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3D characterization, modelling and tailoring of microstructure heterogeneity effects on damage and fracture of 6xxx aluminium alloys

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Contents

General introduction	9
1 Theoretical background	15
1.1 Damage and fracture	15
1.1.1 Review of the different ductile failure mechanisms	15
1.1.2 Physical length scales entering ductile damage process	19
1.1.3 Nucleation, growth and coalescence of voids . . .	21
1.1.4 Simple fracture strain model	50
1.2 Tomography	55
1.2.1 Short history of the tomography	55
1.2.2 The principle of X-ray tomography	55
1.2.3 X-ray set-ups	56
1.2.4 Recent developments in X-ray tomography	57
1.2.5 Laminography	59
1.2.6 Human vision and impacts for the thesis	60
1.3 Aluminium alloys	63
1.3.1 Standard nomenclature: alloy and temper	63
1.3.2 Fracture toughness of age-hardenable Al alloys .	65
1.4 Friction stir processing (FSP)	69
2 Ductile fracture of 6xxx aluminium alloys	71
2.1 Introduction	72
2.2 Experimental methods	75
2.2.1 Materials	75
2.2.2 Microstructure characterization of base alloys . .	77

Contents

2.2.3	Characterization of mechanical properties and damage	77
2.3	Cellular automaton micromechanics based damage model	80
2.3.1	Synopsis of the model	80
2.3.2	Void nucleation model	81
2.3.3	Void growth model	82
2.3.4	Void coalescence model	84
2.3.5	Final failure criterion	86
2.3.6	Summary of the parameters of the model	87
2.3.7	Numerical procedures	87
2.4	Results and discussion	88
2.4.1	Microstructure and mechanical properties	88
2.4.2	Damage evolution: experimental results and model parameter identification	92
2.4.3	Exploitation and assessment of the model	96
2.4.4	Sensitivity analysis	98
2.5	Conclusion	103
2.6	Additional comments on the onset of necking	106
2.7	Additional microtomography data	111
2.8	3D vizualisation of the automaton cellular	114
2.9	Void tracking and effect of initial porosity	119
3	Ductilization by friction stir processing	123
3.1	Introduction	124
3.2	Materials and experimental methods	127
3.2.1	Materials and FSP	127
3.2.2	SEM and X-ray microtomography	127
3.2.3	Uniaxial tensile tests	128
3.3	Results and discussion	129
3.3.1	Intermetallic particles	129
3.3.2	Initial porosity	138
3.3.3	Summary about microstructural evolution upon FSP	140
3.3.4	Uniaxial tensile tests	141
3.4	Damage model	146
3.5	Conclusions	149
3.6	Supplementary material to Paper FSP	155
3.6.1	Pair correlation function	155
3.6.2	Clustering analysis	161

3.6.3	Effect of FSP on grains	164
3.6.4	Abnormal grain growth	164
3.7	Selective formability	167
3.7.1	Concept of FSP in order to improve formability .	167
3.7.2	Validation of the selective formability concept . .	170
4	Damage and fracture anisotropy	173
4.1	Introduction	174
4.2	Experimental methods	177
4.2.1	Materials	177
4.2.2	Macroscopic tensile tests	177
4.2.3	Microstructure characterization	177
4.2.4	In situ tensile testing coupled to X-ray microto- mography	178
4.3	Results	178
4.3.1	Mechanical properties	178
4.3.2	Microstructure characteristics	179
4.3.3	Damage evolution: experimental results	187
4.4	Discussion on the origins of the failure anisotropy	189
4.5	Conclusion	193
5	Fracture behaviour	197
5.1	Introduction	197
5.2	Experimental methods	199
5.2.1	Materials and processing	199
5.2.2	Geometry of the specimens and precracking method	199
5.2.3	Essential work of fracture	201
5.2.4	J_R -curves experimental procedure	204
5.2.5	Tomography and laminography	206
5.2.6	Projection Digital Image Correlation (P-DIC) methodology	207
5.3	Results	219
5.3.1	Uniaxial tensile test	219
5.3.2	EWf results	219
5.3.3	J_R curve results	227
5.3.4	In situ tensile testing in X-ray tomography	229
5.3.5	CT-like sample testing in laminography	232
5.3.6	Results of the P-DIC procedure	240

Contents

5.4 Discussion	250
Conclusions and perspectives	257
List of figures	261
List of tables	275
Bibliography	279

General introduction

Engineering metallic alloys usually fail in a ductile manner at room temperature, either by void nucleation, growth and coalescence or by mechanical instability of the specimen itself. Ductile fracture has been an active field of research for more than fifty years and is a topic of interest in many technological applications, such as in the automobile and aircraft industries. In damage-controlled failure, deformation remains more or less homogeneous up to the end of the damage percolation process when a crack starts propagating in the solid.

It is well recognized in the field of materials science that structural materials have significant microstructural variations that affect damage properties. However, it is very complicate to integrate this effect in a predictive manner on the assessment of the integrity of engineering structures containing crack-like flaws. The current state of the industry is to certify components through the establishment of design allowables; an expensive program of qualifications specified by the governing industry specification (as issued by standardization groups such as SAE Aerospace Materials Division, ASTM, ... [1]).

