weld than on the retreating side of the weld. Assembling the models still gives correct predictions of the fracture location for low advancing speed welds.

Chapter 9

Global mechanical properties of the welds

In this chapter, the transverse tensile properties of friction stir welds will be related to the local tensile properties that were measured as presented in chapter 6 and predicted through modeling as presented in chapters 7 and 8. The first section of this chapter will present and discuss the experimental results of the transverse tensile tests on friction stir welds obtained with different welding parameters. For some of these welds, digital image correlation was used to characterize the local strains during the tensile test.

In the second part of this chapter, the link between the local and global mechanical properties including damage is also made through a full 3D finite element model of the joint. This model is assessed by using the local tensile properties measured with the micro-tensile tests presented in chapter 6. The parameters for the damage model presented in that part are based on the characterization of the coarse α phases responsible for damage initiation also presented in chapter 6. Modeling the global tensile properties including damage for micro-tensile measurements has already been addressed for the friction stir welding of the aluminum alloy 6056 in a parallel study by Gallais [50, 51], in collaboration with the UCL team.

9.1 Experimental results

9.1.1 Results

(a) Uniaxial tensile test on welded sample

The deformation of a weld under transverse loading is heterogeneous and consequently true stress-strain curves cannot be extracted from the force-displacement results. Only pseudo engineering mechanical properties can be determined:

- R_p is a pseudo yield strength defined here at 0.1% offset to linearity since plasticity start first in a very confined zone of the weld;
- R_m is the pseudo ultimate tensile strength;
- e_u is the pseudo ultimate tensile strain (engineering strain at maximum load);
- ϵ_f is the maximum section reduction defined as for uniform materials ($\epsilon_f = \ln(S_i/S_r)$). Note that if the reduced section is not oriented at 90° from the loading direction, the surface of this reduced section is defined by projecting the section onto a plane perpendicular to the tensile direction. This is important in the damage modeling when predicting the fracture strain.

Figure 9.1 shows the evolution of the transverse tensile properties of friction stir welds as a function of the welding parameters. The yield strength and the ultimate tensile strength both increase with increasing advancing and rotational speeds. The ultimate tensile strain generally increases with decreasing rotational speed while the true fracture strain is almost not affected by the rotational speed. The effect of the advancing speed on those two properties is however not systematic. Interestingly, the ultimate tensile strain seems to be maximum for an intermediate welding speed equal to about 350 – 500 mm/min. This maximum seems to be associated with a slight decrease of the fracture strain. Note that the friction stir welding tool was different for very low advancing speed welds. Hence, this maximum in ultimate tensile strain could perhaps be related to a change in tool pin length. Further investigations is needed to confirm the presence of a maximum in the ultimate tensile strain.

The position of fracture is always on the retreating side of the weld for all the welds presented in Figure 9.1. Table 9.1 shows the exact position of fracture for the welds extensively studied in this work. The position of fracture gets closer to the weld centerline as the advancing

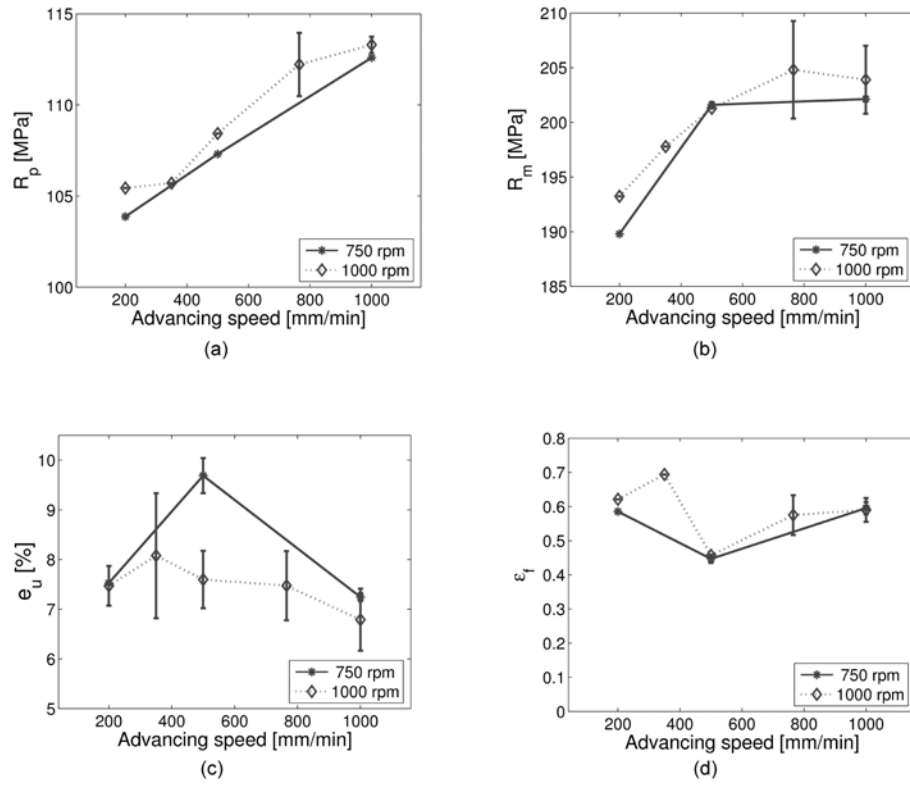


Figure 9.1. Tensile properties of friction stir welds with different welding parameters: (a) yield strength R_p [MPa]; (b) ultimate tensile strength R_m [MPa]; (c) ultimate tensile strain e_u ; (d) fracture strain ε_f .

Experiment number	1	2	3	4	5
Advancing speed [mm/min]	200	200	200	765	1000
Rotational speed [rpm]	500	750	1000	1000	1000
Fracture position (y) [mm]	9.8	10.9	11.8	7.8	5.7

Table 9.1. Fracture position (on the retreating side of the weld) for the welds extensively studied.

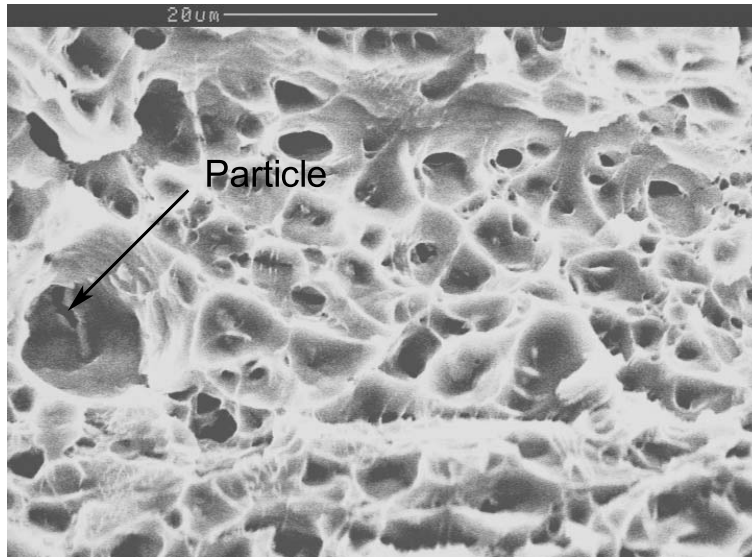


Figure 9.2. SEM micrograph of the fracture surface of a broken friction stir weld tensile sample with welding conditions equal to 1000 mm/min and 1000 rpm.

speed increases and as the rotational speed decreases, i.e. when the weld is colder.

Figure 9.2 shows a SEM micrograph of the fracture surface of a friction stir weld broken in the heat affected zone. Fracture has clearly occurred in a ductile manner by the growth and coalescence of voids. Small particles that are at the origin of the void growth are found inside the voids. The size of these particles matches with the size of the α particles.

(b) Digital image correlation

Figure 9.3 presents the results of digital image correlation (DIC) of a cold weld (1000 mm/min and 1000 rpm) and a hot weld (200 mm/min

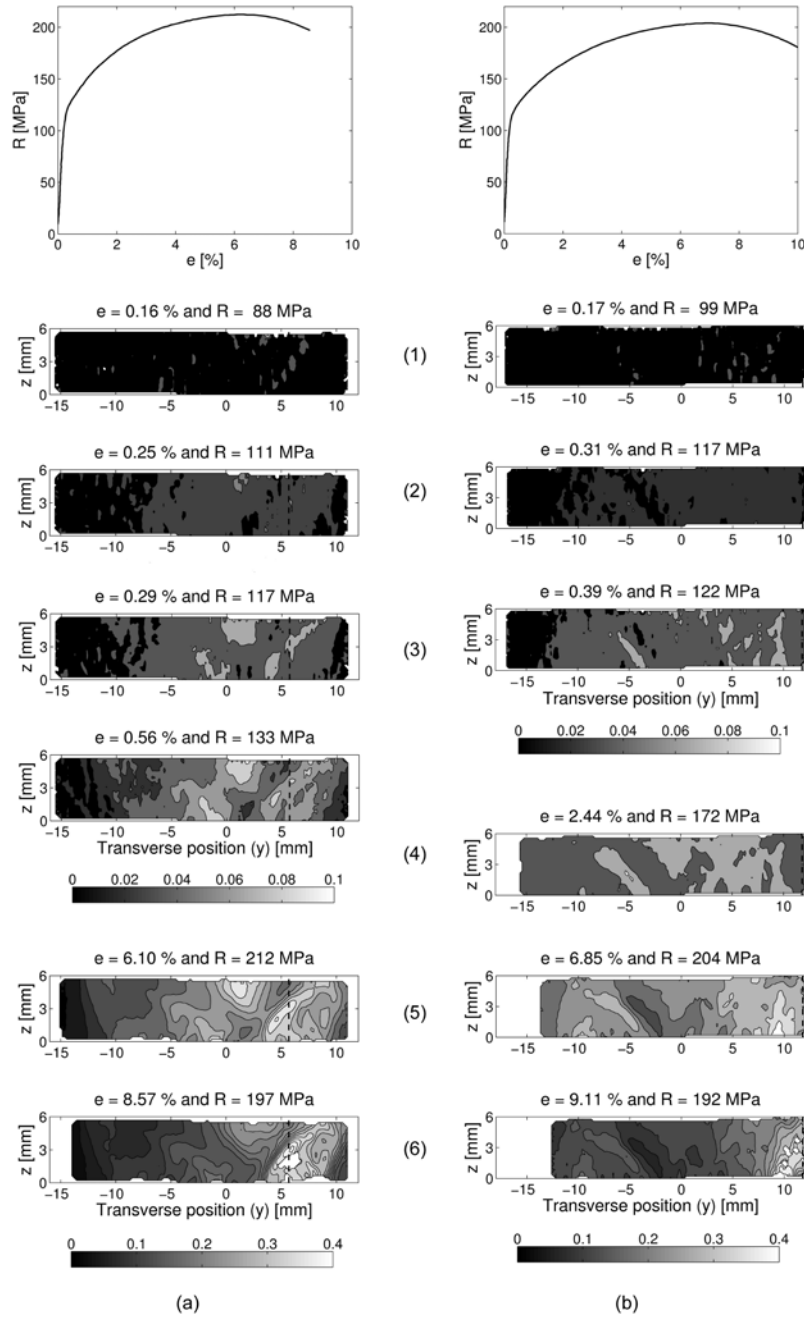


Figure 9.3. Logarithmic strain in the tensile direction ε_{yy} in friction stir welds subjected to transverse tensile testing (DIC results). Welds with welding parameters 1000 rpm and (a) 1000 mm/min; (b) 200 mm/min. The top curve gives the corresponding engineering tensile curve for the given weld. The vertical dotted lines indicates the position of fracture as given in Table 9.1 (page 194).

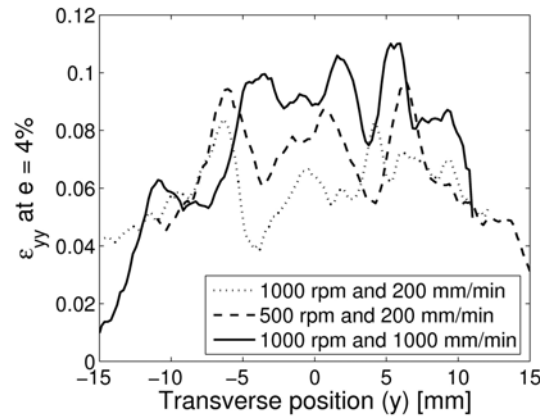


Figure 9.4. Effect of the the welding parameters on the mid-thickness logarithmic strain in the tensile direction ε_{yy} in friction stir welds submitted to transverse tensile testing (DIC results). Results for at a global elongation e of 4%.

and 1000 rpm) at different levels of deformation. The engineering stress-strain curve on top of Figure 9.3 allows linking the local strain in the tensile direction ε_{yy} with the global elongation e and the nominal stress R . The general trends are as follow:

- Figure 9.3(1). Plastic deformation starts on the retreating side of the weld in the heat affected zone.
- Figure 9.3(2). The highest deformations localize in the entire low local yield strength zone which basically corresponds to the thermally affected zone.
- Figure 9.3(3,4). For the cold weld, ε_{yy} localizes in the weld center and the heat affected zone on the retreating side of the weld. For the hot weld, ε_{yy} localizes on the two sides of the weld in the heat affected zone.
- Figure 9.3(5). Necking starts on the retreating side of both weld.
- Figure 9.3(6). Fracture occurs on the retreating side of both welds at the position indicated by the vertical dotted line.

Figure 9.4 compares the results of the digital image correlation at mid-thickness for three extreme welding conditions for an elongation e of 4% (plastic deformation). The following can be observed:

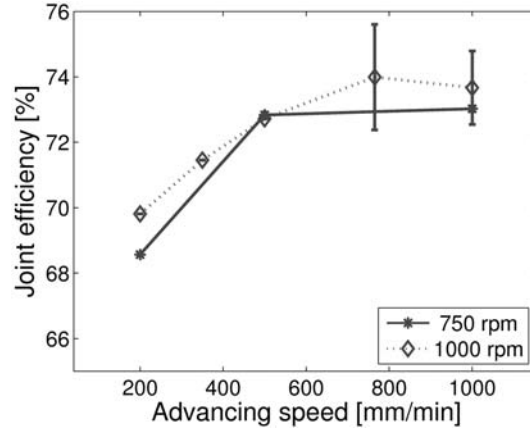


Figure 9.5. Joint efficiency (R_m/R_{mBM}) for different welding conditions.

- Deformation localizes in a relatively wide zone including the weld center in the coldest weld (1000 rpm and 1000 mm/min) while in the hottest weld (1000 rpm and 200 mm/min) deformation is, at this stage of elongation, already very localized in the heat affected zone. For the intermediate welding conditions (500 rpm and 200 mm/min), the deformation behavior is intermediate between the behavior of the hottest and the coldest weld.
- The maximum logarithmic strain in the tensile direction ε_{yy} is higher for colder welds.
- The position of the maximum logarithmic strain in the tensile direction ε_{yy} is closer to the weld center for colder welds.

9.1.2 Discussion

(a) Joint efficiency

It is classical in the welding community to define the so-called "joint efficiency" as the ratio of the ultimate tensile strength of the weld to the ultimate tensile strength of the base material (indicated in Table 3.4, page 33). Figure 9.5 shows the evolution of the joint efficiency as a function of the welding conditions. The joint efficiency ranges between 68 and 75% and increases with increasing advancing and rotational speeds. The values of the joint efficiencies of the welds confirm the good quality of these welds. Indeed, the joint efficiency found in the literature for

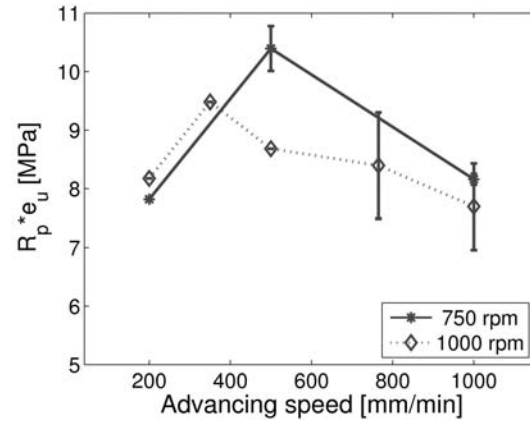


Figure 9.6. Yield strength R_p [MPa] multiplied by the ultimate tensile strain e_u of friction stir welds with different welding parameters. Image of the toughness of the welds.

6xxx aluminum alloys friction stir welds lie between 60% and 90% [75, 92, 110, 142].

(b) Weld toughness indicator

Figure 9.6 presents the yield strength multiplied by the strain at necking $R_p * e_u$ of friction stir welds with different welding conditions. This factor is a good indication of the toughness of the welds. A maximum of $R_p * e_u$ is observed in the range where the strain at necking was found to present a maximum. A lower rotational speed and a relatively low advancing speed seems to give a higher toughness factor. In conclusion, favoring a maximum joint efficiency deteriorates the toughness. Hence, optimal welding conditions for the welding range studied here would be equal to about 500 mm/min and 750 rpm. The joint efficiency for these conditions (73%) is only slightly lower than its maximum value and the toughness level is probably optimal for these welding conditions.

(c) Relevance of the weld pitch

Figure 9.1 has shown that the tensile properties of friction stir welds tend to increase for increasing advancing and rotational speeds. Hence, the weld pitch (ratio of the advancing speed to the rotational speed [mm/rot]) is clearly not a good parameter of the weld global behavior.

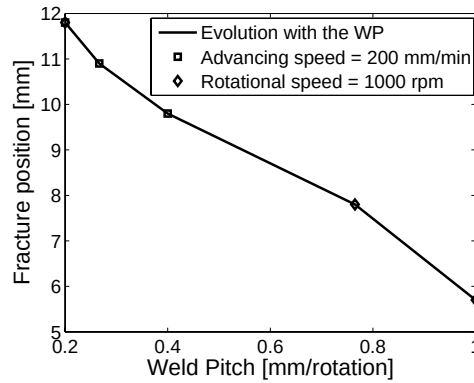


Figure 9.7. Fracture position on the retreating side of the weld as a function of the weld pitch (ratio of the advancing speed to the rotational speed).

Figure 9.4 has shown that the maximum logarithmic strain in the tensile direction ε_{yy} is higher for colder welds (i.e. increasing weld pitch). Hence, it seems that there exists a correlation between the weld pitch and the distribution of the deformations.

Figure 9.7 has shown that the fracture position (given in Table 9.1, page 194) moves closer to the weld center with increasing weld pitch. The distance between the position of the maximum logarithmic strain in the tensile direction ε_{yy} and the weld center (DIC results of Figure 9.4) also decreases with increasing weld pitch. The position of fracture is directly related to the width of the zone thermally affected. It was shown in Figure 6.20 that the width of this zone decreases with increasing advancing speed and decreasing rotational speed, i.e. with increasing weld pitch.

(d) Linking local and global tensile properties

Yield strength. In Figure 9.8, the yield strength R_p of the transverse tensile specimens is compared to the minimum yield strength from all the micro-tensile specimens machined from the same weld, for various welding conditions (see Figure 6.21). This minimum is found in the HAZ or close to the TMAZ, generally on the retreating side of the weld as was shown in Figure 6.22. The evolutions are very similar. Indeed, this minimum yield strength of the micro-tensile samples is directly related to the yield strength of the weld when submitted to transverse tensile testing since the weakest link (i.e. the material that will first yield)

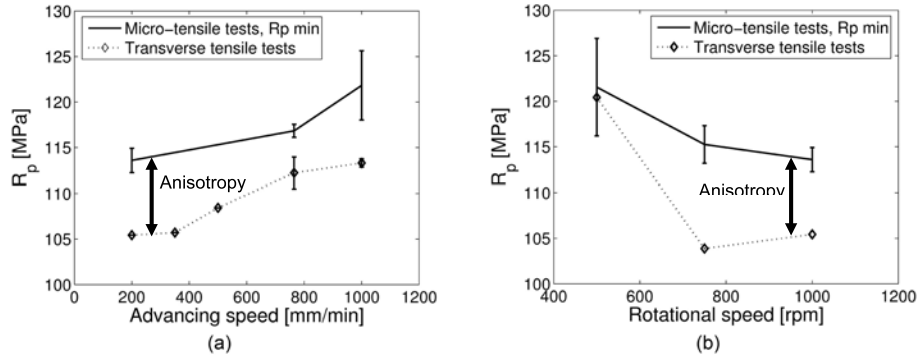


Figure 9.8. Comparison of the 0.1% offset yield strength of transverse tensile specimens and the minimum yield strength of the micro-tensile specimens for different (a) advancing speeds for a rotational speed of 1000 mm/min; (b) rotational speeds for an advancing speed of 200 mm/min.

will determine the yield strength of the entire sample. Nevertheless, Figure 9.8 shows that the minimum yield strength for the micro-tensile specimens is about 10 MPa higher than the welds yield strength when subjected to transverse tensile loading. This is not surprising since it has been shown in Table 3.4 (page 33) that the base material has anisotropic tensile properties and, in particular, the yield strength in the transverse long direction (direction of the transverse tensile tests on the weld) is 10 MPa lower than the yield strength in the extrusion direction.