General introduction

Within this engineering reality, consideration of the microstructure on the damage and fracture properties brings with it increased demands for :

- **Improved material constitutive laws**

While the current state of understanding of the governing mechanisms leading to ductile fracture is advanced, the translation of mechanisms understanding into predictive engineering models remains a challenge (see, for example, the recent blind-predictions challenges from the Sandia National Laboratories [2]). For example, the use of a Gurson damage model (and extended versions) [3, 4, 5] within a finite element framework has seen many decades of development and has been calibrated to be useful in specific applications, but general predictive ability has not been clearly established [6].

One reason behind this shortcoming is that ductile damage has been traditionally treated as a global process, with the material being often treated with "average" microstructural properties assuming a strict progression from nucleation to coalescence. However, there are considerable experimental evidences which indicate that the ductile damage process is strongly affected by microstructure heterogeneities. One difficulty associated with the introduction of an explicit representation of the microstructure in models is that it is computationally expensive, especially for heterogeneous material which require a larger sample in order to be representative of the entire microstructure.

Significant efforts have gone into refining these "average" models and much progress has been made to account for heterogeneities on damage accumulation [7]. However, there is a natural limit to their accuracy. For example, models based on a more or less periodic void distribution cannot properly reproduce the physical mechanisms of the failure process within heterogeneous materials, i.e. voids growth and that will merge to form micro-cracks that propagate throughout the heterogeneous particles field [8]. Multi-scale computational methods that incorporate the effects of microstructure are still under development [8, 9, 10].

- **New experimental methods**

The difficulty associated with the experimental characterization of ductile damage is due to different reasons. First, it is difficult to examine the void coalescence process because it is a quite sudden phenomenon which occurs within a small material volume. Second, void nucleation is inherently a discontinuous process made of a succession of discrete nucleation events. Furthermore, the damage evolution is a 3D phenomenon. It is thus difficult to perform a statistical treatment of the damage evolution by conventional 2D cross-section or by fracture surface analysis.

Nowadays, X-ray tomography can provide three-dimensional (3D) images of a scanned sample with a spatial resolution below $1\mu\text{m}$. It is thus now possible, for example, to get 3D quantitative information concerning the intermetallic particles responsible for primary void nucleation in aluminium alloys.

Moreover, the recent improvement of in situ tomography and laminography techniques, coupled with advanced image analysis and digital volume correlation, brings new possibilities for studying the evolution and the interaction between plasticity, microstructure and damage at micrometer resolution.

- **Improved microstructural description**

An important issue with accounting for microstructure within damage models is the difficulty to associate measurable properties (most often distributed around an average value) with the model parameters (usually described with simple average value). For example, several experimental studies have previously indicated an increase of void nucleation by particle cracking with increasing particle size, which can be related to a size distribution of internal defects. Therefore, it is necessary to take into account explicitly the distributions of particle sizes to describe the damage initiation process [11]. The model microstructure must be representative of the entire material and advance statistical analysis may be required to determine a representative microstructural parameter which can be connected to failure models in a rational way.

General introduction

Even with hundreds of papers published each year on the topic of ductile fracture (see Fig. 1), the problem of properly treating the microstructural heterogeneity effects on ductile damage is still an open issue [7]. Fig. 1 shows that damage accumulation within heterogeneous material has become an increasingly important topic in recent years.

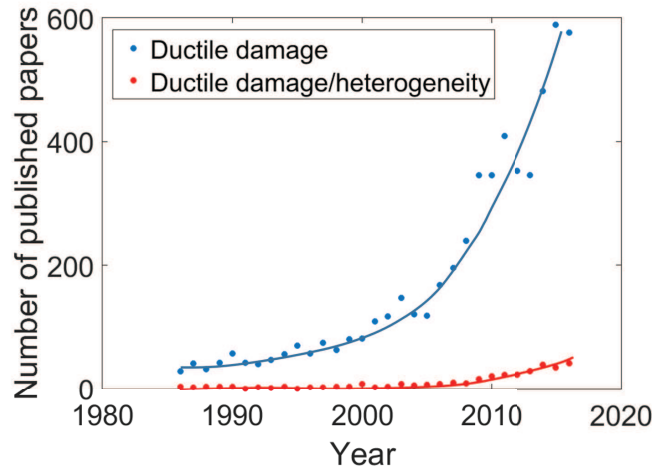


FIGURE 1: Evolution of the number of publications involving (1) ductile damage and (2) microstructural heterogeneity effects on ductile damage and related to materials science applications (according to Elsevier Scopus, www.scopus.com, consulted in July 2017).

The present work is dedicated to the 3D characterization, modelling and retroactive tailoring of the microstructural heterogeneity effects on damage evolution of 6xxx aluminium alloy. It is divided in the following way, as illustrated in Fig 2 :

- In **chapter 1**, the state of the art regarding the micromechanical modelling of ductile fracture is described. Furthermore, the microstructural features that are known to affect damage properties in age-hardenable aluminium alloys are reported. The last section is a short introduction on the tomography technique.
- The ductile failure of three 6xxx serie aluminium alloys has been characterized and modelled in **chapter 2**. The dependence of ductility on strength and on the microstructure characteristics is studied. The objectives of this chapter are to understand how

microstructure heterogeneities affect the damage evolution, how these complexify the application of existing models and to determine which characteristics of the microstructure have a first order role on the damage evolution. This chapter is based on a published paper [12].

- Local stirring using a friction stir processing (FSP) tool is shown, in **chapter 3**, to very significantly increase the fracture strain of the Al alloy 6056 while making it more damage isotropic. The objective of this chapter is to quantify and to correlate the microstructural refinement due to FSP with the ductilization of the Al alloy. The last section consists of a short feasibility study of the use of FSP in order to enhance the formability of thin metallic sheet. This chapter is based on a published paper [13], while the last section was not published.
- In **chapter 4**, a quantitative approach is proposed to relate the propensity of 6xxx aluminium alloys to fracture anisotropy to a simple microscopic parameter characterizing the degree of anisotropy in the spatial distribution of second phase particles. The objective is to understand the condition under which a cluster of coalesced voids starts to behave in a crack like manner instead of as a single large void.
- In **chapter 5**, the critical J-integral and essential work of fracture methods are applied to characterize the effect of sheet anisotropy and FSP on toughness properties. In situ synchrotron laminography is used to study the ductile damage micromechanisms in the immediate vicinity of the notch of CT-like specimens. The objective of this chapter is to determine if the ductilization of the Al 6056 by friction stir processing is associated with an improvement in fracture toughness.

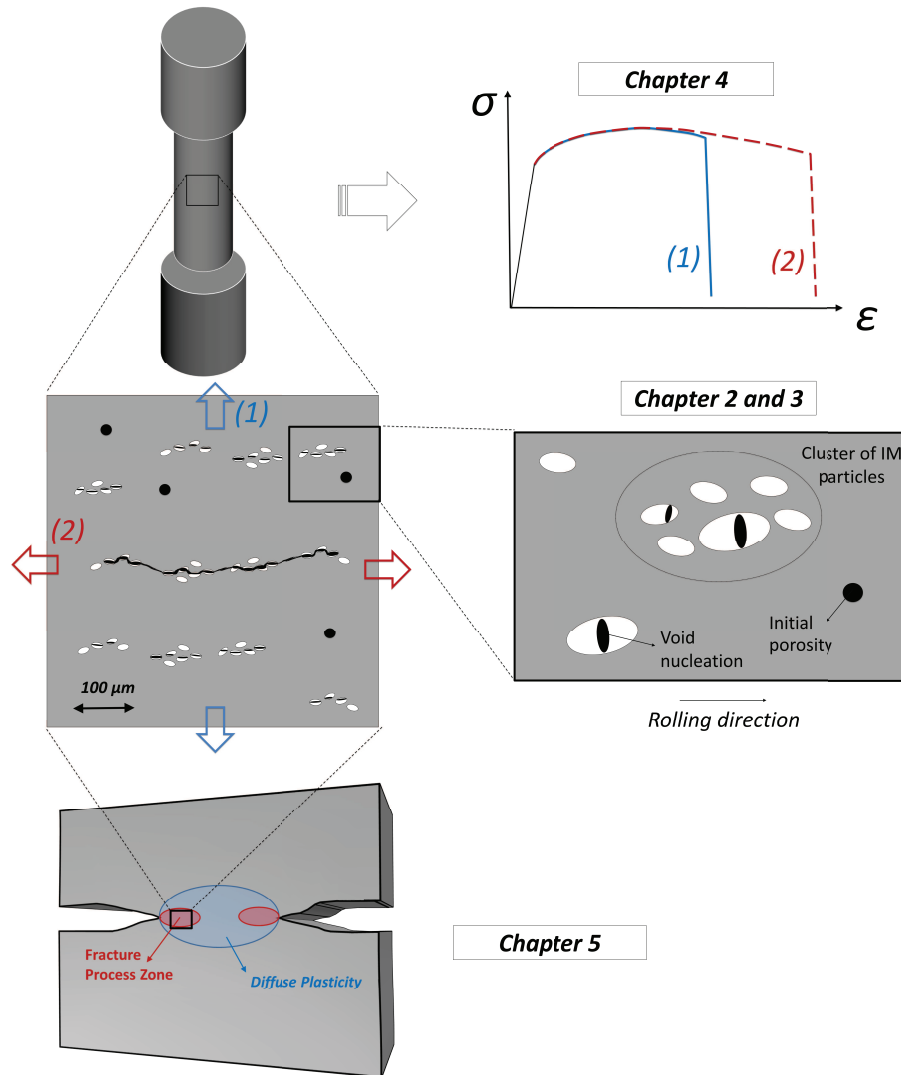


FIGURE 2: Structure of the thesis, as detailed in the text.

Chapter 1

Theoretical background

1.1 Damage and fracture

This literature review is organized as follows. First, the different mechanisms that are usually encompassed by the term "ductile fracture" are reviewed in order to position the scope of this work within the larger scheme. The multiple length scales at which ductile damage mechanisms operate are then defined and the major micromechanics-based models are presented for each elementary mechanism of ductile fracture, i.e. void nucleation, growth and coalescence.