Fracture location. Figure 9.9 shows that the fracture locations given in Table 9.1 (page 194) correspond to the position in the welds where the local ultimate tensile strength is minimum. The local strain measurements in the tensile direction during a transverse tensile test on friction stir welds (see Figure 9.3) have shown that even though the yield strength is relatively uniform in the entire thermally affected zone, the strain hardening capacity of the weld center being higher than in the heat affected zone, fracture will finally take place in the heat affected zone.

Deformation localization. The DIC results have shown that the localization of deformation in the heat affected zone of the cold weld (advancing speed of 1000 mm/min) only occurs very late in the deformation process while it occurs much earlier in the hot weld (advancing speed of 200 mm/min). This difference in behavior between hot and cold welds can be attributed to the low difference in tensile strength proper-

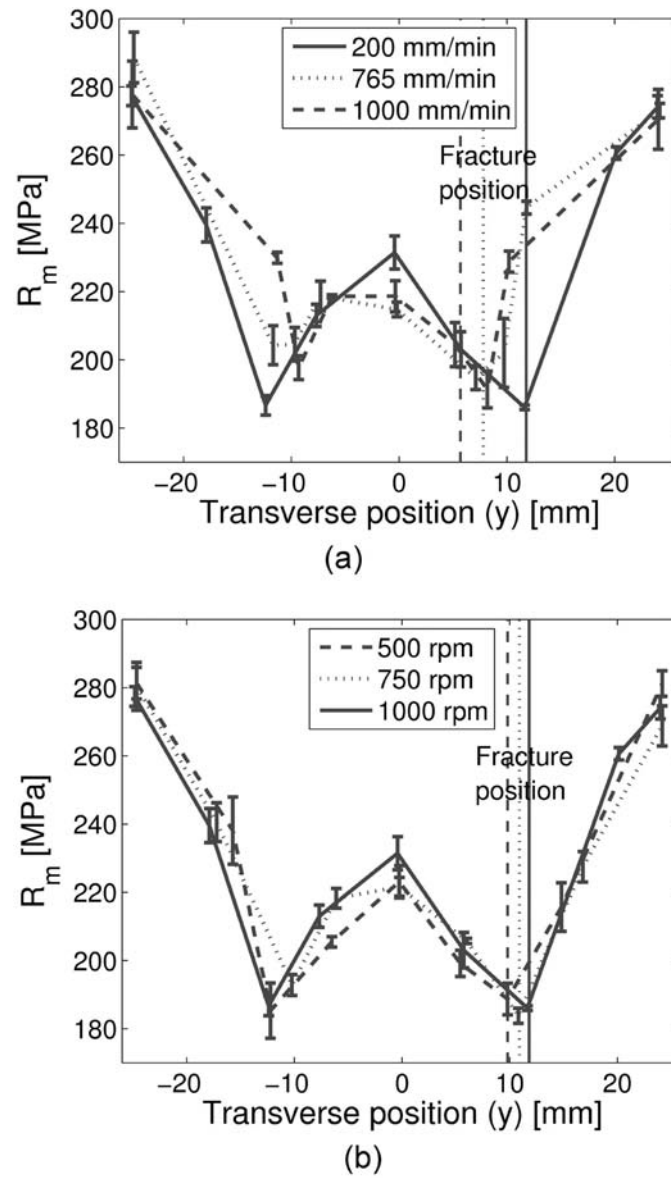


Figure 9.9. Comparison of the fracture position and the ultimate tensile strength R_m for friction stir welds having (a) different advancing speeds for a rotational speed equal to 1000 mm/min; (b) different rotational speeds for an advancing speed equal to 200 mm/min.

ties (and in particular in ultimate tensile strength) when comparing the heat affected zone and the weld nugget. Looking at Figure 9.9(a), we see that the hot weld has a difference in ultimate tensile strength between the weld nugget and the heat affected zone of more than 40 MPa while the cold weld has a difference of less than 20 MPa. The ultimate tensile strength of the weld nugget increases with decreasing advancing speed while it is the contrary in the heat affected zone. Hence, the strength mismatch reduces when the advancing speed decreases.

Asymmetry. Figure 9.3 has shown that yielding starts on the retreating side of the weld. This is consistent with the fact that the retreating side of both welds exhibit a lower yield strength (see Figure 6.22). When strain hardening starts, deformation does not directly localize on a unique side of the weld for the hot weld (advancing speed of 200 mm/min) until necking starts even though fracture will occur on the retreating side of all the welds tested in this study. For cold welds (advancing speed of 1000 mm/min), the retreating side of the weld exhibits higher plastic strain than the advancing side of the weld soon after the beginning of the deformation process. The distribution of temperatures has been studied in part II of this thesis. Figure 5.13 has shown that the temperature difference between the advancing side and the retreating side of the weld is higher in a cold weld. Furthermore, the width of the thermally affected zone is more asymmetric for cold welds (see Figure 6.20(c)). The asymmetry in local deformation is here again more marked in the cold weld (see Figure 9.3). Furthermore, it was shown that in the weld with an advancing speed of 200 mm/min and a rotational speed of 1000 rpm, the retreating side could be hotter (see Figure 5.13). This relates to the fact that, in the hot weld presented in Figure 9.3(b), deformation localizes also on the advancing side of the weld at the beginning of the deformation process (even though fracture ends up occurring on the retreating side of the weld).

9.1.3 Conclusions of the experimental results

This section has presented and discussed the experimental results on the global tensile behavior of friction stir welds generated with various welding conditions. The following conclusions can be made:

- The global tensile behavior of the friction stir welds agree with results usually described in the literature confirming the good quality of the welds produced at UCL.

- The toughness is not maximum for the welding conditions giving the best joint efficiency. Hence a compromise has to be found when choosing the welding conditions.
- The yield strength of a weld corresponds to the yield strength of its weakest zone, which is always located on the retreating side of the welds as confirmed by the DIC experiments.
- The position of fracture is directly related to the minimum of the local ultimate tensile strength and not only to the minimum of the yield strength. It is thus not always correct to infer the location of fracture from the hardness measurements.
- The transverse tensile behavior of hot and cold welds are very different, in particular with respect to the deformation localization process. Indeed, for cold welds deformation localizes in the heat affected zone and on the retreating side only very late in the deformation process contrarily to the hot welds where it is observed at the beginning of strain hardening.
- The asymmetry of deformation in cold welds develops early in the deformation process while it occurs much later in very hot welds.

9.2 Modeling the global tensile properties and damage

9.2.1 Modeling the global tensile properties from the local properties

(a) Model description

The global transverse tensile properties of a weld have been modeled using the general purpose finite element code ABAQUS [1] assuming J2 isotropic plasticity. A transverse tensile specimen with the geometry of the experimental transverse tensile samples (see Figure 3.6) is modeled by providing the local tensile properties measured with the micro-tensile specimens (see Chapter 6) in the various characteristic regions of the weld. Figure 9.10 shows how the tensile properties are distributed throughout the finite element model for the specific case of the transverse tensile test on weld number "3". The position of the transition between microstructural zones of the weld and the inclination of these transitions are based on the hardness measurements (see Figure 6.20) and the weld shapes macrographs (see Figure 4.6) of the five welds. Due to the

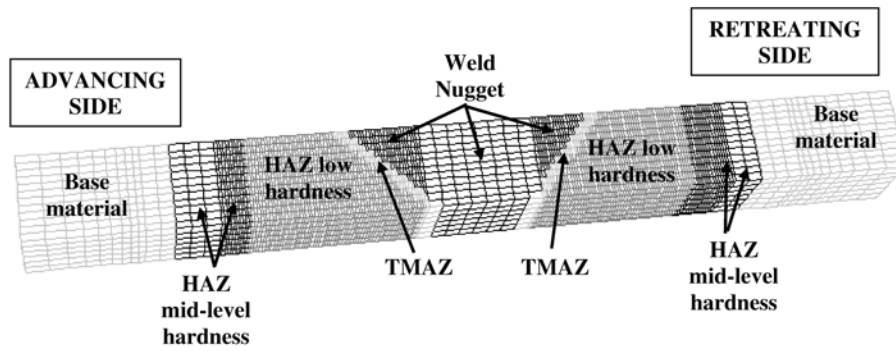


Figure 9.10. Mesh and zones with different material properties for the modeling of the tensile behavior of friction stir weld number "3" (200 mm/min and 1000 rpm).

differences between the advancing and retreating side of the joint, full 3D simulations are performed accounting only for one symmetry plane parallel to the loading direction and to the through thickness direction. The mesh is refined in the region where final fracture from which accurate stress and strain evolution must be extracted for the prediction of damage evolution (see next section).

For each region of the weld, a series of three micro-tensile tests are available. The simulation were repeated three times for each weld in order to evaluate the dispersion in the modeling results due to the experimental scatter. The stress-strain curves were introduced point by point up to the onset of necking and after have been extrapolated using a law of Voce (expression (8.11)).

(b) Assessment of the model

Tensile properties. Figure 9.11 compares the predicted tensile properties to the experimental results for the five welds. The quality of the predictions is relatively good considering that the distribution of the local mechanical properties are introduced approximatively in the finite element model. The discrepancies can be explained as follows:

- The overestimation of the yield strength is partially due to the anisotropy in the tensile properties. This effect was already discussed in Figure 9.8 comparing the yield strength of the micro-tensile samples and that of transverse tensile samples of friction stir welds. Indeed, Table 3.4 (page 33) confirms that the yield strength in the extrusion direction is higher than in the transverse

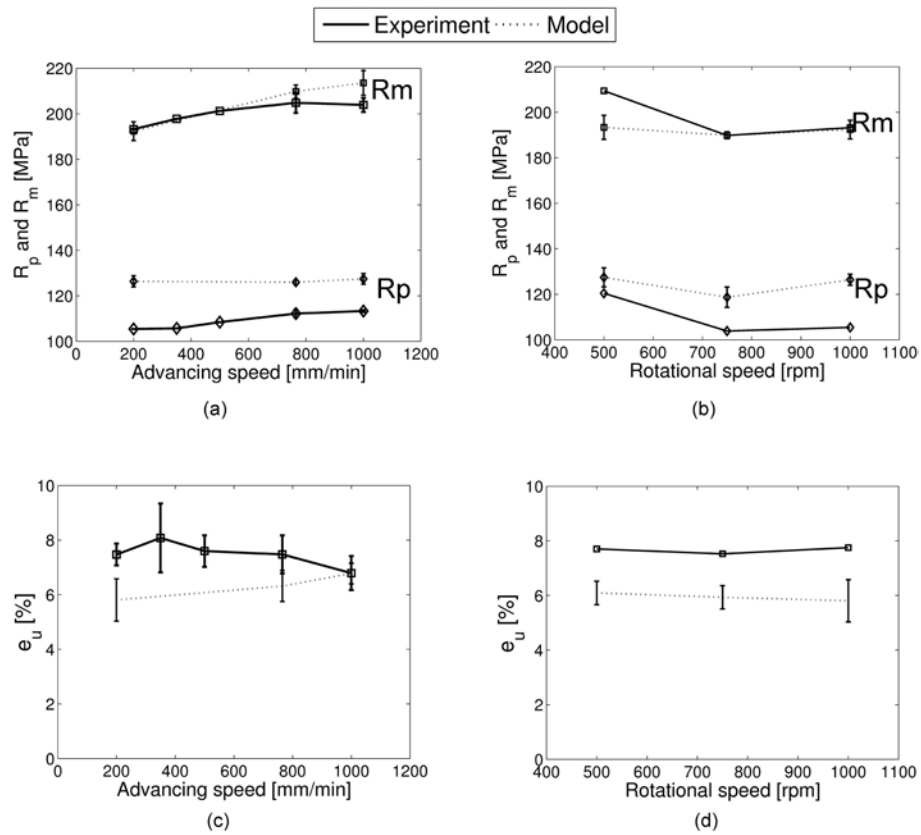


Figure 9.11. Comparison of the predicted and measured engineering tensile properties for (a,c) different advancing speeds for a rotational speed of 1000 rpm; (b,d) different rotational speeds for and advancing speed equal to 200 mm/min; (a,b) yield strength R_p and ultimate tensile strength R_m ; (c,d) ultimate tensile strain e_u .

long direction (direction of the macro-tensile samples of the entire weld) of about 10 MPa. The finite element model is based on isotropic plasticity and is thus not capable of reproducing such an effect.

- The error on the ultimate tensile strength predictions for some welds can also be attributed to anisotropy and to the fact that damage is not taken into account at this level of the modeling.
- The error on the ultimate tensile strain predictions are more significant. Elongations are certainly largely affected by the simple distribution of the tensile properties assumed here with constant properties in various zones and sharp transition between zones. It is anticipated that smoother gradient of properties will tend to delay plastic localization. Furthermore, the global elongation is a sum of the local elongations, adding up errors on the local tensile properties and on the shapes of the weld zones.

Local strains. Figure 9.12 and 9.13 compare the predicted and measured distribution of local strains in the tensile direction ε_{yy} at mid-thickness for different relative global elongations (e/e_u) for friction stir welds number "5" (1000 mm/min and 1000 rpm, the coldest weld, see Figure 9.12) and number "3" (200 mm/min and 1000 rpm, the hottest weld, see Figure 9.13). The predictions are very satisfactory for the coldest weld in which the deformation at the onset of necking was already well predicted (see in Figure 9.11(c) the point at an advancing speed of 1000 mm/min). The predictions are much less satisfactory for the hottest welding conditions. In that case, the onset of necking is already underestimated which lead to unrealistic strain fields after the onset of necking (see Figure 9.11(c,d)). Nevertheless, the model reproduces very well the general trends of the DIC results. In particular:

- Figure 9.12(a): The largest deformations localize in the entire low yield strength zone corresponding to the thermally affected zone.
- Figure 9.12(b,c) and 9.13(b,c): For the cold weld, the deformation localizes in the heat affected zone on the retreating side of the weld as well as in the weld center. For the hot weld, the deformation localizes in the heat affected zone of both sides of the weld with a slightly larger deformation on the retreating side of the weld.
- Figure 9.12(d,e,f) and 9.13(d,e,f): Necking that will cause final fracture starts on the retreating side for the coldest weld but on the advancing side for the hottest weld.

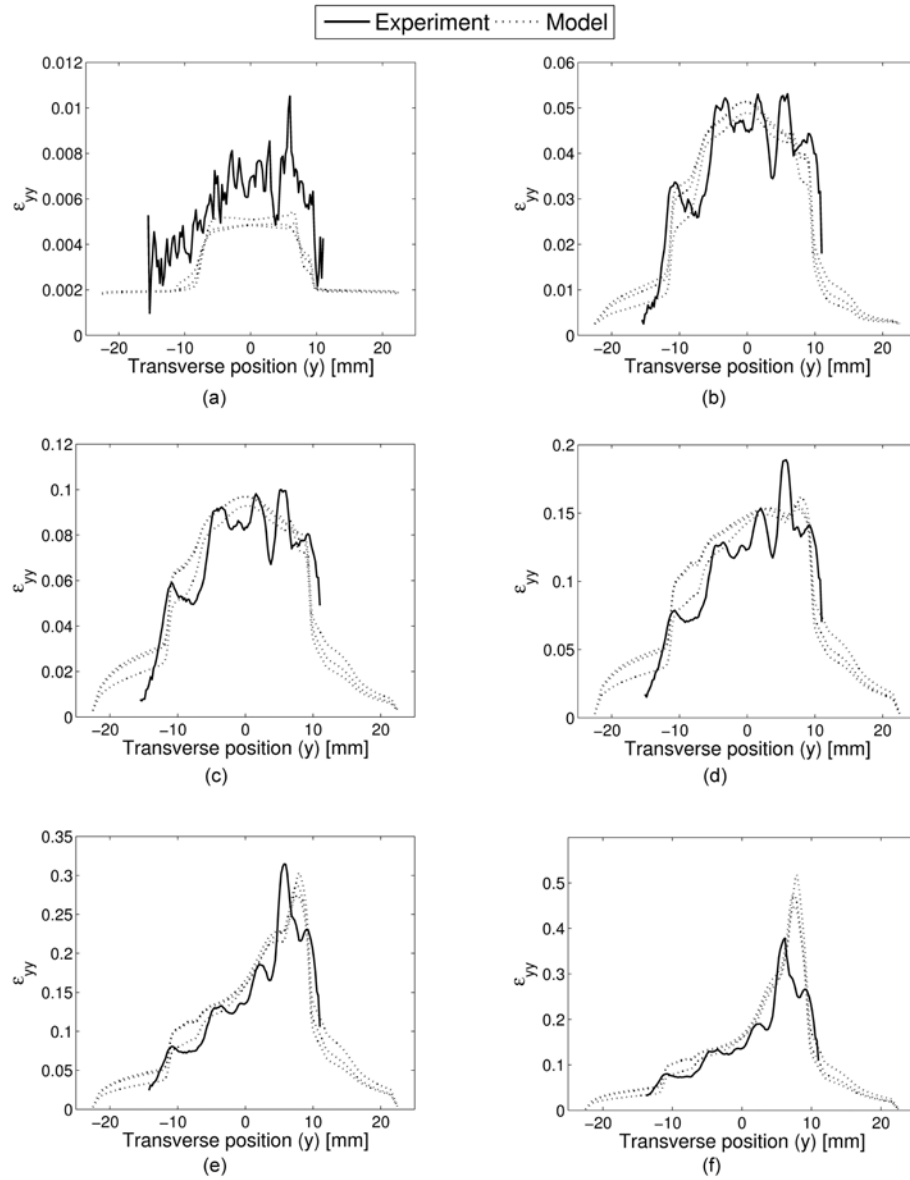


Figure 9.12. Comparison of the predicted and measured (DIC results) distribution of local strains in the tensile direction ε_{yy} at mid-thickness for different relative global elongations e/e_u for friction stir weld number "5" (1000 mm/min and 1000 rpm): (a) $e/e_u = 0.04$; (b) $e/e_u = 0.3$; (c) $e/e_u = 0.6$; (d) $e/e_u = 1$; (e) $e/e_u = 1.3$; (f) $e/e_u = 1.6$.