1.1.1 Review of the different ductile failure mechanisms

The following section, which is mainly based on the recent paper by Tekoglu *et al.* [14], reviews the various possible failure mechanisms usually encompassed by the term "ductile fracture". This schematic categorization of the different failure mechanisms allows to position the scope of this thesis within the broad field of ductile fracture. However, it is only a theoretical view and there is no sharp transition between the different mechanisms.

1.1. Damage and fracture

According to Tekoglu *et al.* [14], the different ductile failure scenarios at room temperature can be classified into 5 different mechanisms (see Fig. 1.1(a) to (e)):

- a. **Failure without damage :** In the absence of any occurrence of damage, a ductile material fails in a purely-plastic way [15]. After yielding of the material, strain localizes inside a diffuse neck or in a shear band and the section reduces to a point of zero section. This mechanism is mostly observed in axial tension with superimposed pressure which can suppress other damage mechanisms. An example of this mechanism is shown in the photograph of a 7075-T4 aluminium alloy loaded in axial tension with a confining pressure [15].
- b. **Failure by localized plastic flow prior to damage :** In this mechanism, failure is associated with the "sudden" localization of plasticity into a shear band before the occurrence of damage. This localization can be due to different softening mechanisms, such as the Portevin–Le-Chatelier effect in Al-alloys [16]. At later stages, voids nucleate, grow and coalesce due to the large plastic strain in the plane of the band until final separation. In this mechanism, the energy associated with the damage percolation is weak compared to the overall energy absorption [16]. This mechanism has recently been observed, using 3D imaging technique, at the crack tip of thin Al alloy 2198 sheet. The clear strain localization observed in the 3D strain field (obtained by digital volume correlation) occurs before damage initiation and then a slanted crack appears in this shear band.
- c. **Failure by localization induced by the accumulated porosity :** In this mechanism, voids appear prior to macroscopic localization and reduce the load-bearing area so much that flow localizes in a narrow band. In other words, the macroscopic localization is driven by the softening due to the growth of the voids. The width of this localization band is expected to be of the order of one or more inter-void spacing [5].
- d. **Onset of coalescence dictates the onset of macroscopic localization :** In this case, the macroscopic localization is the consequence of void coalescence. After the onset of void coalescence,

the plastic deformation localizes in a narrow band limited to the ligaments between many voids. An example of this mechanism is shown in Fig. 1.1(d). Fig. 1.1(d) shows a sequence of tomographic reconstructions showing deformation for a titanium sample containing one array of laser drilled holes.

- e. **Cluster localization :** This mechanism is similar to the previous mechanism **d**, but in this case, coalescence occurs over a limited number of neighbouring voids. These local coalescence events occur in a region involving a few closely spaced voids inside a cluster, and are therefore referred to as cluster localizations. Subsequently, a second void growth process occurs based on these early coalesced microcracks and the final rupture is then triggered by a second coalescence stage between microcracks. An example of this mechanism is shown in Fig. 1.1(e)[17]. The micrograph shows the fracture path after void linkage in Al-plate sample (100 μm thickness) containing a random array of laser drilled holes (10 μm diameter).

Limited attention has been given to the last "cluster localization" mechanism, even though observations in a variety of industrial metallic alloys suggest that such behaviour is important [19, 14]. The role of these clusters of particles (or voids) on damage percolation and ductile tearing is at the core of this thesis.

1.1. Damage and fracture

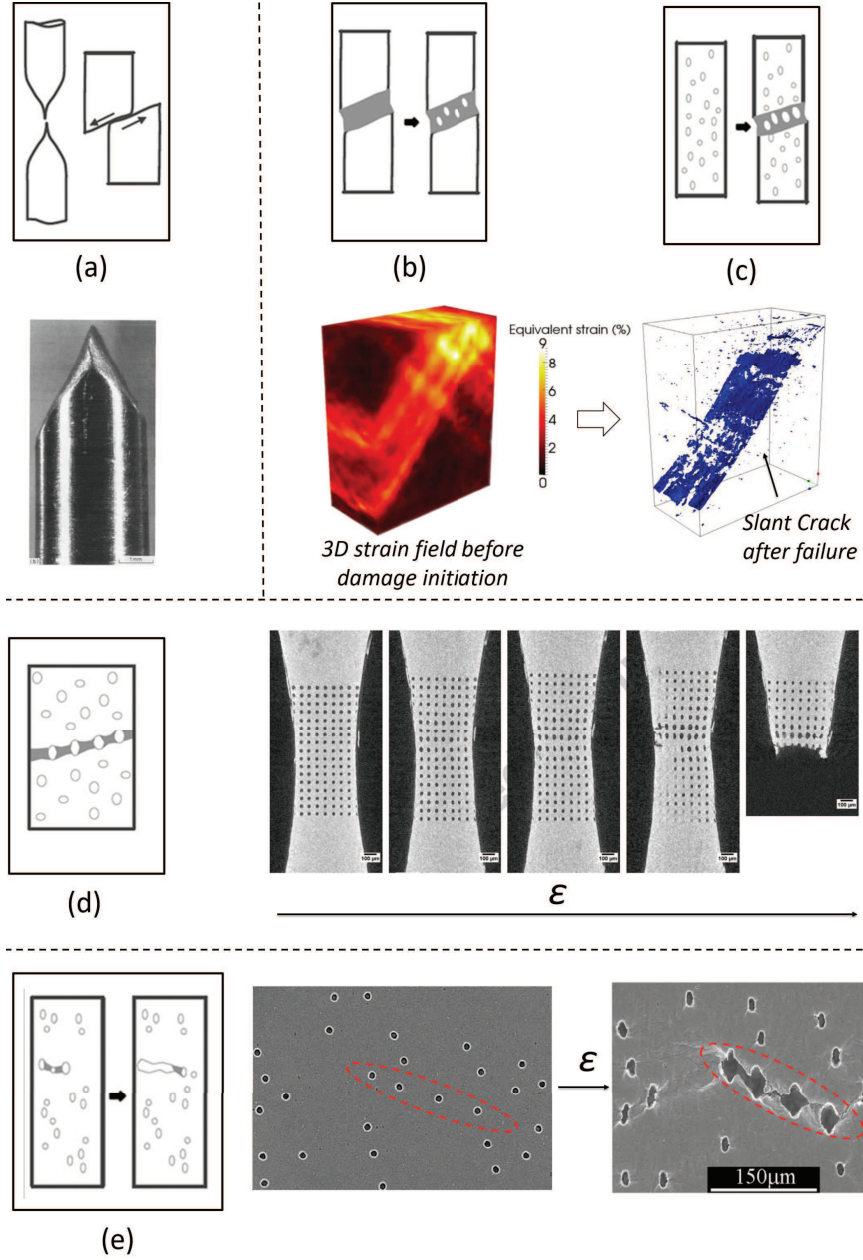


FIGURE 1.1: The five generic scenarios of ductile fracture in single phase polycrystals at temperatures far below melting point temperature. Schematics adapted from [14]. Experimental figures (a), (b), (d), (e) are reproduce from Refs. [15],[16],[18],[17], respectively. More details are given in the text.

1.1.2 Physical length scales entering ductile damage process

This section introduces the different length scales typically associated with the damage process within an engineering metallic alloy. A more detailed description of the physical lengths entering a generic ductile process can be found in Ref.[7].

Several physical lengths play a role in ductile fracture, as schematically represented in Fig. 1.2:

- [1] The largest length scale involved in the failure process is the size of the plastic localization band. If this band appears as a result of damage induced softening (i.e. mechanism (c) of Fig. 1.1), the localization band width directly scales with the primary void spacing [20, 7], see length scale [3]. In some cases, there is no clear link between the voids and the localization band (i.e. mechanism (b) of Fig. 1.1). In this case, very concentrated strained bands may become inactive (i.e. narrowing of the band) and other bands become active at different stages of loading.
- [2] In real metallic materials, an "intermediate" length scale that plays a major role in the failure process is directly linked with the microstructure. This microstructure based length scale is due to an inhomogeneous spatial distribution of the microstructural features that enter the damage process. For example, large and oriented stringer-like clusters are commonly observed in aluminium alloys [21]. It particularly affects the void coalescence step due to void interactions occurring inside a cluster of particles.
- [3] The size of the primary particles, typically larger than $1\mu\text{m}$, has a direct effect on the nucleation process. Several experimental studies have previously indicated an increase of void nucleation by particle cracking with increasing particle size, which can be related to a size distribution of internal defects [11]. The ratio of the void size to the void spacing, i.e. the relative spacing between voids, is the primary parameter controlling the onset of void coalescence [7].
- [4] The distribution of secondary particles, typically of submicron size, can have a direct effect on the ductility. For instance, small

1.1. Damage and fracture

voids in the neighbourhood of large cavities grow much faster than in a single size population [22, 23].