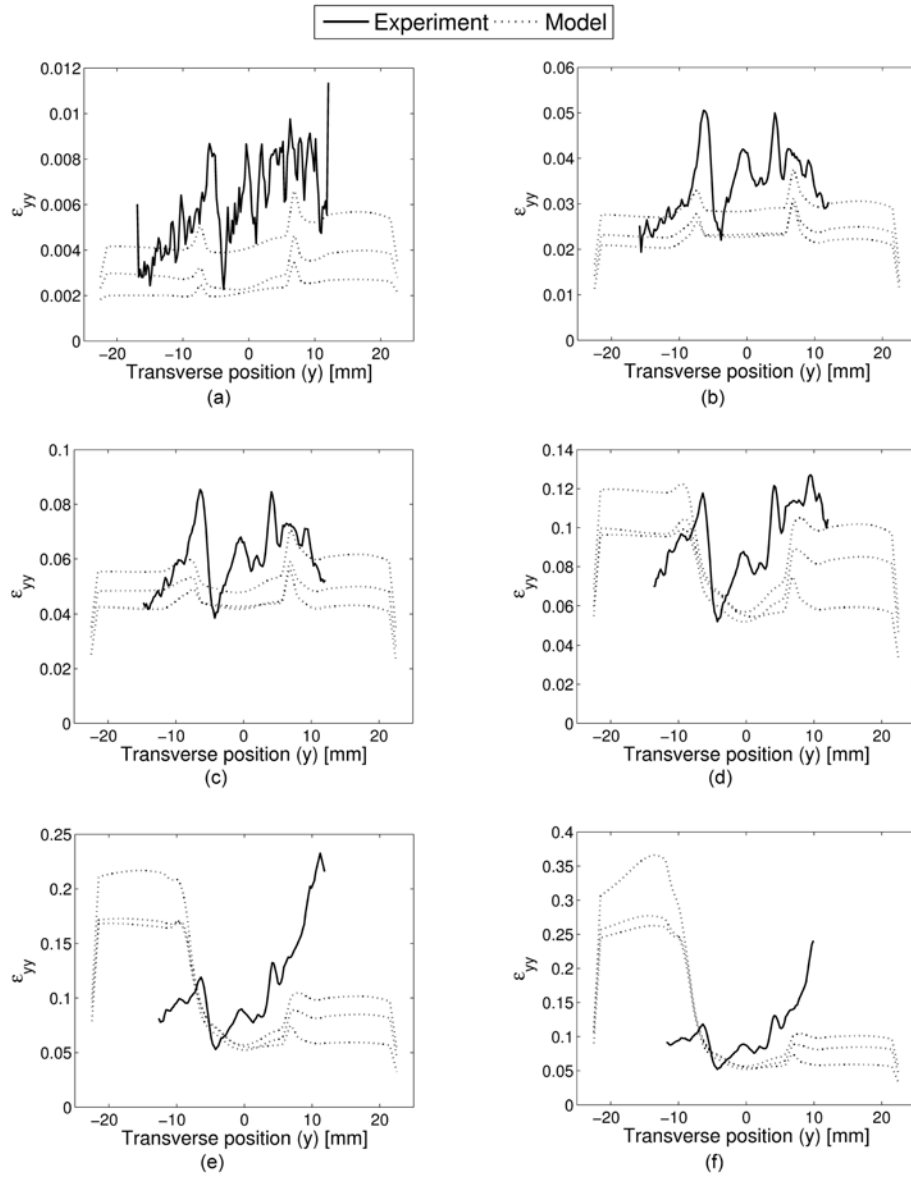


Figure 9.13. Comparison of the predicted and measured (DIC results) distribution of local strains in the tensile direction ε_{yy} at mid-thickness for different relative global elongations e/e_u for friction stir weld number "3" (200 mm/min and 1000 rpm): (a) $e/e_u = 0.04$; (b) $e/e_u = 0.3$; (c) $e/e_u = 0.6$; (d) $e/e_u = 1$; (e) $e/e_u = 1.3$; (f) $e/e_u = 1.6$.

The predicted side of the weld where final localization of deformation occurs does not match the experimental results which always show localization on the retreating side of the weld. Figure 9.14 shows that the position of fracture (the location where deformation localizes at large strains) is generally predicted on the retreating side of the welds for the cold welds while for the hot welds it is not always the case. It can even be on different sides for the same weld when considering different set of micro-tensile sample results. This confirms that, in hot welds, the localization of fracture may vary with only very slight differences in shape and tensile properties of the weld zones. The zone of fracture is however always predicted in the low hardness heat affected zone of the weld (except for one weld where it is predicted in the TMAZ).

9.2.2 Modeling damage

(a) Model description

Damage mechanisms. Damage in a ductile material is characterized by the nucleation, growth and coalescence of small internal voids. These voids generally nucleate by the fracture or decohesion of second phase particles. The voids then grow by plastic deformation. Void coalescence consists in the localization of plastic deformation at the micro-scale inside the intervoid ligament between neighboring voids. In aluminum alloys, the coarse intermetallics particles are usually the sites for void nucleation. Figure 9.15 summarizes the different scales at which the tensile sample of friction stir welds are studied. Damage is studied at the micro-scale of the intermetallic phases in the zone where fracture will occur, i.e. the weakest zone of the weld.

Basics of the damage model. The aim of this section is to present an advanced damage model which captures the evolution of void growth and coalescence based on the microstructural measurements presented in chapter 6. The local fracture strain is computed using the model developed by Pardoen *et al.* [105, 106] which is a generalization of the Gurson model [59] enhanced by Tvegaard *et al.* [138], Needleman *et al.* [102], Thomason [137] (voids coalescence condition), Gologanu *et al.* [54] (voids morphology), Benzerga *et al.* [6] and Lassance *et al.* [72] (void coalescence condition).

Void growth. The voids are assumed to be present from the beginning of loading, i.e. nucleation is assumed to take place in the elastic regime. This assumption is acceptable considering the drastic deforma-

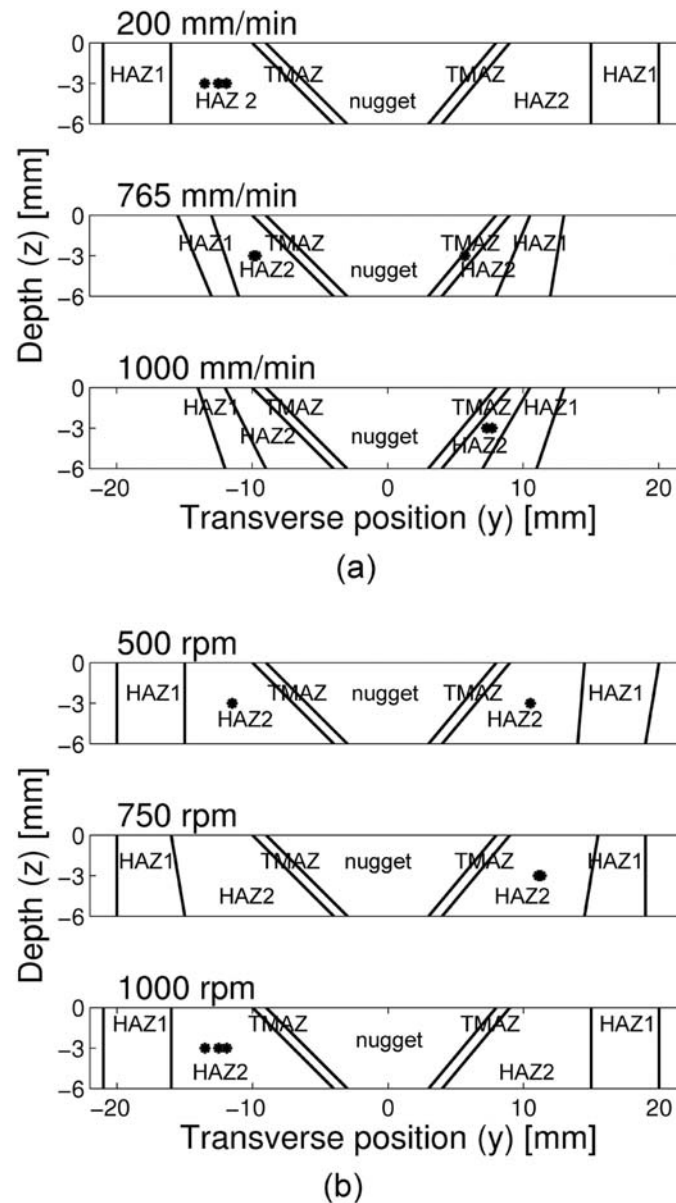


Figure 9.14. Predicted fracture position (*) considering that it corresponds to the location where deformation will localize at large strains for (a) different advancing speeds for a rotational speed of 1000 rpm; (b) different rotational speeds for and advancing speed of 200 mm/min. HAZ1 = heat affected zone mid-hardness level, HAZ2 = heat affected zone low hardness level, TMAZ = thermo-mechanically affected zone; $y > 0$ on the retreating side of the weld.

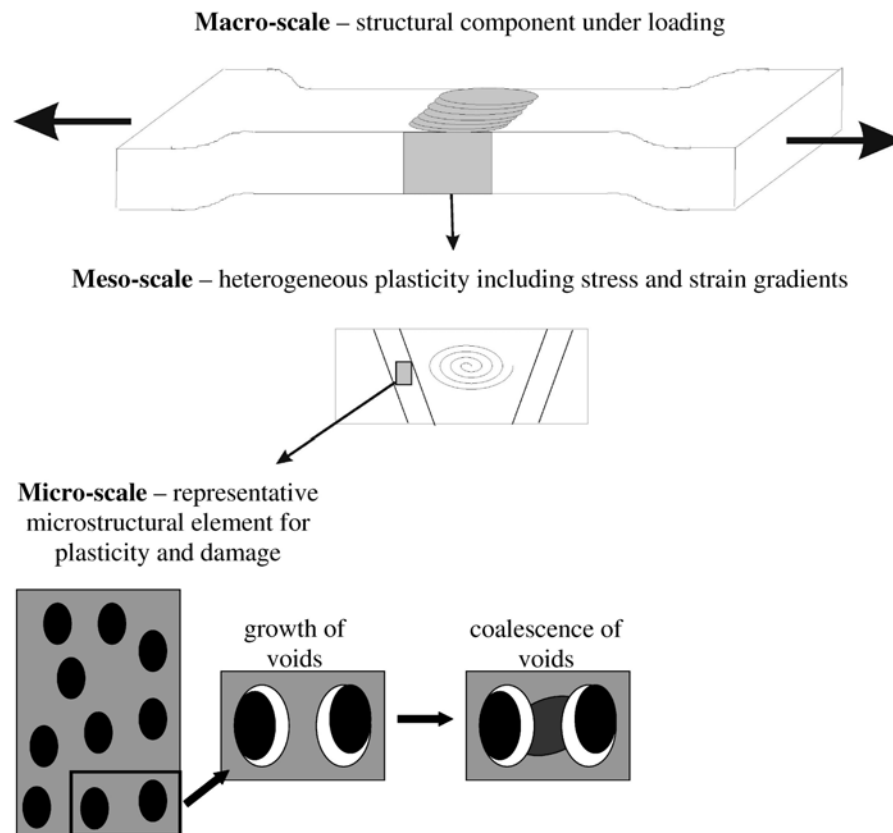


Figure 9.15. Schematic of the multi-scale mechanical analysis of the plasticity and damage of a friction stir weld structural component (inspired from [51]).

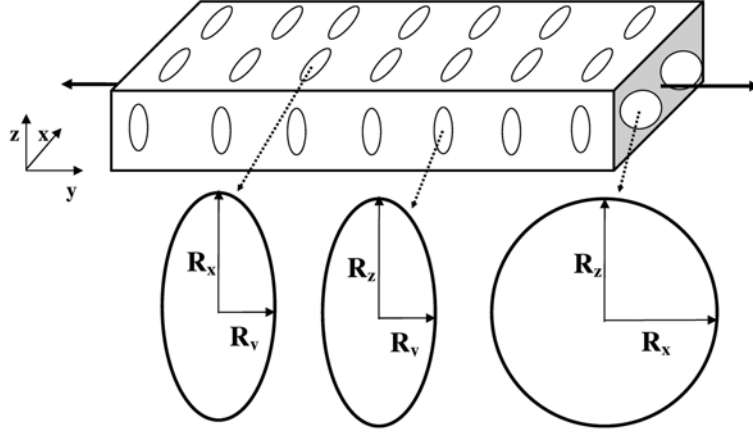


Figure 9.16. Definition of the void radius assuming spheroidal shape with respect to the main loading axis.

tion imposed to the plates during the forming process before welding which can already damage the particles; and considering that the yield strength values inside the weak zones are moderate. The voids are described by the following parameters:

- f is the porosity, i.e. the volume fraction of voids; f_0 is its initial value.
- W is the aspect ratio of the voids. The voids are assumed spheroidal, i.e. they present a circular section in a plane transverse to the axis of traction y (i.e. $R_x = R_z$, where R_x or R_z is the radius of the circular section). The logarithmic aspect ratio of the voids is defined as $S_v = \ln(W) = \ln(R_y/R_{x,z})$, where R_y is the "radius" of the spheroid along the y -axis. The initial values are designated S_{v0} and W_0 . Figure 9.16 clarifies the definition of W .

From the volume conservation of plastic deformation, the increase of the void volume fraction writes:

$$\dot{f} = (1 - f)\dot{\epsilon}_{ii}^p, \quad (9.1)$$

where $\dot{\epsilon}_{ij}^p$ are the ij components of the overall plastic strain tensor. The evolution law for S_v has been derived by Gologanu *et al.* [54] from the micro-mechanical analysis of the growth of spheroidal voids in a J2 perfectly plastic material:

$$\dot{S}_v = \frac{3}{2}(1 + h_1) \left(\dot{\epsilon}^p - \frac{\dot{\epsilon}_{ii}^p}{3} \delta_K \right) : P_d + h_2 \dot{\epsilon}_{ii}^p, \quad (9.2)$$

with analytical expressions for h_1 , h_2 as a function of S_v and f ; P_d is a projector tensor, defined by $e_y \otimes e_y$ (e_y is a unit vector parallel to the main cavity axis¹); δ_K is the Kronecker tensor.

The hardening behavior of the matrix material is related to the overall stress and plastic strain rate through the energy balance initially proposed by Gurson [59]:

$$\sigma_y \dot{\varepsilon}_y^p (1 - f) = \sigma \dot{\varepsilon}^p. \quad (9.3)$$

where the mean yield strength of the matrix σ_y is a function of the mean plastic strain ε_y^p .

In order to calculate the evolution of the three variables f , S_v and σ_y as well as the stress state σ , use is made of an associated flow rule:

$$\dot{\varepsilon}_{ij}^p = \dot{\gamma} \frac{\partial \Phi}{\partial \sigma_{ij}} \quad (9.4)$$

where Φ is the flow potential proposed by Gologanu *et al.* [54] for a porous material involving spheroidal voids:

$$\Phi = \frac{C}{\sigma_y^2} \|\sigma' + \eta_d \sigma_h^g X\|^2 + 2q(g+1)(g+f) \cosh\left(\kappa \frac{\sigma_h^g}{\sigma_y}\right) - (g+1)^2 - q^2(g+f)^2 = 0 \quad (9.5)$$

where (see Gologanu *et al.* [54], Pardoen *et al.* [105], Benzerga *et al.* [6] for complete expressions)

- σ' is the deviatoric tensor part of the Cauchy stress tensor;
- σ_h^g is a generalized hydrostatic stress defined by $\sigma_h^g = \sigma : J$; J is a tensor associated to the void axis and defined by:

$$J = (1 - 2\alpha_2)e_y \otimes e_y + \alpha_2 e_x \otimes e_x + \alpha_2 e_z \otimes e_z; \quad (9.6)$$

- X is a tensor associated to the void axis and defined by:

$$X = \frac{2}{3}e_y \otimes e_y - \frac{1}{3}e_x \otimes e_x - \frac{1}{3}e_z \otimes e_z; \quad (9.7)$$

- $\|\cdot\|$ is the von Mises norm;
- C , η_d , g , κ and α_2 are analytical functions of the state variables S_v and f ;
- q is a parameter that has been calibrated as a function of f_0 , W_0 and the strain hardening exponent n .

The stresses are calculated from

$$\dot{\sigma}_{ij} = L_{ijkl} (\dot{\varepsilon}_{kl} - \dot{\varepsilon}_{kl}^p) \quad (9.8)$$

where L_{ijkl} are the elastic moduli.

¹For the sake of simplicity, we will assume here that the main void axis e_y does not rotate and that it remains parallel to the loading direction, i.e. the y direction.

Void coalescence. The first stage of void growth by relatively homogeneous plastic deformation of the matrix surrounding the voids is interrupted by the localization of the plastic flow in the ligament between the voids. This localization corresponds to the onset of coalescence. From that point on, a second mode of void growth, called void coalescence, starts, driven by plasticity localized between the primary voids. The problem is quite similar to an internal necking process at a micro-scale. Thomason [137] proposed the following condition for the onset of coalescence by internal necking:

$$\frac{\sigma_{yy}}{\sigma_y(1-\chi^2)} = \frac{2}{3} \left[\alpha_d \left(\frac{1-\chi}{\chi W} \right)^2 + 1.24 \sqrt{\frac{1}{\chi}} \right] \quad (9.9)$$

where parameter α_d has been fitted as a function of the average value of the strain hardening exponent n [105] for $0 \leq n \leq 0.3$:

$$\alpha_d(n) = 0.1 + 0.22n + 4.8n^2 \quad (9.10)$$

Criterion (9.9) states that coalescence occurs when the stress normal to the localization plane reaches a critical value. This critical value decreases as the voids open (W increases) and get closer to each other (χ increases). Hence, the dominant parameter controlling the transition to the coalescence mode is the relative void spacing χ defined as:

$$\chi = \left(\frac{f \lambda_d}{\gamma_d W} \right)^{\frac{1}{3}} \quad (9.11)$$

where λ_d is the particle distribution factor and γ_d is a geometric factor which depends on the arrangement of voids. Here we will assume that the voids are uniformly distributed (i.e. $\lambda_d = 1$) in a periodic simple cubic array (i.e. $\gamma_d = 0.605$), see Figure 9.16. The porosity affects the coalescence indirectly through the link with the void spacing χ and through its softening effect on the applied stress σ_{yy} . In the present study, coalescence by internal necking is assumed to occur in a band normal to the main void axis which is certainly an approximate approach in the case of shear-driven void sheet coalescence mechanism.

Decoupling flow and damage. A version of the model has been implemented by Pardoen *et al.* in ABAQUS as a User defined MATERIAL subroutine. Here, in order to keep the simulation time acceptable, the calculations have been run in two steps. The simulations based on the J2 flow theory described in section 9.2 provide the evolution of the overall strain components and stress triaxiality in the element having the highest

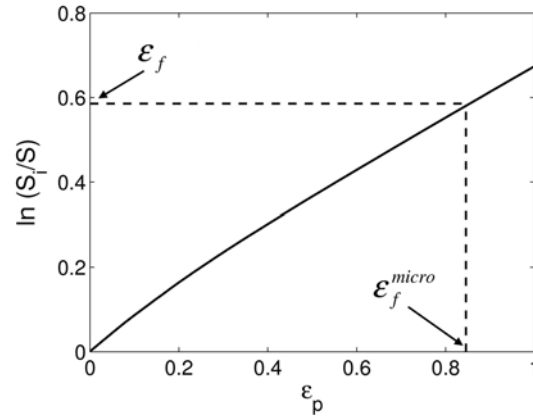


Figure 9.17. Evolution of the macroscopic strain defined as $\ln(S_i/S)$ (where S_i is the initial transverse section and S is the section at any time during the tensile test) as a function of the equivalent plastic strain ϵ_p at the position where it is maximum for weld number "2". Determination of the microscopic true strain at fracture ϵ_f^{micro} from the macroscopic true strain at fracture ϵ_f .

equivalent plastic strain. The history of the stress triaxiality as a function of the effective equivalent plastic strain is then applied to the damage model for the prediction of the onset of coalescence. In order to run the model independently, one must know at least the evolution of the stress triaxiality (defined as the ratio of the absolute value of the hydrostatic stress on the von Mises stress) as a function of the equivalent plastic strain ϵ_p taken at the position where it is maximum².

Linking the local maximum equivalent plastic strain to the macroscopic true fracture strain. The true fracture strain ϵ_f given by expression (3.2) ($\epsilon_f = \ln(S_i/S_r)$) is based on the macroscopic section reduction³. Modeling the transverse tensile behavior of a sample allows relating this macroscopic mechanical property to the equivalent plastic strain ϵ_p at the position where it is maximum. Figure 9.17 shows how the relationship between $\ln(S_i/S)$ (where S_i is the initial transverse section and S is the minimum section at any time during the tensile test) and ϵ_p at the position where it is maximum allows obtaining the local equivalent plastic strain ϵ_f^{micro} that corresponds to the experimental macroscopic true fracture strain ϵ_f defined by expression (3.2).

²The plastic strain tensor is note ϵ^p while ϵ_p is the equivalent plastic strain.

³The reduced section is defined as the projected section of the 3D reduced section on a plane perpendicular to the traction direction.

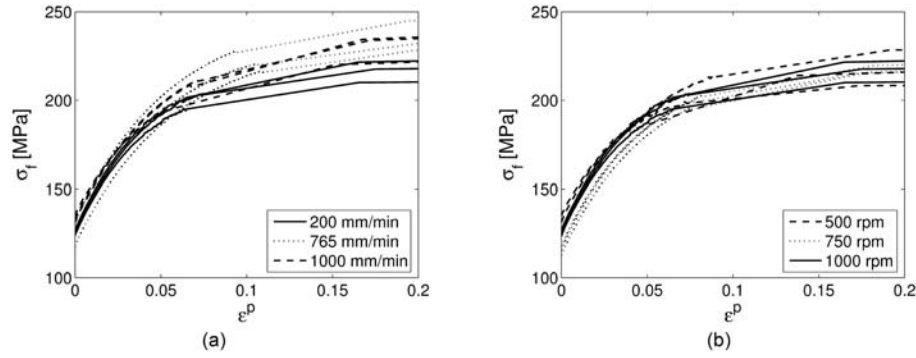


Figure 9.18. Flow stress as a function of the plastic strain curve of the weakest zone for different welding conditions for the three micro-tensile samples tested: (a) effect of the advancing speed for a rotational speed equal to 1000 rpm; (b) effect of the rotational speed for an advancing speed equal to 200 mm/min.

(b) Application of the model using the micro-tensile tests

Microstructural parameters of the damage model. The macro-graph of a broken sample in Figure 9.2 has shown that the α particles are at the origin of the voids. Hence, we will consider that the initial void volume fraction and aspect ratio corresponds to the α particles volume fraction and aspect ratio. The volume fraction and the aspect ratio of these precipitates is unaffected by the welding process (see Table 6.4, page 103). Hence, the volume fraction and the aspect ratio of the α phases measured for the base material (see Table 6.1, page 88) are used as reference for the model. In particular,

- the initial volume fraction of voids f_0 is equal to 1.2%;
- the initial aspect ratio of the voids W_0 is equal to $(1/1.7)$ since the tensile direction is perpendicular to the extrusion direction and the α phases are elongated in the extrusion direction (see Figure 9.16).

Stress-strain curves of the weakest zones. Figure 9.18 shows the stress-strain curves of the weakest zone of the friction stir welds introduced in the damage model. The strength in low advancing speed welds is slightly lower than the stresses for the high advancing speed welds. The effect of the rotational speed is not systematic. In the code, the flow stress-strain curve is not introduced point by point (like in the J2 plasticity calculations presented in section 9.2) but by fitting the curve

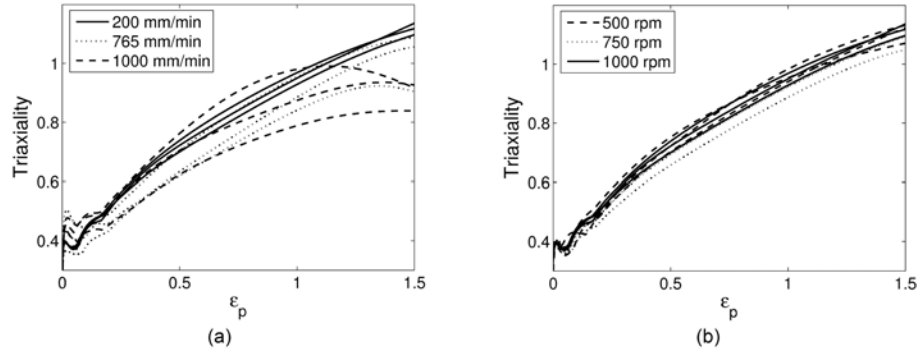


Figure 9.19. Evolution of the stress triaxiality in the most deformed element as a function of the equivalent plastic strain ε_p of that finite element for different welding conditions for the three micro-tensile samples tested in each zone of the welds: (a) effect of the advancing speed for a rotational speed equal to 1000 rpm; (b) effect of the rotational speed for an advancing speed equal to 200 mm/min.

with the following expression:

$$\frac{\sigma_f}{\sigma_y} = \frac{E\varepsilon_y}{\sigma_y} \quad \text{when } \sigma_f < \sigma_y \quad (9.12)$$

$$\frac{\sigma_f}{\sigma_y} = \left(1 + \frac{E\varepsilon_y^p}{\sigma_y}\right)^n \quad \text{when } \sigma_f > \sigma_y \quad (9.13)$$

where E is the Young modulus, σ_y is the initial yield strength, ε_y^p is the plastic strain and n is the strain-hardening exponent, assumed independent of strain.

Stress triaxiality evolutions. Figure 9.19 shows the evolution of the stress triaxiality taken at the finite element where the equivalent plastic strain is maximum for different welding conditions. The stress triaxiality evolution with equivalent plastic strain is not much influenced by the welding conditions except that in the coldest weld (1000 mm/min and 1000 rpm) the stress triaxiality tends to saturate and even decreases at a certain level of equivalent plastic strain. Nevertheless, the void growth rate is an exponent function of the stress triaxiality and minute differences in the stress triaxiality can have a major impact on the predictions.

Predictions of the true fracture strain. Table 9.2 compares the measured and predicted true fracture strains (microscopic and macroscopic). The agreement is quite good. Figure 9.20 shows the effect of the welding parameters on the predictions. The slight variation of the

Exp. number	1	2	3	4	5
Advancing speed [mm/min]	200	200	200	765	1000
Rotational speed [rpm]	500	750	1000	1000	1000
Exp. ε_f	0.43	0.59	0.62	0.60	0.55
Exp. + model ε_f^{micro}	0.60 (± 0.02)	0.84 (± 0.01)	0.92 (± 0.01)	0.82 (± 0.04)	0.83 (± 0.03)
Model ε_f^{micro}	0.7	0.85	0.78	0.82	0.87
Model ε_f	0.50	0.58	0.53	0.57	0.59

Table 9.2. Comparison of the predicted and measured true fracture strain linking the macroscopic true fracture strain ε_f (defined by $\varepsilon_f = \ln(S_i/S_r)$ where S_i is the initial transverse section and S_r is the reduced transverse section at fracture) and the microscopic true fracture strain ε_f^{micro} (result of the simulation of a weld).

true fracture strain with increasing advancing speed is not well captured by the model. Unfortunately, the welding conditions that show important variations of the true fracture strain (around 350-500 mm/min) cannot be modeled due to the absence of micro-tensile results for these welds. A maximum of the true fracture strain at an intermediate rotational speed is predicted while experimentally the true fracture strain increases with increasing rotational speed. Nevertheless, the identified effect of the welding parameters are in the error-bar of the measurements of the true fracture strain⁴.

Discussion. The stress triaxiality, which is the key parameter controlling damage kinetics, is a function of the strength gradient between the weak zone and the surrounding zones. The strength gradient depends on two factors; the strength mismatch and the width of the weak zone. In cold welds, even though the strength mismatch is smaller than in hot welds the weak zone is also narrower which lead to an increase of the stress triaxiality values. Hence, the two effects being opposite, the stress triaxiality levels are only weakly influenced by the welding conditions.

Figure 9.19 shows that the experimental dispersion in the tensile properties of the different zones of the weld causes a dispersion in the stress triaxiality evolution. Furthermore, Figure 9.21 shows that, even if changing the width of the heat affected zone by up to 20% has only a minor

⁴An error-bar is available for only a limited number of experimental results.

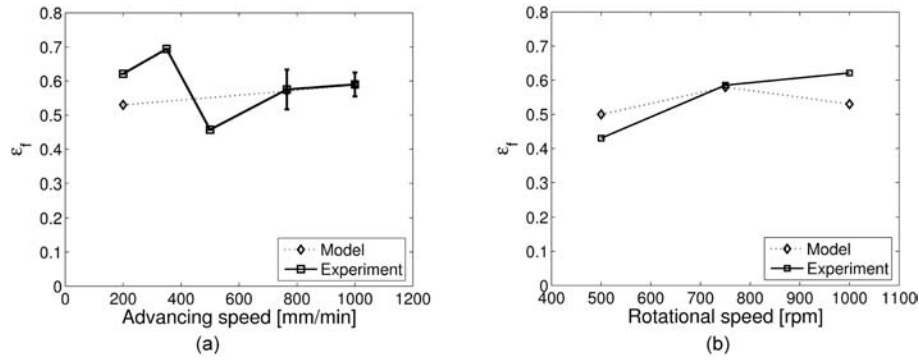


Figure 9.20. Comparison of the predicted and measured macroscopic true fracture strain ε_f (defined as $\varepsilon_f = \ln(S_i/S_r)$ where S_i is the initial transverse section and S_r is the reduced transverse section at fracture): (a) effect of the advancing speed for a rotational speed equal to 1000 rpm; (b) effect of the rotational speed for an advancing speed equal to 200 mm/min.

effect on the strength predictions, the stress triaxiality is moderately reduced. Hence, if the heat affected zone is larger (or narrower) than what is introduced in the model, the true fracture strain will be underestimated (or overestimated). Nevertheless, the inaccuracies in the local tensile properties and in the shape of the weld zones do not seem to significantly affect the predictions of the true fracture strain but probably affect the predictions of the small effects of the welding conditions on the true fracture strain.

The microscopic true fracture strain of welds subjected to transverse tensile loading can be compared to the local true fracture strain of the micro-samples machined within the heat affected zone on the retreating side of the weld (position where fracture will occur). This local true fracture strain ranges between 1.2 to 1.8. The microscopic true fracture strain of the welds are lower (around 0.7 to 0.9) since the plastic confinement drastically increases the triaxiality leading to a decrease of the microscopic true fracture strain.

9.2.3 Conclusions of the models

This section has described the modeling results of the global transverse tensile behavior including damage evolution within friction stir welds, for different welding conditions. Model results correlate very well with global tensile properties measured during the transverse tensile tests, confirming the ability to predict the global tensile properties of

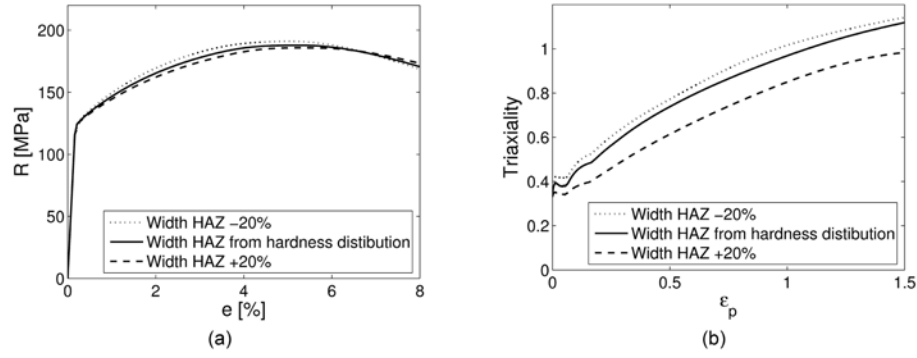


Figure 9.21. Effect of a change in the width of the heat affected zone of 20% for weld number "3" (200 mm/min and 1000 rpm): (a) tensile curve; (b) evolution of the stress triaxiality in the most deformed element as a function of the equivalent plastic deformation ϵ_p .

friction stir welds based on the knowledge of the local tensile properties.

The micro-mechanics based damage model is completely predictive and captures quite accurately the values of the true fracture strain without parameter adjustment. Unfortunately, the macro-samples for which micro-tensile samples are available do not show significant effects of the welding conditions on the the true fracture strain. This has been attributed to the compensation of two antagonistic effect: the strength mismatch is usually accompanied by an increase of the weakest zone width.

Chapter 10

Complete process modeling

The previous chapters have presented various models shown in Figure 1.2 devoted to the specific stages of the friction stir welds process:

- Chapter 5 presented a finite element based thermal model relating the welding parameters to the thermal cycles;
- Chapter 7 presented a physical model relating the thermal cycles to the microstructure evolution;
- Chapter 7 also presented a model relating the microstructure to the yield strength in different parts of the weld;
- Chapter 8 presented a physical model relating the microstructure and yield strength to the strain hardening capacity in different parts of the weld;
- Chapter 9 presented a finite element based model relating the local tensile properties to the global transverse tensile properties including a micromechanical damage model.

These models have been validated on experimental results on both base material heat treatments and friction stir welds. The decoupling of the different models has been favored by taking experimental measurements as input to the models when possible. It is now possible to assemble the models in order to look at the predictive potential of the entire chain of models. At this stage, the total power input is the only parameter of the assembled models that has to be determined experimentally for a given set of welding conditions when modeling friction stir welds in aluminum alloy 6005A. It must be noted however that, the total power input can be more simply estimated using the fit presented in equation 4.1 which assumes that the total power input depends only on the advancing speed in a linear manner.

10.1 Results of the model

10.1.1 Coupling the thermal cycles, microstructure evolution and local mechanical properties models

Comparison with experimental results. Figure 10.1 summarizes, for the two friction stir welds that have been extensively studied throughout this thesis (named the "coldest" and the "hottest" weld), the results of the microstructure evolution, yield strength and strain hardening models using as input the thermal cycles predicted by the thermal model presented in chapter 5. The temperature distribution of those two welds have been presented in Figure 5.11. The quality of the predictions of the thermal model being quite good, the application to the predictions of the microstructure evolution, of the local yield strength and of the local strain hardening (see chapters 7 and 8) does not introduce extra significant errors.

Effect of the advancing speed. Figure 10.2 and 10.3 describe the predictions of the model for various advancing speeds and a rotational speed equal to 1000 rpm. The following can be observed and discussed based on the results of the previous chapters:

- The thermally affected zone widens when the advancing speed decreases due to the higher maximum temperature at a given distance from the weld centerline (see Figure 10.2(a)).
- The minimum of the solid solution concentration does not correspond to the minimum of the yield strength when the advancing speed increases (see Figure 10.2(b,d)).
- The maximum precipitate radius increases with decreasing advancing speed (see Figure 10.2(c)) due to the decrease of the cooling rate with decreasing advancing speeds.
- The yield strength presents a clear minimum in the heat affected zone for the low advancing speed welds (see Figure 10.2(d)) due to insufficient solid solution concentration in the heat affected zone required for natural aging.
- The maximum of the dynamic recovery rate β increases with decreasing advancing speed (see Figure 10.2(e)) due to the increase of the "dislocation lift" (see section 8.2.4(b)) when the precipitate radius increases.

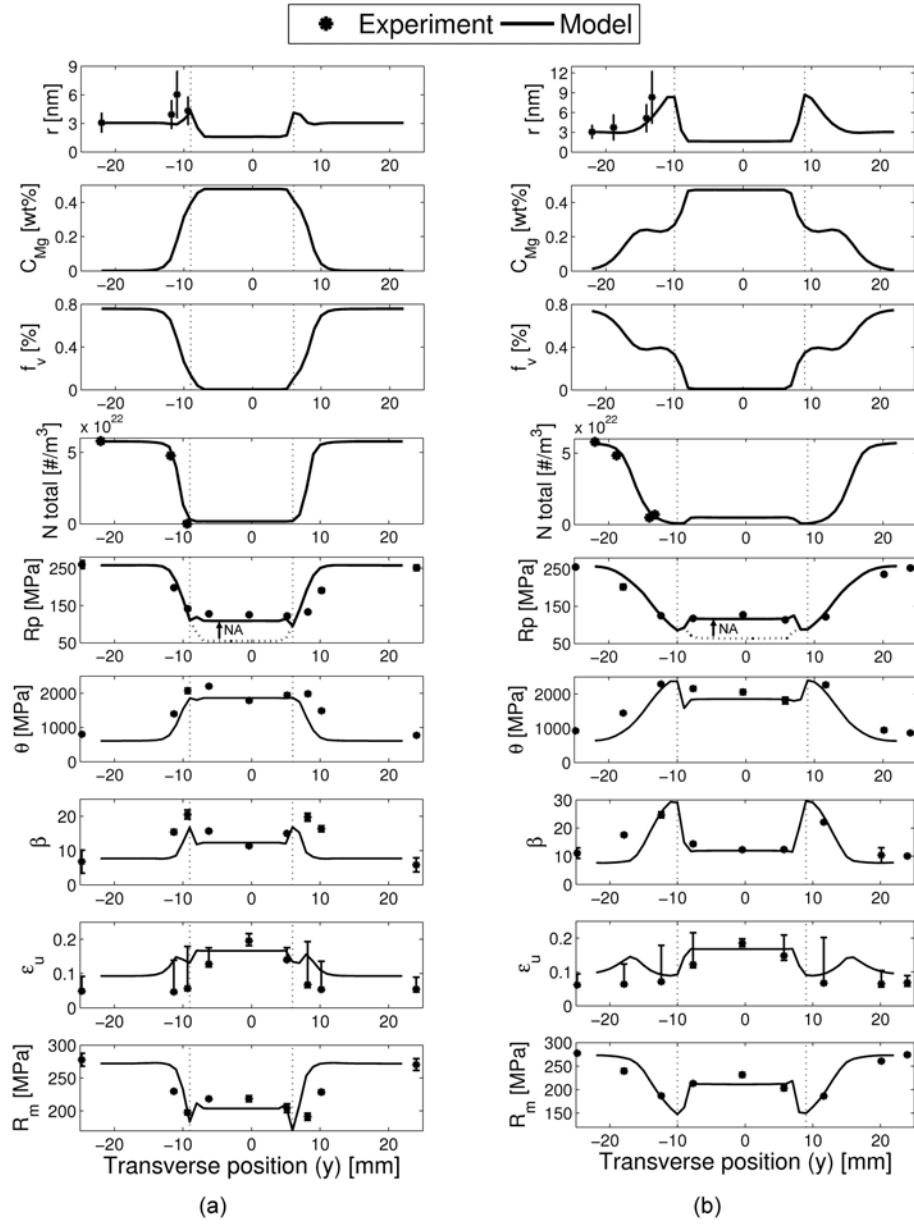


Figure 10.1. Comparison of the measured and predicted (based on the thermal model) microstructure and tensile properties as a function of the position along a transverse section at mid-thickness of a weld with a rotational speed 1000 rpm and (a) an advancing speed equal to 1000 mm/min; (b) an advancing speed equal to 200 mm/min; r = equivalent radius; C_{Mg} = solid solution concentration; f_v = volume fraction; N_{total} = precipitates density; R_p = yield strength; θ = dislocation storage parameter; β = dynamic recovery rate; ϵ_u = true strain at necking; R_m = ultimate tensile strength. NA = natural aging.