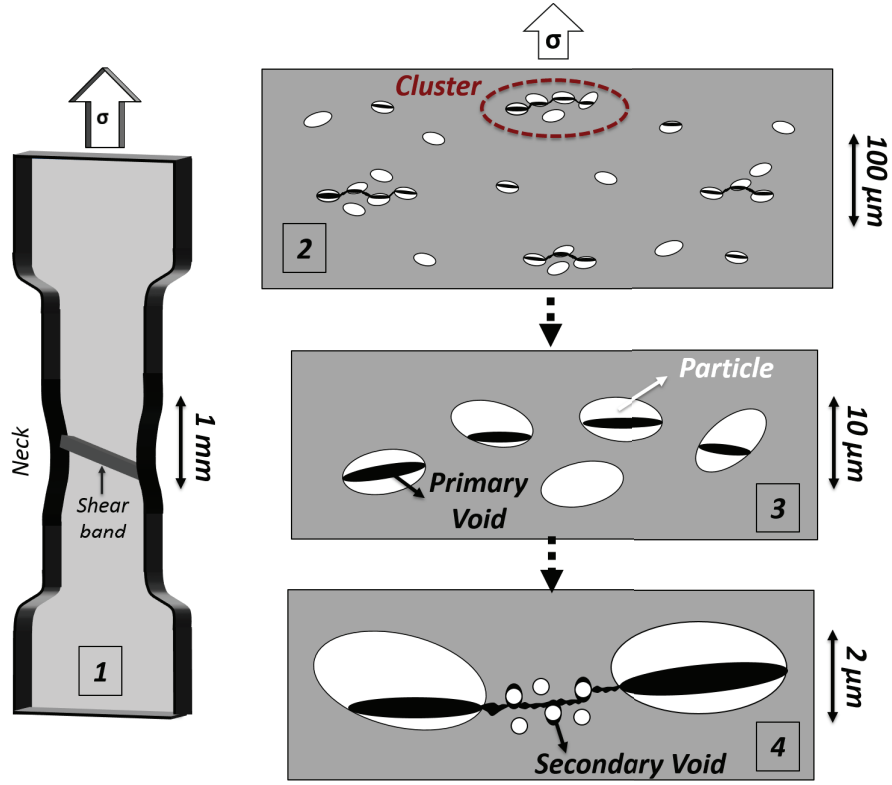


FIGURE 1.2: Schematic description of the microstructural features and length scales entering a generic damage process.

1.1.3 Nucleation, growth and coalescence of voids

As already explained, ductile fracture is defined as the physical process that leads to the formation of cracks by plasticity-controlled void nucleation, growth, and coalescence of voids [24]. In the following section, classical micromechanical models for the three stages of ductile damage are presented with focus placed on models pertinent to this thesis. Many other models have been proposed and enhanced over the years, as covered in several recent review papers [25, 7, 24, 26, 27].

1.1.3.1 Void nucleation

Void nucleation criterion

Generally speaking, void nucleation model can be divided into three categories; (i) critical stress criteria, (ii) strain criteria and (iii) energy criteria.

One of the first criterion for void nucleation by interface decohesion has been proposed by Ashby [28]. In this model, the normal stress across the particle-matrix interface increases with plastic strain due to the accumulation of dislocations and void nucleation occurs when the stress at the interface exceeds a critical stress.

Later, Tanaka *et al.* [29] derived an energy based void initiation criterion, with a particular interest in very small particles. Indeed, the elastic strain energy stored in very small particles could be insufficient for the creation of internal interfaces. This analysis showed that above a particle size of about 25 nm, interfacial cracks are energetically favourable. Void nucleation can thus be modelled with a stress criterion for larger particles. On the other hand, when a particle size is smaller than this critical size, the attainment of a critical strain by the energy criterion is the necessary and sufficient condition for void nucleation. This critical size was initially estimated at about 25 nm but is more likely to be above 100 nm [25].

There has been a long debate as to whether or not there exists a critical strain to nucleate cavities, i.e. if a strain criterion is applicable

1.1. Damage and fracture

or not. It is important to realise that a critical stress inside or at the interface of the particle is produced by the work-hardening of the matrix around the particle. This local work hardening is a function of the plastic strain in the material, so that a strain-based criterion refers fundamentally to a local critical stress condition [30]. Needleman [31] compared a stress-controlled criterion with a strain-controlled nucleation and observed quite different predictions for the onset of plastic localization. This is due to the hydrostatic tension dependence of the stress-controlled criterion which induces early flow localization. In practice, strain-controlled void nucleation laws are frequently preferred because they are more simple to implement numerically [24]. However, strain-controlled criteria do not capture the dependence of void nucleation upon stress triaxiality [25].

Argon *et al.* [32] proposed a stress based criterion for void nucleation by interface decohesion:

$$\sigma_{inh} + \sigma_m = \sigma_c \quad (1.1)$$

where σ_{inh} is the interfacial tensile stress from the strain inhomogeneity effect, σ_m is the hydrostatic stress, and σ_c is the critical stress to reach for void nucleation. Argon [32] developed approximate expressions for σ_{inh} , expressed as $\sigma_{inh} = k_A \sigma_{eq}$, where σ_{eq} is the equivalent Von Mises stress and k_A is a geometrical factor given for a limited number of idealized cases (slender rods, platelets and round particles). In the case of a very slender rod, the axial stress inside the inclusion increases slightly from the ends toward the middle by no more than a few percent [32]. The value for the interfacial tensile stress used in eq. (1.1) can thus also be taken as the principal stress inside the particle. This assumption will be used in order to compare the expressions for σ_{inh} proposed by Argon to the Beremin model (see next paragraphs).

The expression proposed by Argon for σ_{inh} considers no interaction between particles and holds only when the volume fraction of particle is small. When the volume fraction of particle is large, the spacing between particles becomes of the order of their diameter and the particles start to interact. Argon *et al.* [33] have considered the effect of particle interaction in the case of rigid spherical particles within a simple power-law hardening matrix. The method was initially proposed by Ashby [28] and considers that the displacement incompatibility at the

matrix/particle interface is dissipated by spreading the incompatibility within a "secondary" plastic zone, i.e. an additional contribution to the distant macroscopic stress field. The result introduces a new parameter in the expression of σ_{inh} :

$$\sigma_{inh} = k_A \sigma_{eq} + C_1 \frac{L_p}{R_p} + C_2 \left(\frac{R_p}{L_p} * \varepsilon_{eq} \right)^n \quad (1.2)$$

where L_p is the distance between particles, R_p is the particle radius, C_1 and C_2 are numerical coefficients, n is the hardening coefficient and ε_{eq} is the macroscopic plastic strain. By assuming that particles are non-interacting when the secondary plastic accommodation zones do not touch each other, Argon showed that particles act in the matrix as if they were totally isolated for volume fraction lower than about 1%. However, this might not be true for heterogeneous distribution of particles. Indeed, a cluster of particles implies a larger value of the local particle volume fraction.