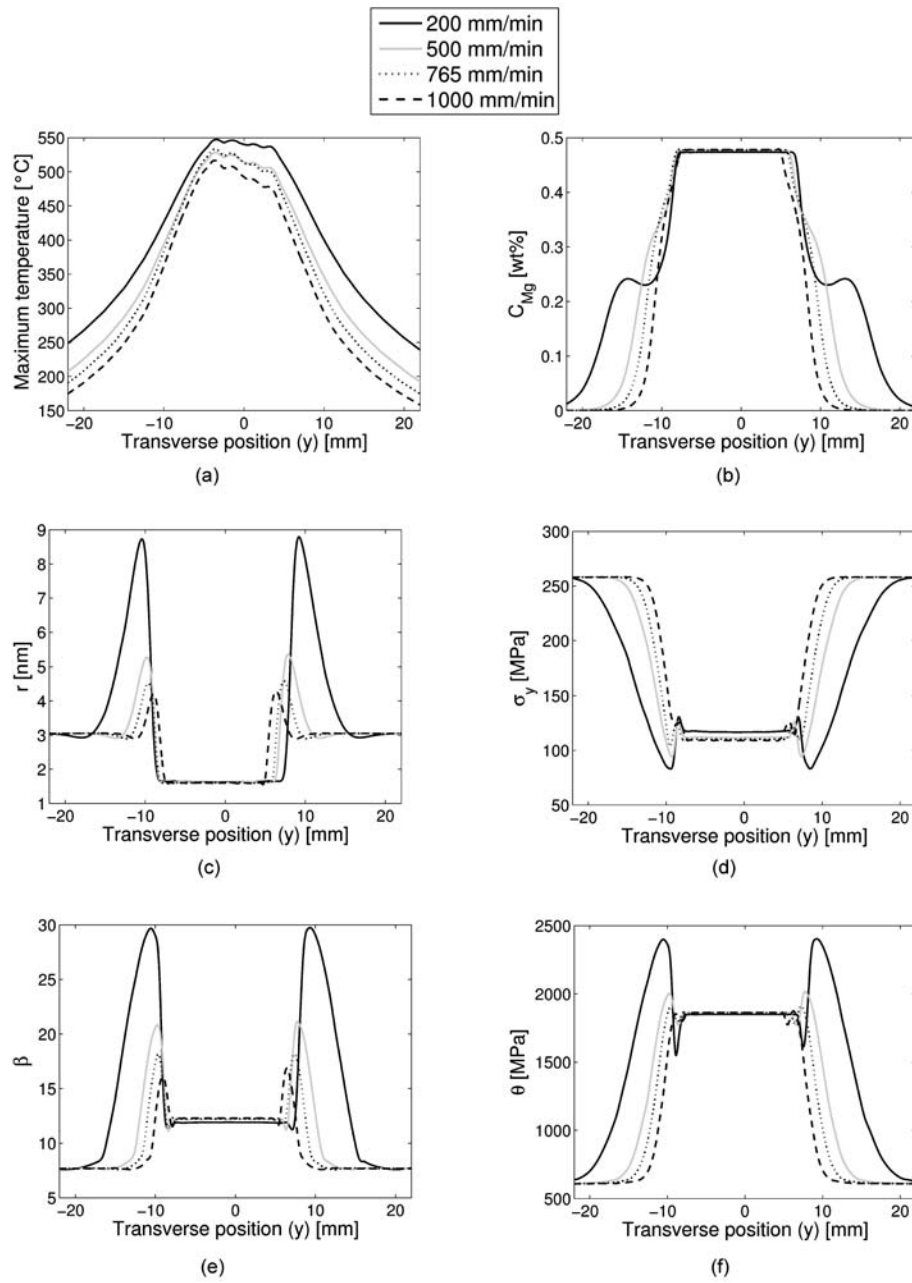


Figure 10.2. Effect of the advancing speed for a rotational speed equal to 1000 rpm on the predicted microstructure and tensile properties as a function of the position along a transverse weld section at mid-thickness: (a) maximum temperature; (b) solid solution concentration C_{Mg} ; (c) equivalent radius r ; (d) yield strength σ_y ; (e) dynamic recovery rate β ; (f) dislocation storage parameter θ .

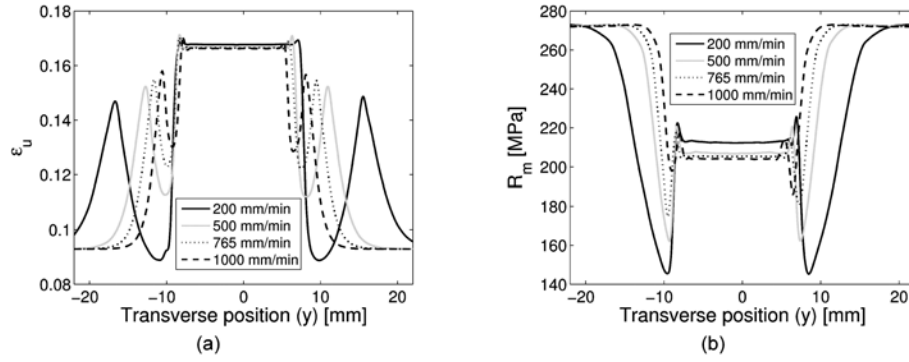


Figure 10.3. Effect of the advancing speed for a rotational speed equal to 1000 rpm on the predicted strength and strain at necking as a function of the position along a transverse weld section at mid-thickness: (a) true strain at necking ϵ_u ; (b) ultimate tensile strength R_m .

- The dislocation storage parameter θ presents a maximum in the heat affected zone if the advancing speed decreases (see Figure 10.2(f)) due to more efficient dislocation storage when the precipitate radius increases.
- The dislocation storage parameter θ is relatively high in the weld center due to the maximum driving force for dynamic precipitation in that region of the weld involving the largest solid solution concentration.
- The minimum of the true strain at necking and of the ultimate tensile strength decreases with decreasing advancing speed. The increase of the maximum dynamic recovery rate β with decreasing advancing speed is thus more effective for decreasing the strength and strain at necking of the heat affected zone than the dislocation storage parameter θ is effective for increasing its strength and strain at necking.
- The tensile properties do not vary with the advancing speed in the weld center (see Figure 10.2(d,e,f) and 10.3(a,b)) since, in that region of the weld, the dissolution of the precipitates is complete.

Effect of the rotational speed. Figure 10.4 and 10.5 show the effect of the rotational speed on the important field variables and parameters for an advancing speed equal to 200 mm/min. For this low advancing speed, the rotational speed mainly affects the width of the thermally

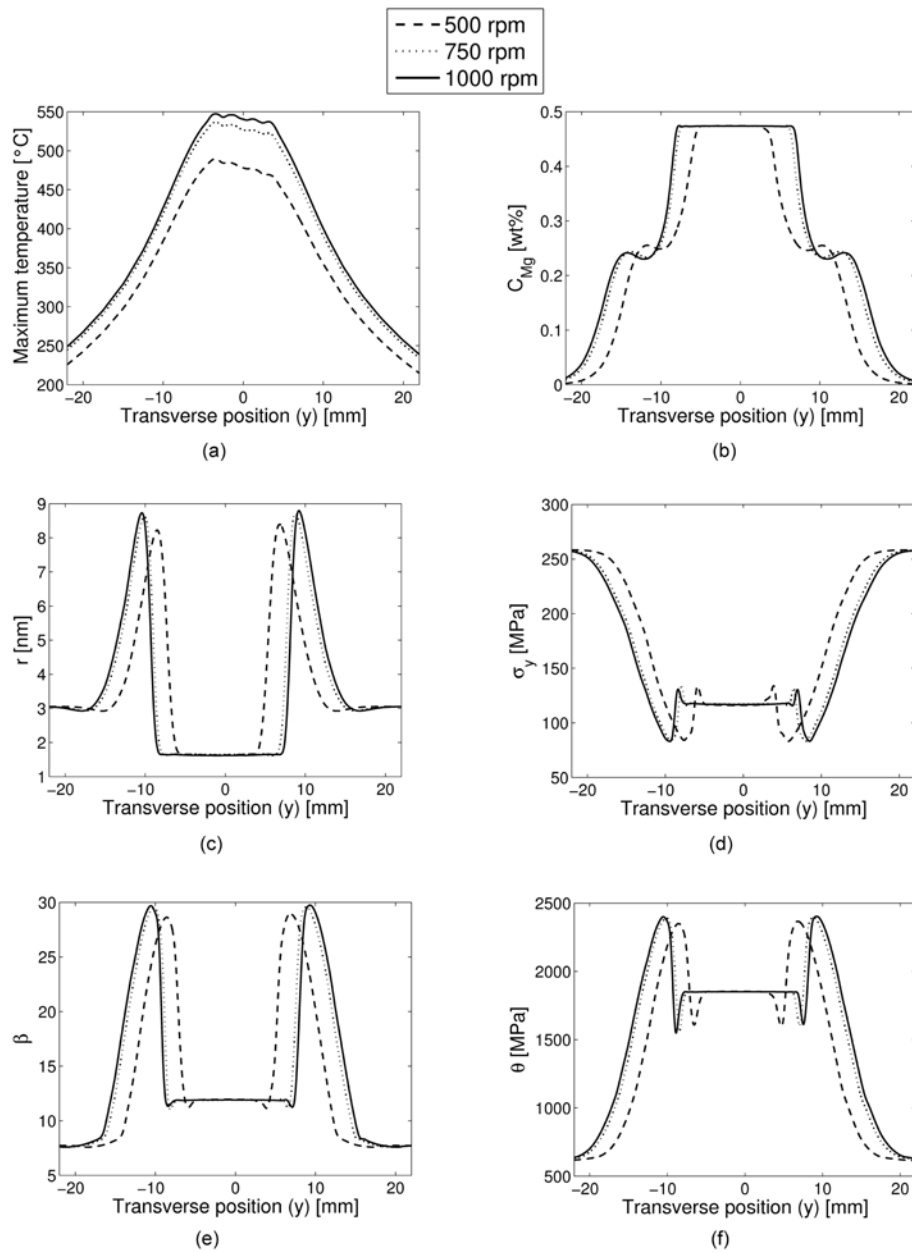


Figure 10.4. Effect of the rotational speed for an advancing speed equal to 200 mm/min on the predicted microstructure and tensile properties as a function of the position along a transverse weld section at mid-thickness: (a) maximum temperature; (b) solid solution concentration C_{Mg} ; (c) equivalent radius r ; (d) yield strength σ_y ; (e) dynamic recovery rate β ; (f) dislocation storage parameter θ .

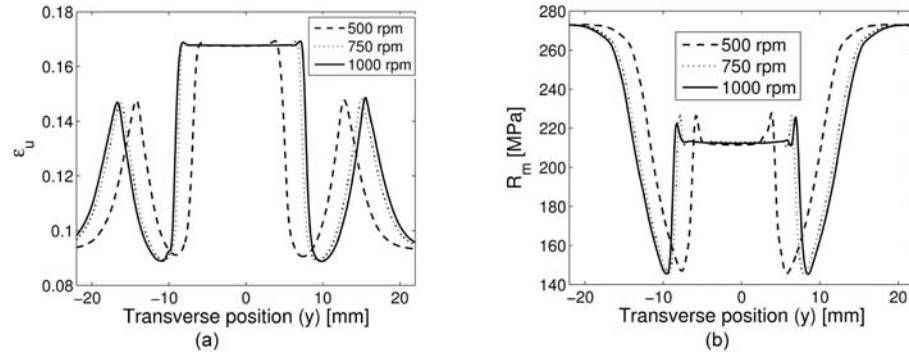


Figure 10.5. Effect of the rotational speed for an advancing speed equal to 200 mm/min on the predicted strength and strain at necking as a function of the position along a transverse weld section at mid-thickness: (a) true strain at necking ϵ_u ; (b) ultimate tensile strength R_m .

affected zone. A slight decrease of the maximum precipitate radius and a slight increase of the solid solution concentration at minimum yield strength can be observed when the rotational speed decreases which causes a slight decrease of the dynamic recovery rate β and of the dislocation storage parameter θ . The minimum yield strength, strain at necking and ultimate tensile strain are almost independent of the rotational speed. This analysis is consistent with the general observation found at several steps of our study that the rotational speed is not a primary factor of the process except for its effect on the width of the thermally affected zone.

10.1.2 Prediction of the global tensile properties

Figure 10.6 compares with the experimental results the mechanical properties of the welds predicted using the complete model chain. Globally the effect of the advancing speed on the properties is well captured.

Cold welds (i.e. weld pitch larger than 0.7). Figure 10.6(a,b,c,d) shows that the yield strength, the ultimate tensile strength and the ultimate tensile strain are correctly reproduced for the welds with the high advancing speeds (i.e. the cold welds). Nevertheless, for these welds, necking is predicted in the weld center which does not agree with the experimental observation.

Hot welds (i.e. weld pitch lower than 0.7). Figure 10.6(a,b) shows that the yield strength of the welds with the low advancing speeds (i.e.

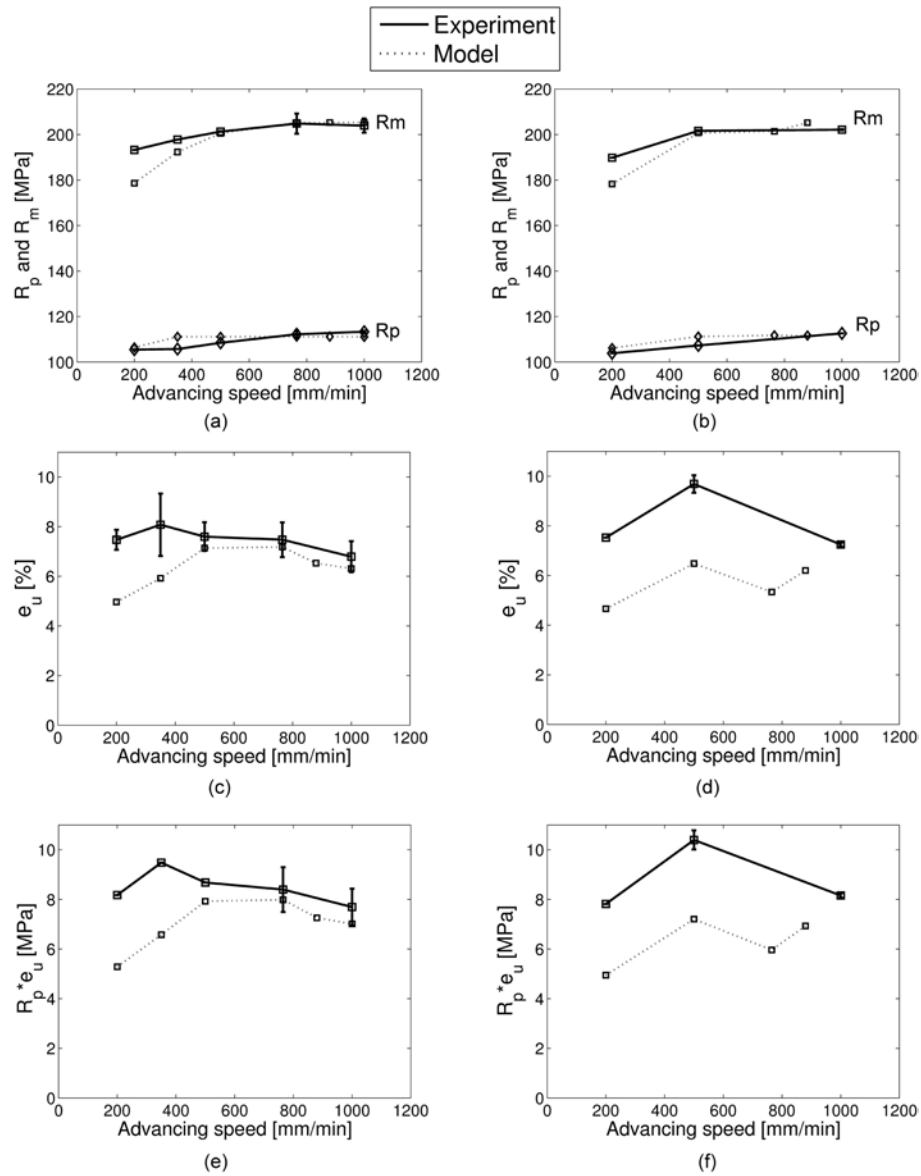


Figure 10.6. Comparison of the measured and predicted (by the modeling chain) engineering transverse tensile properties for different advancing speeds for (a,c,e) a rotational speed of 1000 rpm; (b,d,f) a rotational speed equal to 750 rpm. (a,b) Yield strength R_p and ultimate tensile strength R_m ; (c,d) ultimate tensile strain e_u ; (e,f) yield strength R_p [MPa] multiplied by the ultimate tensile strain e_u (image of the toughness of the welds).

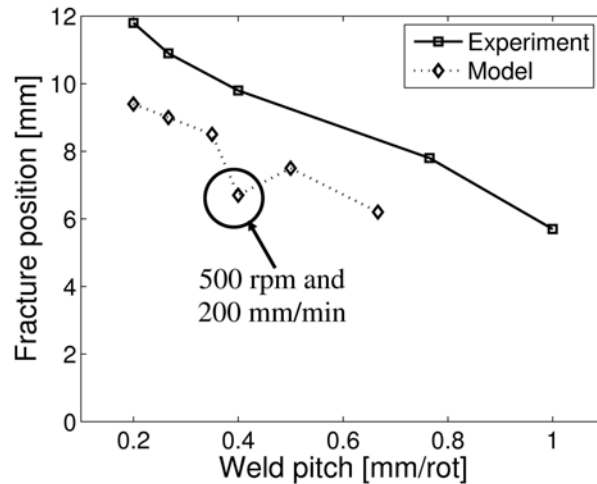


Figure 10.7. Comparison of the predicted and measured fracture position as a function of the weld pitch (ratio of the advancing speed to the rotational speed).

the hot welds) is well predicted. The ultimate tensile strength and the ultimate tensile strain of these welds are underestimated. For these welds necking is well predicted in the heat affected zone and on the retreating side of the weld, like in the experiments. Figure 10.7 shows the predicted and experimental variation of the fracture position (distance from the weld centerline) as a function of the weld pitch¹. The position of fracture is determined in the model based on the position where necking starts. Figure 10.7 shows that the fracture position is predicted slightly too close to the weld center. The out-of-the-trends effect of the weld pitch for weld with a rotational speed equal to 500 rpm is difficult to explain.

Toughness indicator. Figure 10.6(e,f) shows a maximum of the toughness indicator $R_p * e_u$ for an advancing speed of 500 mm/min associated to the maximum of the ultimate tensile strain for that advancing speed. Nevertheless, the fact that the fracture position changes between hot and cold welds might artificially affect this prediction.

Assessment of the damage model using the modeling chain. For the cold welds necking starts in the weld center. The triaxiality history in that zone is smaller than in the true fracture zone which would lead to overestimations of the ductility. Hence, improper prediction of

¹The cold welds have not been included in the model results of Figure 10.7 since fracture is predicted in the weld center for these welds.

the necking position has a major influence on fracture analysis. Nevertheless, we attempted the modeling. The overestimation of the true fracture strain is found to be less than 10%. For the hot welds, up to now, numerical problems do not allow to get converged results. These numerical problems come from the very limited size of the weakest zone compared to what was introduced in section 9.2. Hence, deformation localizes in a very confined zone (one or two finite elements) since too little finite elements are available in the weak zone for a section reduction to be predictable. This problem could probably be solved by adaptive remeshing in the zone of large deformations.

10.2 Discussion

This integrated modeling is not only useful as a predictive tool in applications, it also improves the understanding of several coupled effects which could not be explained by treating the phenomena independently.

10.2.1 Precipitation in the weld center

The evolution of the microstructure and of the mechanical properties in the weld center can be predicted based on the thermal model. Remember that no thermocouple could be inserted in the weld mechanically affected zone since they would be damaged by the welding process. Figure 10.8 shows that, in the weld nugget, the fine precipitates dissolve (the precipitates density decreases) during heating. When the friction stir welding tool moves away, the temperature decreases favoring re-precipitation in the weld nugget. Such precipitates have not been observed by TEM. The predicted very low density and relatively low equivalent radius could explain why they have not been observed experimentally. Nevertheless, the prediction of the presence of these precipitates in the weld center could be an artifact of the model. Indeed, it has already been discussed in section 6.1.3 that precipitation of β'' is favored if germs are available (GP zones in particular). The model does not take this effect into account and assumes that germs are always available. The presence of these germs is not obvious during a rapid thermal cycle at high temperatures.

10.2.2 Zone of deformation localization in cold welds and effect of natural aging

When the difference in tensile properties (and in particular in ultimate tensile strength) between the weld center and the heat affected

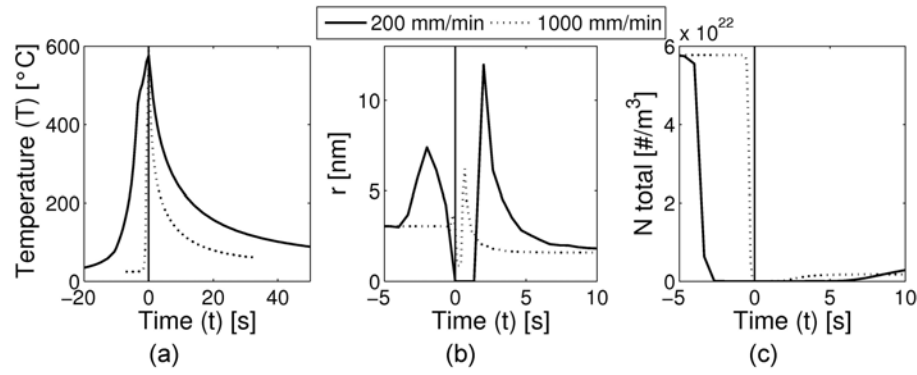


Figure 10.8. Re-precipitation during the cooling cycle in the weld center for two friction stir welds with rotational speed of 1000 rpm. (a) Temperature cycles as a function of time, time = 0 at maximum; (b) Equivalent radius as a function of time; (c) Precipitates total density as a function of time.

zone is smaller, like in the coldest welds (see Figure 10.1(a)), the necking in a transverse tensile sample is predicted in the weld center rather than in the heat affected zone like it is experimentally observed. The reason for this discrepancy is probably that the precipitate radius of the heat affected zone of these welds was underestimated causing an underestimation of the "dislocation lift" effect (i.e. an underestimation of the dynamic recovery rate β). The large peak in the dynamic recovery rate β is the main reason for the lower ultimate tensile strain in the heat affected zone compared to the weld center. Note that we are in a situation where tiny variations in parameters have a large impact on the predictions. Hence, these predictions do not cast doubts about the model but only confirms some of its quantitative limitations.

The effect of the solid solution concentration is also fundamental to understand the difference in the ultimate tensile strength between the weld center and the heat affected zone. Indeed, once the solid solution concentration is sufficient for natural aging to occur (i.e. the solid solution concentration C_{Mg} exceeds a critical value C_{lim}), the yield strength of the heat affected zone will drastically increase affecting the predicted value of the ultimate tensile strength. Figure 10.9 shows that the difference between the ultimate tensile strength of the weld center and the heat affected zone is largely affected by the value of C_{lim} . It is also interesting to note that very low values of C_{lim} would give barely no difference between the ultimate tensile strength of the weld center and the heat affected zone. As a side effect, this is a confirmation that the choice of a

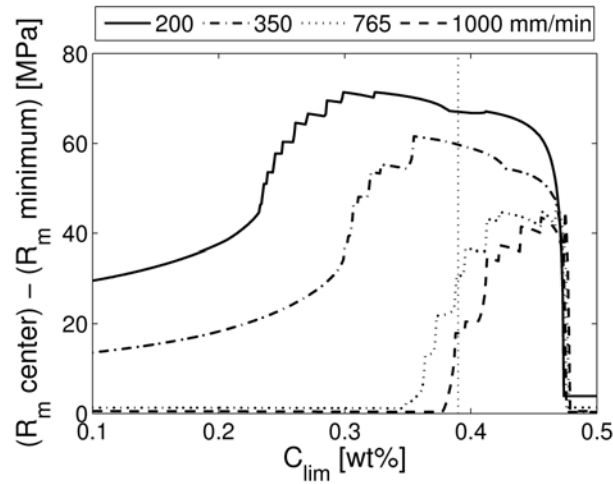


Figure 10.9. Difference in the tensile strength between the weld center and the heat affected zone as a function of the limit solid solution concentration for natural aging C_{lim} for welds with a rotational speed equal to 1000 rpm and various advancing speeds. The vertical line indicates the value of C_{lim} chosen in this study.

rather high value of C_{lim} (see section 7.3.2) correctly reflects the behavior of the friction stir welds in 6005A that does present a lower ultimate tensile strength in the heat affected zone than in the weld center.

10.2.3 Strain hardening capacities of hot welds

Figure 10.6 has shown that the ultimate tensile strength and strain are underestimated for hot welds. Figure 10.1(b) has shown that the dynamic recovery rate β is overestimated in the heat affected zone in hot welds. Hence, the strain hardening capacity of this zone is slightly overestimated causing premature necking when subjecting the weld to transverse tensile testing. Figure 10.10 shows that, for the hottest weld (200 mm/min and 1000 rpm), increasing the minimum ultimate tensile strength to the value experimentally measured by the micro-tensile samples (about 180 MPa) gives a much better prediction of the ultimate tensile strength of the weld.

10.2.4 Asymmetry

It was shown in chapter 6 (Figure 6.22) that the yield strength of the retreating side of the weld is lower than the yield strength of the advancing side of the weld even though the advancing side is the hottest

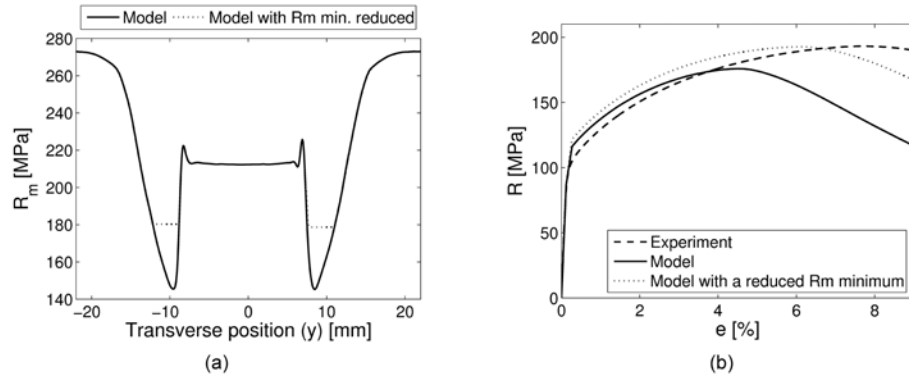


Figure 10.10. Effect of increasing the minimum ultimate tensile strength to the minimum value measured on the micro-tensile samples on the predicted engineering tensile curve of weld number "3" (200 mm/min and 1000 rpm): (a) corrected distribution of the ultimate tensile strength R_m distribution; (b) consequence on the engineering tensile curve.

side of the weld. Assembling the models help understanding this aspect specific to the friction stir welding process. Figure 10.11 presents the variation of the precipitates radius and volume fraction as well as of the solid solution concentration at the position where the local yield strength and ultimate tensile strength are minimum² for both advancing and retreating sides of the weld as a function of the advancing speed for two different rotational speeds. Figure 10.11 shows that³:

- $r_A < r_R$. The maximum precipitate radius is slightly larger on the retreating side than on the advancing side of the weld. This difference is equal to only 0.1 nm. This gives an advancing side of the weld slightly stronger than the retreating side of the weld.
- $C_{MgA} > C_{MgR}$. The solid solution concentration C_{Mg} is slightly higher on the advancing side of the weld than on the retreating side of the weld. The contribution of the solid solution to the yield strength (called σ_{ss} in chapter 7) is thus larger on the advancing side than on the retreating side of the weld. The higher solid solution concentration on the advancing side of the weld also favors

²Generally speaking, these two minimum are at the same distance from the weld centerline but the calibration of the curves has been made on the ultimate tensile strength minimum. The positions of the precipitate radius maximum are also quite close to the yield strength and ultimate tensile strength minimum.

³The subscript A and R correspond to the values respectively on the Advancing side of the weld and on the Retreating side of the weld.

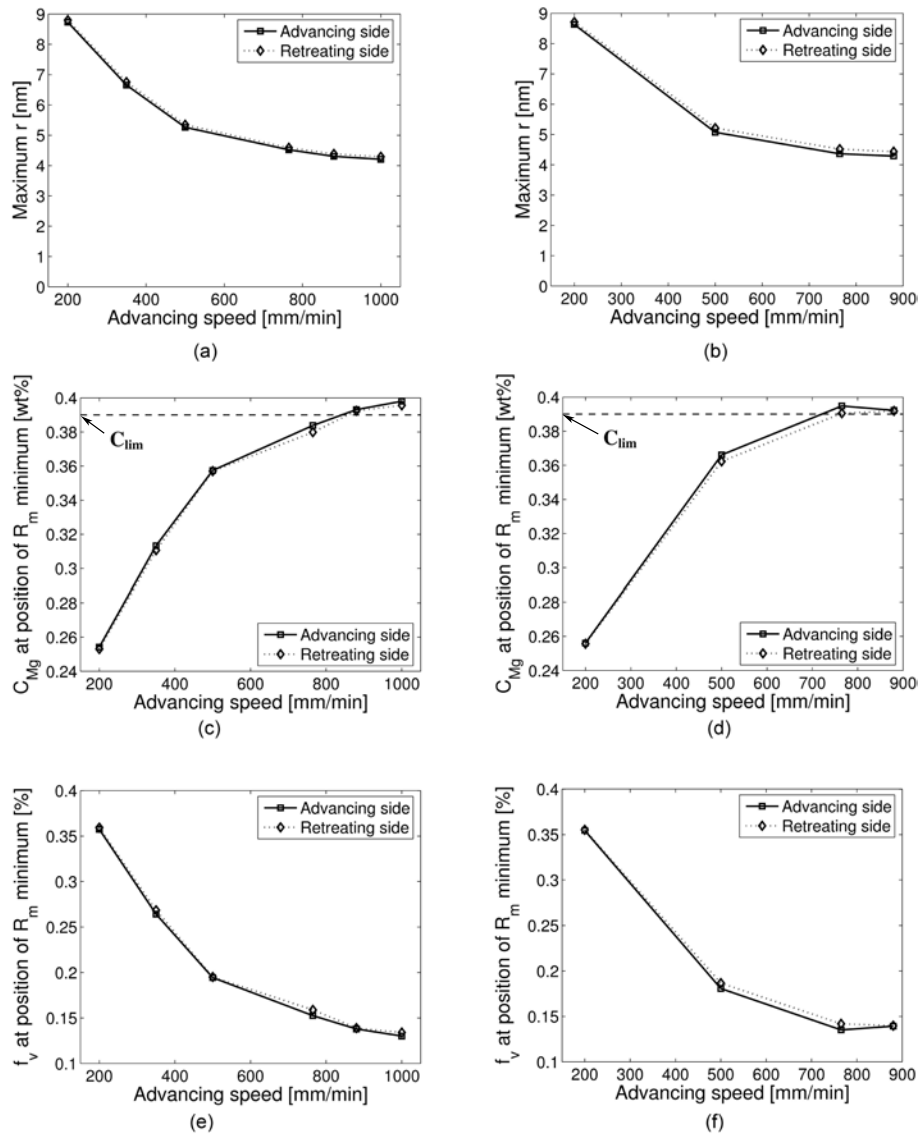


Figure 10.11. Effect of the advancing speed on (a,b) the maximum radius of precipitates r ; (c,d) solid solution concentration C_{Mg} at the position where the ultimate tensile strength is minimum; (e,f) volume fraction of precipitates f_v at the position where the ultimate tensile strength is minimum comparing the advancing and the retreating side of the weld; (a,c,e) the rotational speed is equal to 1000 rpm; (b,d,f) the rotational speed is equal to 750 rpm.

natural aging if the limit solid solution concentration for natural aging C_{lim} is reached in the weakest zone of the weld (it is the case for cold welds). Another effect of a high solid solution concentration is to increase the capacity to dynamic precipitation, providing also an advancing side stronger than the retreating side.

- $f_{vA} < f_{vR}$. The volume fraction of precipitates is slightly larger on the retreating side of the weld than on the advancing side of the weld. This gives a retreating side of the weld stronger than the advancing side of the weld.

As a conclusion, the weakest side of the weld results from a competition between minute differences in the contributions to the strengthening effects which explains that apparently contradictory results are published in the literature. It is important to note that the differences between the advancing side of the weld and the retreating side of the weld remain a second order effect compared to the effect of the advancing speed also shown in Figure 10.11.

Figure 10.12 presents the variation of the minimum yield strength, the true strain at necking at the transverse position where the ultimate tensile strength is minimum and the minimum ultimate tensile strength as a function of the advancing speed for two different rotational speeds. The true strain at necking is the only tensile property that is systematically slightly higher on the advancing side of the weld than on the retreating side of the weld⁴. The predicted fracture position of hot welds is on the retreating side of the weld and for these welds the ultimate tensile strength is very slightly lower on that side.

Finally, it should be noted that the competition between the two sides of the weld regarding fracture is probably also affected by the presence of residual stress and the asymmetry in their distribution. Figure 10.13 shows the result of the "cut compliance method" used to measure the residual stresses in the longitudinal direction that V. Pierard [107] has applied to measure the residual stresses of weld number "5". The residual stresses in the longitudinal direction are higher on the retreating side of the weld for that weld extensively studied in this thesis. Even though the residual stresses are generally largely reduced when machining the tensile samples, remnant residual stresses could favor fracture on the retreating

⁴This effect cannot be confirmed by our experimental results since premature necking was systematically observed in the micro-tensile samples.

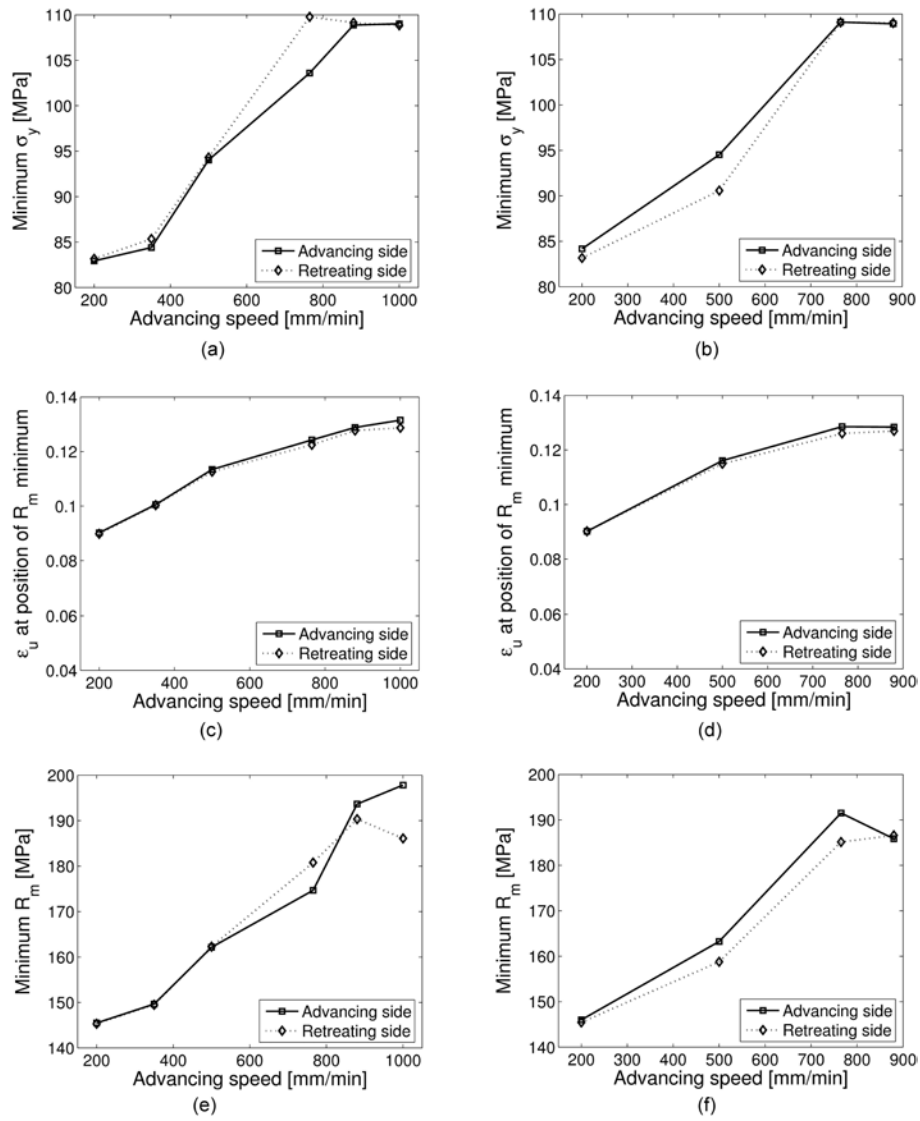


Figure 10.12. Effect of the advancing speed on the (a,b) minimum yield strength σ_y ; (c,d) true strain at necking ϵ_u at the position where the ultimate tensile strength is minimum; (e,f) minimum ultimate tensile strength R_m , comparing the advancing and the retreating side of the weld; (a,c,e) the rotational speed is equal to 1000 rpm; (b,d,f) the rotational speed is equal to 750 rpm.

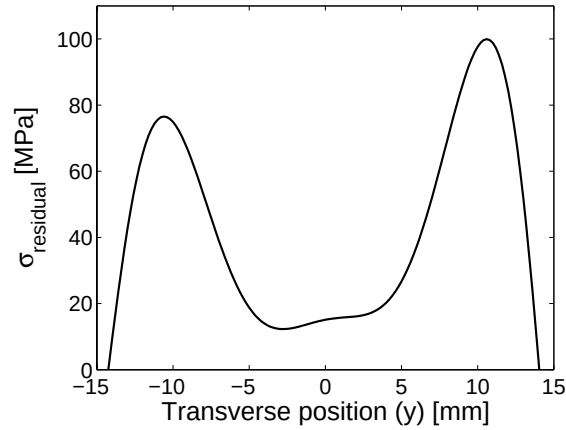


Figure 10.13. Residual stresses in the longitudinal direction σ_{residual} as a function of the position along the transverse section of a friction stir weld with welding parameters 1000 mm/min and 1000 rpm (results from [107]).

side of the weld when subjecting a friction stir weld to transverse tensile loading.

10.3 Conclusion

In this chapter the models presented in part II and III of this thesis have been assembled. Encouraging results have been found. In particular, the models allowed a better understanding of the asymmetry of friction stir welds and the reason why fracture of friction stir welds occur on the retreating side of the weld. The prediction of re-precipitation in the weld center needs experimental confirmation. Natural aging, and in particular the limit solid solution concentration for natural aging, has been shown to tremendously affect the localization process when subjecting a friction stir weld to transverse tensile loading.

Chapter 11

Conclusions

The Friction stir welding process gives high quality welds. Hence, there has been a growing interest for this process over the last 15 years in all areas of industry. Nevertheless, many aspects of the process are still poorly understood and require further studies in order to favor new applications.

11.1 Guidelines

This thesis has been divided in three main parts reflecting the steps required for bridging the process parameters to the end-use tensile properties of friction stir welds¹.

- **Part II** presented the thermal and mechanical cycles that the material overcomes during the welding process. A thermal model including material convection was developed and assessed in order to relate the welding parameters to the thermal cycles.
- **Part III** presented the local microstructures and mechanical properties of the different zones of a friction stir weld. After a complete characterization of the base material and the welds, a microstructure evolution model and a yield strength evolution model have been presented and validated. The third chapter of part III proposed a novel model generalizing the classical Kocks-Mecking-Estrin model for the prediction of strain hardening. In particular, we have introduced the effect of dynamic precipitation, of the reduced efficiency of Orowan loop storage in the case of precipitates coherent with the matrix and of the so-called "dislocation

¹Part I was devoted to the introduction, background, experimental methods and base material presentation.

lift" phenomenon in order to improve the description of the strain hardening behavior. These effects have been described by analytical expressions. More than its interest for the present application as friction stir welds, this model contributes in general to a better understanding of the strain hardening of aluminum alloys.

- **Part IV** presented a model for predicting the tensile behavior of friction stir welds when subjected to transverse tensile loading from the local properties measured by micro-tensile samples. A damage model is also presented in the first chapter of part III. The complete model chain is then assessed for a wide variety of welding conditions.

Through complete experimental characterization and physically based multiscale and multiphysics models, this study has allowed improved understanding and the building of a predictive scheme of the behavior of friction stir welds, in a specific application to aluminum alloy 6005A.

11.2 Specific contributions to the understanding of the behavior of friction stir welds

Importance of the backing plate. The thermal model has enlighten the importance of the backing plate on the cooling of the workpiece. A good contact between the workpiece and the backing plate under the tool and a worst contact behind the tool is needed to accurately reproduce the temperature measurements during welding.