The Beremin model [34] is based on the decomposition of the stress in the particle as the sum of the stress in the matrix and of an additional stress transfer arising from the deformation mismatch between the particle and the matrix¹. Particles are treated as brittle solids and the fracture of the particle is assumed to take place when the maximum principal stress in the particle reaches a critical value :

$$\left(\sigma_{\text{princ}}^{\text{max}} \right)^{\text{part}} = \sigma_{\text{crit}}^{\text{part}} \quad \text{or} \quad \left(\sigma_{\text{princ}}^{\text{max}} \right)^{\text{int}} = \sigma_{\text{crit}}^{\text{int}} \quad (1.3)$$

Using Eshelby's theory [35] and the secant elasto-plastic modulus extension proposed by Berveiller and Zaoui [36] for the deforming matrix, the maximum principal stress in the particle is given by :

$$\left(\sigma_{\text{princ}}^{\text{max}} \right)^{\text{part}} = \Sigma_{\text{princ}}^{\text{max}} + k_b E_p \varepsilon_{eq} \quad (1.4)$$

where $\Sigma_{\text{princ}}^{\text{max}}$ is the maximum principal stress in the matrix, k_b is a stress concentration factor which depends on the particle shape and orientation and E_p is the secant modulus of the matrix. In order to derive

¹F.M. Beremin is the name of a French research group founded in the early 1980s including researchers from Ecole des Mines (Paris), Framatome (Saint-Marcel) and BCCN (Dijon).

1.1. Damage and fracture

this expression, it has been assumed that E_p is much smaller than the Young's modulus of the particle and the elastic strain in the particle has been neglected. The secant modulus $E_p = \sigma_{eq} / \varepsilon_{eq}$ and eq. (1.4) becomes :

$$\left(\sigma_{\text{princ}}^{\text{max}}\right)^{\text{part}} = \Sigma_{\text{princ}}^{\text{max}} + k_b \sigma_{eq} \quad (1.5)$$

For axisymmetric loadings, $\Sigma_{\text{princ}}^{\text{max}} = \sigma_m + \frac{2}{3}\sigma_{eq}$ where σ_m is the mean stress and this gives :

$$\left(\sigma_{\text{princ}}^{\text{max}}\right)^{\text{part}} = \sigma_m + \left(\frac{2}{3} + k_b\right)\sigma_{eq} = \left(T + \frac{2}{3}\right)\sigma_{eq} + k_b\sigma_{eq} \quad (1.6)$$

where T is the stress triaxiality factor ($T = \sigma_m / \sigma_{eq}$). In this form, eq. (1.6) is very similar to that already derived by Argon, see eq. (1.1).

The factor k_b appearing in eq. (1.6) depends on the shape of the ellipsoidal particle through the Eshelby's tensor $\underline{\underline{S}}$ [37]. In the seminal work by Beremin, the Eshelby's tensor has been computed using Osborn's demagnetizing factors [38]. Fig. 1.3 shows the evolution of the geometrical factor k_b in the case of axisymmetric ellipsoidal particles. The aspect ratio W_p is defined as the particle length along the loading direction divided by the diameter of the central cross-section. For a perfect sphere, k_b is equal to 1 and is obviously independent of the loading direction. In the case of a prolate particle ($W_p \gg 1$), the factor k_b increases with increasing W_p . This is due to the fact that as the particle becomes more and more elongated, the state in the particle tends toward isostrain. Note that the value of k_b also increases for W_p smaller than 2 and does not tend to 1 as W_p tends to 1 (spheroidal case). In order to understand this unexpected behaviour, the evolution of k_b has been calculated with the Eshelby's tensor factors found in a more recent paper [39]. It gives another expression for the factor k_b , written k_b' . The evolution of k_b' with W_p is shown in Fig. 1.3. While the general trend are in good agreement, the value of k_b' is higher than k_b for a same W_p . In order to check which expression is better, a few cell computations have been performed². Cells simulations allows to compute the factor k_C for which eq. (1.6) would give the exact value of $\left(\sigma_{\text{princ}}^{\text{max}}\right)^{\text{part}}$. The

²Cell simulations have been performed with the help of Ir. J. Chevalier, PhD student in the Imap laboratory

matrix is modelled as a linear strain hardening material ³ because the definition of the plastic modulus E_p used in eq. (1.5) has a clear meaning in this case. Fig. 1.3 shows that k_b' is a good approximation of the cell computation values k_C . Furthermore, elongated particle are quite similar to the elongated slender rod case for which Argon developed an analytical expression (k_A). It can thus be directly compared to k_b (see eq.(1.1), with $k_A = L/R$ for a slender round rod of length $2L$ and radius R).