Heat generation during the welding process. Based on thermocouple measurements for a wide variety of welding conditions and the maximum temperature predicted at the hottest point, the thermal model of this study has shown that heat is probably mainly generated within a small surface layer surrounding the tool. Nevertheless, modeling heat generation only by friction (i.e surface heat sources) without introducing the effect of material flow leads to unrealistic predictions of the maximum temperature reached at the hottest point. Introducing material flow through a very simple assumed velocity field is efficient in leading to satisfactory predictions of the thermal cycles during welding.

β precipitates evolution. The β precipitates and its meta-stable precursors are largely affected by the thermal cycles during welding.

- The base material fine precipitates (β'') are largely affected by the thermal cycles with dissolution of these precipitates in the heat affected zone even at the beginning of the hardness drop zone which is not discussed in literature.
- Indications of the precipitation of the β phase in the weld center have been found.

Importance of strain hardening on the transverse tensile behavior of the welds. The weld center is stronger than the heat affected zone of a friction stir weld even though the yield strength of these different zones are similar (especially for high advancing speed welds). The strain hardening model has allowed explaining the low strain hardening capacity of the heat affected zone due to the high local dynamic recovery rate (related to the "dislocation lift" effect) and by the low dislocation storage level (related to un-efficient storage of Orowan loops for medium precipitate radii). Conversely, the weld center has a higher strain hardening capacity than the heat affected zone due to dynamic precipitation which artificially increases the dislocation storage rate. Hence, fracture of transverse tensile samples occurs in the heat affected zone for all the friction stir welds. Nevertheless, the localization process in the heat affected zone is delayed in cold welds due to the smaller differences in strength between the weld center and the heat affected zone compared to that observed in very hot welds.

Natural aging effect on friction stir welds in aluminum alloy 6005A. In aluminum alloy 6005A, a high solid solution concentration is needed for natural aging to proceed probably due to the low copper content of the alloy. Hence, no natural aging occurs in the hardness drop zone of a friction stir weld. The consequence of the absence of natural aging in that region of the weld is to decrease the strength where it is already minimum and to favor the localization of deformation in the heat affected zone under transverse tensile loading.

Effect of the welding conditions on the behavior of friction stir welds. The advancing speed plays a more important role in the weld local properties than the rotational speed at least in the range of welding conditions of this study.

- The heating and cooling rate decrease with increasing advancing speed and are almost not affected by the rotational speed. These heating and cooling rates are essential to the evolution of the

mechanical properties of the heat affected zone (the weakest zone of the weld). Indeed, the microstructure evolution model has shown that the maximum radius increases if the cooling rate decreases.

- The volume fraction of precipitates at minimum hardness is much higher for low advancing speed welds and seems only weakly affected by the rotational speed. The volume fraction of precipitates in the minimum hardness zone tremendously affects the capacity for weld property improvement through post-welding aging (including natural aging).

The power input, equal to the total heat input during the process, increases with increasing advancing speed and is only weakly affected by the rotational speed. The joint efficiency (ratio of the ultimate tensile strength of the weld to the ultimate tensile strength of the base material) increases with increasing advancing and increasing rotational speeds. As a consequence, the weld pitch (ratio of the advancing speed to the rotational speed), often used as the unique parameter for describing the friction stir welding process, is certainly not the sole parameter to consider. The only observations that correlate well with the weld pitch, are the maximum temperature away from the centerline and the fracture position of a transverse tensile sample of a weld (linked to the thermally affected zone width).

Asymmetry of the friction stir welds. The fact that the advancing side of the weld is generally hotter than the retreating side of the weld has been explained by a material convection effect. Even if the advancing side of the weld is hotter, the retreating side is the weakest side of the weld and is the side where fracture of a transverse tensile sample will always occur. The full model predicts a competition between different strengthening effects (i.e. precipitation strengthening and the solid solution strengthening), which, for many welding conditions, causes the retreating side to be the weakest side of the weld.

Optimal welding conditions. Indications of a maximum of the toughness of the welds (the yield strength multiplied by the ultimate tensile strain) has been found for an advancing speed equal to 500 mm/min and a rotational speed equal to 750 rpm. These conditions do not correspond to a maximum joint efficiency since the joint efficiency increases with increasing advancing speed and rotational speed. Hence, we go back to one statement of the introduction of this thesis: *high welding speeds should not be in conflict with the quality of the welds produced...* but, compromises can be found.

11.3 Perspectives

It would be of great interest to extend the present study to the following aspects.

Welding forces and torque evolution. The effect of the welding parameters on the welding forces and torque has been qualitatively discussed in this study based on our measurements. It has been shown that the power input is mainly affected by the advancing speed. Our results have been confronted to literature but no model has been developed to predict the welding forces and torque. Further understanding and predicting these effect is certainly of great interest.

Contact conditions between the workpiece and the backing plate. Since thermal contact have an essential influence on the thermal cycles, there is a need for further understanding the contact conditions prevailing during the friction stir welding process. Indeed, no clear explanation of why the thermal model overestimates the temperature in the backing plate can be given at this stage. More temperature measurements in the backing plate could be suggested as a way to address this point.

Material flow visualization and modeling. Concerning flow visualization, the α particles have been shown to be broken up by the large deformations in the weld center and to follow the direction of the marks left by the tool on the top surface of a weld. Hence, studying the distribution of these particles could be a way to better understand the flow patterns in the thermomechanically affected zone as has already been addressed by Sutton *et al.* [130]. Concerning the flow modeling, a critical step in the improvement of the current thermal model would be to predict the velocity field rather than assume a simple distribution of that field. This could be first done by assuming that the material behaves as a viscous fluid.

Predicted re-precipitation in the weld center. The microstructure evolution model predicts that fine precipitates are formed in the weld center when the weld cools down. No evidences of this re-precipitation have been found in the microstructure characterization. This point needs either confirmation or eventually calls for better capturing the precipitate nucleation stage (from GP zones) with the microstructure model.

Need for heat treated samples precipitates characterization.

The strain hardening model parameters have been identified based on the predictions of the microstructure evolution model since TEM observations and image analysis were too time consuming. In order to support the strain hardening model calibration and validation, small angle X-ray scattering (SAXS) measurements on heat treated samples could be suggested as a way to assess experimentally the fine precipitates evolution with heat treatment time.

Applying the models to other alloys. The full modeling chain has been validated on a specific aluminum alloy (i.e. the aluminum alloy 6005A). It would be interesting to validate this modeling chain on a wider range of materials keeping in mind that the microstructure evolution model is valid only for materials which involve homogeneous precipitation. Nevertheless, solutions for taking heterogeneous precipitation into account can be found in the literature [50].

Effect of the residual stresses. The residual stresses of our friction stir welds have been measured in a parallel study [107] by the "cut compliance" method. Predicting and understanding the influence of these residual stresses on the global behavior of a friction stir weld would be useful for the industrial application of the process.

Applying the damage model as a part of the modeling chain.

The damage model could not be applied when assembling all the models due to numerical convergence difficulties related to steep properties gradient and problem with mesh refinement. Adaptive re-meshing could certainly help solving that problem.

Effect of β precipitation in the weld center on its true fracture strain. Differential scanning calorimetry and post-welding heat treatment results gave hint that a precipitation of $\beta(\text{Mg}_2\text{Si})$ occurs in the weld center. This precipitation has no effect on the true fracture strain of classical friction stir welds since fracture occurs in the heat affected zone which is not concerned by β precipitation. Nevertheless, the effect of this precipitation on the true fracture strain of the nugget would certainly be interesting to understand in the perspective of friction stir processing (i.e. new process that aims at giving to a material the properties of the weld nugget).

Effect of the dispersoid precipitation on the true fracture strain.

Gallais *et al.* [50, 51] have shown that dispersoids can play an important role in the fracture of aluminum alloys. The density of dispersoids of the 6005A aluminum alloy is much lower than that of the 6056 alloy studied by Gallais *et al.* which probably justifies the fact that dispersoids do not play a dominant role in the damage mechanism of the 6005A aluminum alloy. Determining the limiting dispersoids density that start to affect the damage mechanism of aluminum alloys would certainly be of great interest in materials optimization.

Bibliography

- [1] ABAQUS, Version 6.3, 2002, User's Manual, Hibbit, Karlsson and Sorensen, Providence, RI
- [2] S.J. Anderson, H.W. Zandbergen, J. Jansen, C. Traeholt, U. Tundal and O. Reiso : The crystal structure of the β'' phase in Al-Mg-Si alloys, *Acta Mater.*, 46(9) (1998), 3283-3298
- [3] A. Askari, S.T. Silling, B. London and M. Mahoney : Modeling and analysis of friction stir welding process, *Proc. FSW Visu. Model. Seminar*, Germany, 2003
- [4] Z.S. Basinski : Thermally activated glide in face-centered cubic metals and its application to the theory of strain-hardening, *Phil. Mag.* 4 (1959), 393-432
- [5] G.J. Bendzsak, T.H. North and C.B. Smith : An experimentally validated 3D model for friction stir welding, *Proc. 2th Int. Symp. FSW*, Gothenburg, Sweden, 2000
- [6] A.A. Benzerga, J. Besson and A. Pineau: Anisotropic ductile fracture - Part II: theory, *Acta Mater.*, 52 (2004), 4639-4650
- [7] Y. Brechet and F. Louchet : On the stability of orowan loops around coherent spherical ordered precipitates, *Acta Metall.*, 37(9) (1989), 2469-2473
- [8] G. Buffa, J. Hua, R. Shivpuri and L. Fratini : A continuum based FEM model for friction stir welding-model development, *Mat. Sci. Eng. A*, 419 (2006), 389-396
- [9] G.B. Burger, A.K. Gupta, P.W. Jeffrey and D.J. Lloyd : Microstructural control of aluminum sheet used in automotive applications, *Materials Characterization*, 35 (1995), 23-39
- [10] G. Bussu and P.E. Irving : The role of residual stress and heat affected zone properties on fatigue crack propagation in friction stir welded 2024-T351 aluminium joints, *Int. J. of Fatigue*, 25 (2003), 77-88
- [11] M. Cabibbo, E. Meccia, E. Evangelista : TEM analysis of a friction stir-welded butt joint of Al-Si-Mg alloys, *Mater. Chemistry and Physics*, 81 (2003), 289-292
- [12] Y. J. Chao and X. Qi : Thermal and thermomechanical modeling of friction stir welding of aluminum alloy 6061-T6, *J. Mater. Process. Manuf. Sci.* 7 (1998), 215-233

- [13] Y.J. Chao, X. Qi, W. Tang : Heat Transfer in Friction Stir Welding - Experimental and Numerical Studies, ASME Journal of Manufacturing Science and Engineering, 125 (2003), 138-145
- [14] C.M. Chen and R. Kovacevic : Finite element modeling of friction stir welding-thermal and thermomechanical analysis, Int. J. Mach. Tools Manuf., 43 (2003), 1319-1326
- [15] L.M. Cheng, W.J. Poole, J.D. Embury and D.J. Lloyd : The Influence of Precipitation on the Work-Hardening Behavior of the Aluminum Alloys AA6111 and AA7030, Metall. Mater. Trans. A, 34A (2003), 2473-2481
- [16] P. Colegrove, M. Painter, D. Graham and T. Miller : Three-Dimensional flow and thermal modelling of the friction stir welding process, Proc. 2nd Int. Symp. on Friction stir welding, Gothenburg, Sweden, June 2000
- [17] P.A. Colegrove and H.R. Shercliff : Experimental and numerical analysis of aluminium alloy 7075-T7351 friction stir welds, Sci. Technol. Weld. Joining, 8(5) (2003), 360-368
- [18] P.A. Colegrove and H.R. Shercliff : Two-dimensional CFD modelling of flow round profiled FSW tooling, Sci. Technol. Weld. Joining, 9(6) (2004), 483-492
- [19] P.A. Colegrove and H.R. Shercliff : Development of Trivex friction stir welding tool Part 1 - two-dimensional flow modelling and experimental validation, Sci. Technol. Weld. Joining, 9(4) (2004), 345-351
- [20] P.A. Colegrove and H.R. Shercliff : Development of Trivex friction stir welding tool Part 2 - three-dimensional flow modelling, Sci. Technol. Weld. Joining, 9(4) (2004), 352-361
- [21] D. Chu and J.W. Morris : The influence of microstructure on work hardening in aluminium, Acta Mater., 44 (7) (1996), 2599-2610
- [22] C. Dale Donne, E. Lima, J. Wegener, A. Pyzalla, T. Buslaps : Investigations on residual stresses in friction stir welds, Proc. 3rd Int. Symp. FSW, Kobe, Japan, Sept. 2001
- [23] A. Deschamps and Y. Brechet : Influence of predeformation and ageing of an Al-Zn-Mg alloy-II. Modeling of precipitation kinetics and yield stress, Acta Mater., 47 (1999), 293-305
- [24] A. Deschamps, Y. Brechet, C.J. Necker, S. Saimoto and J.D. Embury : Study of large strain deformation of dilute solid solutions of Al-Cu using channel-die compression, Mater. Sci. Eng. A, 207 (1996), 143-152
- [25] A. Deschamps, Y. Brechet and F. Livet : Influence of copper addition on precipitation kinetics and hardening in Al-Zn-Mg alloy, Mater. Sci. Technol., 15 (1999), 993-1000
- [26] A. Deschamps, M. Nieuwczas, F. Bley, Y. Brechet, J.D. Embury, L. Le Sinq, F. Livet and J.P. Simon : Low-temperature dynamic precipitation in a super-saturated Al-Zn-Mg alloy and related strain hardening, Philo. Mag. A, 79(10) (1999), 2485-2504

- [27] T. De Vuyst, L. D'Alvise, A. Simar, B. de Meester and S. Pierret : Finite Element Modeling of friction stir welding of aluminium alloy plates-inverse analysis using a genetic algorithm, *Welding in the World*, 49(3/4)(2005), 47-55
- [28] Demi-produits aluminium : Pechiney Rhenalu, may 1997
- [29] A. Denquin, D. Allehaux, M.-H. Campagnac and G. Lapasset : Microstructural evolution and strength mismatch within a friction stir welded 6056 aluminium alloy, *Proc. 3rd Int. Symp. FSW*, Kobe, Japan, Sept. 2001
- [30] A. Denquin, D. Allehaux, M.-H. Campagnac and G. Lapasset : Relationship between microstructural variations and properties of a friction stir welded 6056 aluminium alloy, *Welding in the World*, 46(11/12) (2002), 14-19
- [31] T. Dickerson, Q. Shi and H.R. Shercliff : Validation of friction stir welding process models, *Proc. 4th Int. Symp. FSW*, Park City, Utah, USA, May 2003
- [32] P. Dong, F. Lu, J.K. Hong and Z. Cao : Coupled thermomechanical analysis of friction stir welding process using simplified models, *Sci. Technol. Weld. Joining*, 6(5)(2001), 281-287
- [33] S.D. Dumolt, D.E. Laughlin and J.C. Williams : Formation of a modified β' phase in aluminum alloy 6061, *Scripta Metal.*, 18 (1984), 1347-1350
- [34] I. Dutta and S.M. Allen : A calorimetric study of precipitation in commercial aluminium alloy 6061, *J. of materials Sc. Letters*, 10 (1991), 323-326
- [35] G.A. Edwards, G.L. Dunlop and M.J. Couper : Fine-scale precipitation in Al alloy 6061, *4th Int. Conf on Aluminium alloys*, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA, 1994, p. 668
- [36] G.A. Edwards, K. Stiller, G.L. Dunlop and M.J. Couper : The precipitation sequence in Al-Mg-Si alloys, *Acta mater.*, 46(11) (1998), 3893-3904
- [37] S. Esmaili, L.M. Cheng, A. Deschamps, D.J. Lloyd and W.J. Poole : The deformation behaviour of AA6111 as a function of temperature and precipitation state, *Mat. Sci. Eng. A*, 319-321 (2001), 461-465
- [38] S. Esmaili, X. Wang, D.J. Lloyd and W.J. Poole : On the Precipitation-Hardening Behavior of the Al-Mg-Si-Cu Alloy AA6111, *Metallur. Materials Trans A*, 34 (2003), 751-763
- [39] S. Esmaili, D.J. Lloyd and W.J. Poole : Modeling of precipitation hardening for the naturally aged Al-Mg-Si-Cu alloy AA6111, *Acta Mater.* 51 (2003), 3467-3481
- [40] O.S. Es-Said, J.G. Morris and H.D. Merchant: Homogenization and Annealing of Aluminium and Copper Alloys, H.D. Merchant *et al.*, Metallurgical Society, 1998, 183
- [41] Y. Estrin and H. Mecking : A unified phenomenological description of work hardening and creep based on one-parameter models, *Acta Metall.*, 32 (1) (1984), 57-80

- [42] Y. Estrin: Unified Constitutive Laws of Plastic Deformation, A.S. Krausz, K. Krausz (Eds.), Academic Press, San Diego, 1996, 69-106
- [43] L. Fourment, S. Guerdoux, M. Miles and T. Nelson : Numerical simulation of the friction stir welding process using both Lagrangian and Arbitrary Lagrangian Eulerian formulations, Proc. 5th Int. Symp. FSW, Metz, France, Sept. 2005
- [44] G. Fribourg: Relation entre microstructure et propriétés mécaniques dans un alliage d'aluminium de la série 7xxx, Master report (2005), Université de Technologie de Compiègne (UTC), France
- [45] O. Frigaard, O. Grong, B. Bjorneklett and O. Midling : Modelling of the thermal and microstructure fields during friction stir welding of aluminium alloys, Proc. 1st Int. Symp. FSW, Thousand Oaks, California, USA, June 1999
- [46] O. Frigaard: A process model for friction stir welding of age hardening aluminium alloys, Ph.D. Thesis (1999), The Norwegian University of Science and Technology (NTNU)
- [47] O. Frigaard, O. Grong, O.T. Midling : A Process Model for Friction Stir Welding of Age Hardening Aluminum Alloys, Metal. Mater. Trans. A, 32A (2001), 1189-1200
- [48] H. Fujii, L. Cui, M. Maeda and K. Nogi : Effect of tool shape on mechanical properties and microstructure of friction stir welded aluminum alloys, Mat. Sci. Eng. A, 419 (2006), 25-31
- [49] C. Gallais, A. Denquin, A. Pic, A. Simar, T. Pardoen and Y. Bréchet : Modelling the relationship between process parameters, microstructural evolutions and mechanical behaviour in a friction stir welded 6XXX aluminium alloy, Proc. 5th Int. Symp. FSW, Metz, France, Sept. 2005
- [50] C. Gallais: Joints soudés par friction malaxage d'alliages d'aluminium de la série 6xxx: caractérisation et modélisation, Ph.D. Thesis (2005), Institut National Polytechnique de Grenoble, France
- [51] C. Gallais, A. Simar, D. Fabrègue, A. Denquin, G. Lapasset, B. de Meester, Y. Bréchet, T. Pardoen : Multiscale analysis of the strength and ductility of 6056 aluminium friction stir welds, to be published
- [52] V. Gerold, Precipitation Hardening IN "Dislocations in Solids", North-Holland Publishing Company (Eds.), Netherlands, 1979, p234
- [53] M. Goerdeler and G. Gottstein : A microstructural work hardening model based on three internal state variables, Mater. Sci. Eng. A, 309-310 (2001), 377-381
- [54] M. Gologanu, J.B. Leblond, G. Perrin and J. Devaux: Recent extensions of Gurson's model for porous ductile metals, P. Suquet editor, Continuum Micromechanics, Springer-Verlag, Berlin, 61-106
- [55] S. Gregory, Friction Stir Welding - State of the Art, ALUWELD Report, catholic University of Louvain-la-Neuve, Dec. 2004

- [56] A.K. Gupta, D.J.Lloyd and S.A. Court : Precipitation hardening processes in an Al-0.4%Mg-1.3%Si-0.25%Fe aluminum alloy, *Mater. Sci. Eng A*, 301 (2000), 140-146
- [57] A.K. Gupta, D.J.Lloyd and S.A. Court : Precipitation hardening in Al-Mg-Si alloys with and without excess Si, *Mater. Sci. Eng A*, 316 (2001), 11-17
- [58] A.K. Gupta and D.J.Lloyd : The precipitation in a super purity Al-Mg-Si alloy, 3rd Int. Conf. Aluminium alloys
- [59] A.L. Gurson: Continuum theory of ductile rupture by void nucleation and growth: Part I- Yield criteria and flow rules for porous ductile media, *J. Eng. Mater. Tech.*, 99 (1977), 2-15
- [60] P. Haasen: Solid Solution Hardening, in *Dislocations in Solids* edited by FRN Nabarro, North-Holland, Amsterdam, 1979, Vol 4, p155
- [61] P. Heurtier, M.J. Jones, C. Desrayaud, J.H. Driver, F. Montheillet and D. Allehaux : Mechanical and thermal modelling of Friction Stir Welding, *J. Mater. processing Tech.*, 171 (2006), 348-357
- [62] H. Hirasawa : Precipitation process of Al-Mn and Al-Cr supersaturated solid solution in presence of age hardening phases, *Scripta Metal.*, 9 (1975), 955-958
- [63] S.M. Hirth, G.J. Marshall, S.A. Court and D.J. Lloyd: Effects of Si on the aging behaviour and formability of aluminium alloys based on AA6016, *Mat. Sci. Eng. A*, 319-321 (2001), 452-456
- [64] J.W. Hutchinson and K.W. Neale : Influence of strain-rate sensitivity on necking under uniaxial tension, *Acta Metall.*, 25 (1977), 839-846
- [65] M.H. Jacobs : The Structure of the Metastable Precipitate Formed During Aging of a Al-Mg-Si Alloy, *Phil. Mag.*, 26 (1972), 1-13
- [66] N. Kamp, A. Sullivan, R. Tomasi and J. D. Robson: Modelling of heterogeneous precipitate distribution evolution during friction stir welding process, *Acta Mater.* (2006), to be published
- [67] A. Kelly and R.B. Nicholson: Precipitation hardening, *Progress in Mater. Sci.*, 10 (1963), p151
- [68] M.Z.H. Khandkar, J.A. Khan and A.P. Reynolds : Prediction of temperature distribution and thermal history during friction stir welding: input torque based model, *Sci. Technol. Weld. Joining*, 8(3)(2003), 165-174
- [69] N.C.W. Kuijpers, W.H. Kool and S. van der Zwaag : DSC study on Mg-Si phases in as cast AA6xxx, *Mater. Sci. Forum*, 396-402 (2002), 675-680
- [70] N.C.W. Kuijpers, W.H. Kool, P.T.G. Koenis, K.E. Nilsen, I.Todd and S. van der Zwaag : Assessment of different techniques for quantification of α -Al(FeMn)Si and β -AlFeSi intermetallics in AA 6xxx alloys, *Mater. Charact.*, 49 (2003), 409-420

- [71] S.G. Lambrakos, R.W. Fonda, J.O. Milewski and J.E. Mitchell : Analysis of friction stir welds using thermocouple measurements, *Sci. Technol. Weld. Joining*, 8(5) (2003), 385-390
- [72] D Lassance, F. Scheyvaerts and T. Pardoen: Growth and coalescence of penny-shaped voids in metallic alloys, *Eng. Fract. Mech.*, 73 (2006), 1009-1034
- [73] D. Lassance: Modeling of the damage mechanism in AlMgSi alloys - Understanding the role of homogenization on the extrudability, Ph.D. Thesis (2006), Université catholique de Louvain, Louvain-la-Neuve, Belgium
- [74] D. Lawrjaniec, A. Abisror, C. Decker, M. Koçak, J. Dos Santos, S. Gardiner: Simulation numérique du procédé de soudage "Friction stir welding", *Soudage et techniques connexes* (may 2003), 3-8
- [75] W.B. Lee, Y.M. Yeon and S.B. Jung : Evaluation of the microstructure and mechanical properties of friction stir welded 6005 aluminum alloy, *Materials Science and Technology*, 19 (2003), 1513-1518
- [76] G. Liu, L.E. Murr, C.S. Niou, J.C. Mc Clure and F.R. Vega : Microstructural aspects of the friction-stir welding of 6061-T6 aluminum, *Scripta Mater.*, 37 (1997), 355-361
- [77] H.J. Liu, H. Fujii, M. Maeda and K. Nogi : Comparative study on fracture locations of friction stir welded joints for different types of aluminum alloys, *Proc. 4th Int. Symp. FSW*, Park City, Utah, USA, May 2003
- [78] L. Lodgaard and N. Ryum : Precipitation of dispersoids containing Mn and/or Cr in Al-Mg-Si alloys, *Mater. Sci. Eng A*, 283 (2000), 144-152
- [79] O. Lohne and A.L. Dons : Quench sensitivity in aluminum-magnesium-silicon alloys containing manganese or chromium, *Scand. J. Met.*, 12 (1983), 34-36
- [80] J.P. Lynch, L.M. Brown and M.H. Jacobs : Microanalysis of age-hardening precipitates in aluminium alloys, *Acta metall.*, 30 (1982), 1389
- [81] Z.Y. Ma, R.S. Mishra and M.W. Mahoney, *Friction Stir Welding and Processing II - Proc. TMS*, 2003, 221-230
- [82] M. Maeda, H. Liu, H. Fujii and T. Shibayanagi : Temperature Field in the Vicinity of FSW-Tool during Friction Stir Welding of Aluminum Alloys, *IIV Meeting*, Nagoya, Japan, 2004, 221-227
- [83] M.W. Mahoney, C.G. Rhodes, J.G. Flintoff, R.A. Spurling, W.H. Bingel : Properties of Friction-Stir-Welded 7075 T651 Aluminum, *Metall. Mater. Trans. A*, 29 (1998), 1955-1964
- [84] K. Matsuda, Y. Uetani, H. Anada, S. Tada and S. Ikeno: *Aluminium Alloys: Their Physical and Mechanical Properties* (Vol 1), L. Arnberg *et al.*, Norwegian Institute of Technology and SINTEF Metallurgy, Trondheim, 1992, p.220
- [85] K. Matsuda, D. Teguri, Y. Uetani, T. Sato and S. Ikeno : Cu-segregation at the Q'/α -Al interface in Al-Mg-Si-Cu alloy, *Scripta Materiala*, 47 (2002), 833-837

- [86] K. Matsuda, Y. Sakaguchi, Y. Miyata, Y. Uteni, T. Sato, A. Kamio and S. Ikeno : Precipitation sequence of various kinds of metastable phases in Al-1.0mass% Mg2Si-0.4mass% Si alloy, *J. Mater. Sci.*, 35 (2000), 179-189
- [87] R.W. Mc Cune, H. Ou, C.G. Armstrong and M. Price : Modelling friction stir welding with the finite element method - a comparative study, 5th Int. Symp. FSW, Metz, France, Sept. 2005
- [88] J.C. Mc Clure, Z. Feng, T.Tang, J.E. Gould, L.E. Murr and X. Guo: A thermal model of friction stir welding, 5th Int. Conf. Trends Welding Research, Pine Mountain, G.A., USA, June 1998, ASM Int., 590-595
- [89] H. Mecking and U.F. Kocks : Kinetics of flow and strain-hardening, *Acta Metall.*, 29 (1981), 1865-1875
- [90] W.F. Miao and D.E. Laughlin: Precipitation hardening in aluminum alloy 6022, *Scripta Mater.*, 40(7) (1999), 873-878
- [91] W.F. Miao and Laughlin D.E. : Effects of Cu Content and Preaging on Precipitation Characteristics in Aluminum Alloy 6022, *Metall. Mater. Trans. A*, 31A (2000), 361
- [92] R.S. Mishra and Z.Y. Ma : Friction stir welding and processing, *Mat. Sci. Eng. R*, 50 (2005), 1-78
- [93] G. Moreau and Q. Van Haute, Outil à force constante pour le soudage par Friction Malaxage sur fraiseuse à commande numérique, End of Study work at catholic University of Louvain-la-Neuve, Belgium, June 2004
- [94] G. Mrowka-Nowotnik and J. Sieniawski : Influence of heat treatment on the microstructure and mechanical properties of 6005 and 6082 aluminium alloys, *J. Mater. Processing Technology*, 2005, 162-163, 367-372
- [95] M. Murayama and K. Hono : Pre-precipitate clusters and precipitation processes in Al-Mg-Si alloys, *Acta Mater.*, 47 (1998), 1537-1548
- [96] O.R. Myhr and O. Grong : Process modelling applied to 6082-T6 aluminium weldments-I. Reaction kinetics, *Acta Metall. Mater.*, 39(11) (1991), 2693-2702
- [97] O.R. Myhr and O. Grong : Process modelling applied to 6082-T6 aluminium weldments-II. Applications of model, *Acta Metall. Mater.*, 39(11) (1991), 2703-2708
- [98] O.R. Myhr and O. Grong : Modelling of non-isothermal transformations in alloys containing a particle distribution, *Acta Mater.*, 48 (2000), 1605-1615
- [99] O.R. Myhr, O. Grong and S.J. Andersen : Modelling of the age hardening behaviour of Al-Mg-Si alloys, *Acta Mater.*, 49 (2001), 65-75
- [100] O.R. Myhr, O. Grong, S. Klokkehaug and H.G. Fjaer: Modelling of the microstructure and strength evolution during ageing and welding of Al-Mg-Si alloys, *Mathematical Modelling of Weld Phenomena*, Bhadeshia HKDH, editor, vol 6 London, Manley Publishing (2002)

- [101] O.R. Myhr, O. Grong, H.G. Fjaer and C.D. Marioara : Modelling of the microstructure and strength evolution in Al-Mg-Si alloys during multistage thermal processing, *Acta Mater.*, 52 (2004), 4997-5008
- [102] A. Needleman and V. Tvergaard: An analysis of ductile rupture in notched bars, *J. Mech. and Phys. of Solids*, 32 (1984), 461-490
- [103] E. Nes, K. Marthinsen and B. Ronning : Modelling the evolution in microstructure and properties during processing of aluminium alloys, *J. Mater. Proc. Tech.*, 117 (2001), 333-340
- [104] M. Nicolas and A. Deschamps : Characterisation and modelling of precipitate evolution in an Al-Zn-Mg alloy during non-isothermal heat treatments, *Acta Mater.*, 51 (2003), 6077-6094
- [105] T. Pardoen and J.W. Hutchinson: An extended model for void growth and coalescence, *J. Mech. and Phys. of Solids*, 48 (2000), 2467-2512
- [106] T. Pardoen and J.W. Hutchinson: Micromechanics-based model for trends in toughness of ductile metals, *Acta. Mater.*, 51 (2003), 133-148
- [107] V. Pierard, Analyse des contraintes résiduelles par la méthode "cut compliance" appliquée aux soudures par friction malaxage, End of Study work at catholic University of Louvain-la-Neuve, Belgium, June 2006
- [108] W.J. Poole and D.J. Lloyd: Modelling the stress-strain behaviour of aluminium alloy AA6111, *Proc. 9th Int. Conf. on Aluminium Alloys* (2004), J.F. Nie, A.J. Morton and B.C. Muddle, *Inst. Mater. Eng. Australasia Ltd. (Eds.)*
- [109] J.H. Record, J.L. Covington, T.W. Nelson, C.D. Sorensen and B.W. Webb : Fundamental Characterization of friction stir welding, 5th Int. Symp. FSW, Metz, France, Sept. 2005
- [110] A.P. Reynolds, W.D. Lockwood and T.U. Siedel : Microstructure of Friction Stir Welded Joints in AA5182, *Mat. Sci. Forum*, 331-337 (2000), 1719-1724
- [111] C.G. Rhodes, M.W. Mahoney, W.H. Bingel, R.A. Spurling and C.C. Bampton : Effects of friction stir welding on microstructure of 7075 aluminum, *Scripta Mater.*, 36 (1997), 69-75
- [112] P. Rosakis, A.J. Rosakis, G. Ravichandran, J. Hodowany : A thermodynamic internal variable model for the partition of plastic work into heat and stored energy in metals, *J. Mecha Physics solids*, 48(3)(2000), 581-607
- [113] Y.S. Sato, H. Kokawa, M. Enmoto and S. Jogan : Microstructural Evolution of 6063 Aluminum during Friction-Stir Welding, *Metall. Mater. Trans. A*, 30 (1999), 2429-2437
- [114] Y.S. Sato, H. Kokawa, M. Enmoto, S. Jogan, T. Hashimoto : Precipitation Sequence in Friction Stir Weld of 6063 Aluminum during Aging, *Metall. Mater. Trans. A*, 30 (1999), 3125-3130
- [115] Y.S. Sato, S.H.C Park, H. Kokawa : Distribution of tensile property and microstructure in friction stir weld of 6063 aluminum, *Metall. Mater. Trans. A*, 32 (2001), 3023-3031

- [116] T.V. Schegoleva: *Phys Metals Metall.*, 25 (1968),56
- [117] H. Schmidt, J. Hattel and J. Wert : An analytical model for the heat generation in friction stir welding, *Modelling Simul. Mater. Sci. Eng.*, 12 (2004), 143-157
- [118] H. Schmidt and J. Hattel : Heat source models in simulation of heat flow in friction stir welding, *Proc. ISOPE-2004*, Toulon, France, N° 2004-FSW-11
- [119] H. Schmidt and H. Hattel : Modelling thermomechanical conditions at the tool/matrix interface in Friction Stir Welding, *Proc. 5th Int. Symp. FSW*, Metz, France, Sept. 2004, TWI
- [120] T.U. Seidel and A.P. Reynolds : Two-dimensional friction stir welding process model based on fluid mechanics, *Sci. Technol. Weld. Joining*, 8(3) (2003), 175-183
- [121] H.R. Shercliff and M.F. Ashby : A process model for age hardening of aluminium alloys-I. The model, *Acta Metall. Mater.*, 38 (1990), 1789-1802
- [122] H.R. Shercliff and M.F. Ashby : A process model for age hardening of aluminium alloys-II. Applications of the model, *Acta Metall. Mater.*, 38 (1990), 1803-1812
- [123] H.R. Shercliff and P.A. Colegrove : Modelling of friction stir welding, *Math. Model. Weld Phenomena* 6 (2002), p.927-974, Manley Publishing, London
- [124] Q. Shi, T. Dickerson and H.R. Shercliff : Thermo-mechanical FE. modelling of friction stir welding of Al-2024 including tool loads, *Proc. 4th Int. Symp. FSW*, Park City, Utah, USA, May 2003
- [125] A. Simar, T. Pardoen and B. de Meester : Influence of friction stir welding parameters on the power input and temperature distribution in aluminium alloys, *Proc. 5th Int. Symp. FSW*, Metz, France, Sept. 2005
- [126] A. Simar, T. Pardoen and B. de Meester : Modelling temperature histories in friction stir welding including material convection effects, *7th Int. Conf. "Trends in Welding Research"*, Pine Mountain, Georgia, USA, May 2005
- [127] A. Simar, J. Lecomte-Beckers, T. Pardoen and B. de Meester : Effect of boundary conditions and heat source distribution on temperature distribution in friction stir welding, *Sci. Technol. Weld. Joining*, 11(2) (2006), 170-177
- [128] M. Song and R. Kovacevic : Thermal modeling of friction stir welding in a moving coordinate system and its validation, *Int. J. Mach. Tools Manuf.*, 43 (2003), 605-615
- [129] J.Q. Su, T.W. Nelson, R.S. Mishra, M.W. Mahoney : Microstructural investigation of friction stir welded 7050-T651 aluminium, *Acta Mater.*, 51 (2003), 713-729
- [130] M.A. Sutton, B. Yang, A.P. Reynolds and R. Taylor : Microstructural studies of friction stir welds in 2024-T3 aluminum, *Mater. Sci. Eng. A*, 323 (2002), 160-166

- [131] M.A. Sutton, A.P. Reynolds, B. Yang and R. Taylor : Mode I fracture and microstructure for 2024-T3 friction stir welds, *Mater. Sci. Eng. A*, 354 (2003), 6-16
- [132] L.E. Svensson, L. Karlsson, H. Larsson, B. Karlsson, M. Fazzini and J. Karlsson : Microstructure and mechanical properties of friction stir welded aluminium alloys with special reference to AA 5083 and AA 6082, *Sci. Technol. Weld. Joining*, 5(5) (2000), 285-296
- [133] W. Tang, X. Guo, J.C. McClure and L.E. Murr : Heat input and temperature distribution in friction stir welding, *J. Mater. Process. Manuf. Sci.*, 7 (1998), 163-172
- [134] G. Thomas : The ageing characteristics of aluminum. alloys : Electron-transmission studies of Al-Mg-Si alloys, *J. Inst. Met.*, 90 (1961-1962), 57-63
- [135] W.M. Thomas, E.D. Nicholas, J.C. Needham, M.G. Murch, P. Templesmith, C. J. Dawes, GB Patent Application No 9125978.8, december 1991; US Patent No 5460317, oct. 1995
- [136] W.M. Thomas, E.D. Nicholas and S.D. Smith: Friction stir welding tool developments, *Aluminium 2001 - Proc. TMS*, 2001, 213-224
- [137] P.F. Thomason: *Ductile Fracture of Metals*, Oxford, Pergamon Press (1990)
- [138] V. Tvegaard: On localization in ductile materials containing voids, *Int. J. of Fracture*, 18 (1982), 237-251
- [139] P. Ulysse : Three-dimensional modeling of the friction stir-welding process, *Int. J. Mach. Tools Manuf.*, 42 (2002), 1549-1557
- [140] T. Ungar, J. Lendvai, I. Groma and J. Illy : The Plastic Behaviour and the Microstructure of an Al - 4.5wt% Zn - 1.5wt% Mg Alloy, *Mater. Sc. Forum*, 13/14 (1987), 389-398
- [141] K.R. Van Horn: *Aluminium properties, physical metallurgy and phase diagrams*, Vol1, 1967
- [142] A. von Strombeck, J.F. dos Santos, F. Torster, P. Laureano and M. Koçak : Fracture toughness behaviour of FSW joints on aluminium alloys, *Proc. 1st Int. Symp. FSW*, Thousand Oaks, California, USA, June 1999
- [143] R.P. Wahi and M. von Heimendahl : Occurrence of the metastable phase β'' in an aluminum-silicon-magnesium alloy, *Phys. Stat. Sol. (a)*, 24 (1974), 607-612
- [144] X.Wang, W.J. Poole, S. Esmaili, D.J. Lloyd and J.D. Embury : Precipitation Strengthening of the Aluminum Alloy AA6111, *Metallur. Materials Trans A*, 34 (2003), 2913-2924
- [145] J. Weertman and J.R. Weertman, *Elementary dislocation theory*, The Macmillian Company, New York
- [146] S. Xu, X. Deng, A.P. Reynolds and T.U. Seidel : Finite element simulation of material flow in friction stir welding, *Sci. Technol. Weld. Joining*, 6(3) (2001), 191-193

-
- [147] B. Yang, J. Yan, M.A. Sutton, and A.P. Reynolds : Banded microstructure in AA2024-T351 and AA2524-T351 aluminum friction stir welds: Part I. Metallurgical studies, Mater. Sci. Eng. A, 364 (2004), 55-65
- [148] R. S. Yasser, D.P. Field and H. Weiland : The effect of predeformation on the β'' and β' precipitates and the role of Q' phase in an Al-Mg-Si alloy, Scripta Materiala, 53 (2005), 299-303
- [149] M. Zahedul, H.Khandkar and J. A. Khan : Thermal modeling of overlap friction stir welding for Al-alloys, J Mater. Process. Manuf. Sci, 10 (2001),91-105
- [150] L. Zhen and S.B. Kang : DSC analyses of the precipitation behavior of two Al-Mg-Si alloys naturally aged for different times, Materials letters, 37 (1998), 349-353