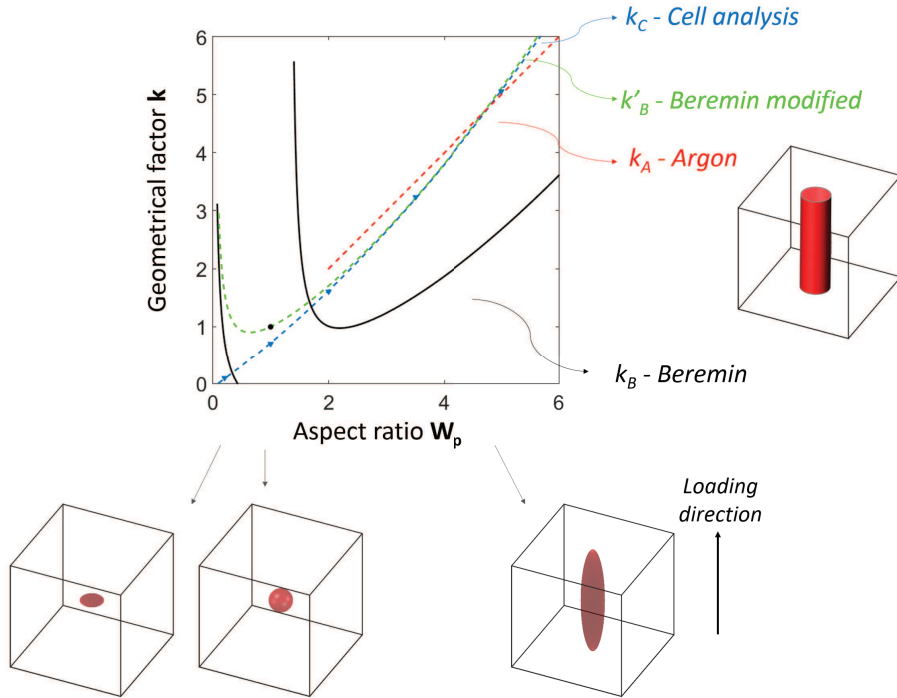


FIGURE 1.3: Effect of the particle shape on the different void nucleation criteria. For k_C , the points correspond to unit-cell computation results and the dashed line has been fitted ($k_C = 0.061W_p^2 + 0.72W_p - 0.067$).

In the seminal paper of the Beremin group [34], a strong temperature dependence of σ_{crit}^{part} was noted when comparing eq. (1.4) with experimental results obtained at different temperatures. Thus, it was proposed to use the tangent modulus $E_t = ((\sigma_{eq} - \sigma_0) / \epsilon_{eq})$ instead of

³ $\sigma_{eq} = 250 + 800\epsilon_{eq}$ [MPa], which correspond roughly to a 6xxx Al alloy in the T4 state.

1.1. Damage and fracture

the secant modulus $E_p = \sigma_{eq}/\varepsilon_{eq}$ since the strain inhomogeneity effect should not include the yield stress σ_0 . Fig. 1.4(b) compares the factor k_C obtained by cell computations for two different elasto-plastic behaviours (see Fig. 1.4(a).) In both cases, a similar hardening behaviour has been used (similar E_t) but with different yield stress (σ_0). Fig. 1.4(b) shows that the evolution of k_C is almost independent of the matrix yield stress when the secant modulus E_p is used. On the other hand, the evolution of k_C is quite different with the use of the tangent modulus E_t . In other words, it seems that the secant modulus gives better results despite what was concluded in [34].

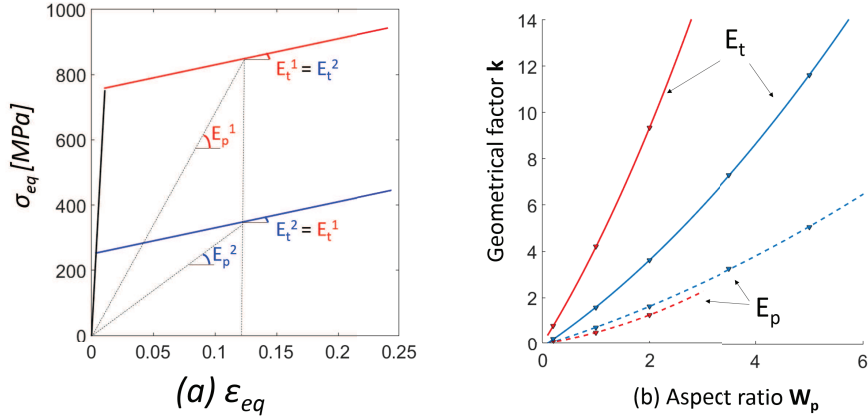


FIGURE 1.4: Fig.(b) shows the evolution of the stress concentration factor k_C . The elasto-plastic properties which have been used for the matrix are shown in Fig.(a)

Since the interaction law given in eq. (1.4) is based on Eshelby's theory, the stress field is considered as homogeneous within the particle and at the interface with the matrix. This implies that it is only possible to distinguish particle cracking and interface decohesion by the use of two different critical stresses for the two void nucleation mechanisms. However, the stress and strain fields inside the elastic particle are not uniform for a matrix undergoing plastic deformation [24] and it is not possible to explain the dependence of void nucleation upon particle orientation and stress triaxiality. Indeed, Lassance *et al.* [40] characterized the damage process of two 6xxx series Al alloys and observed that the dominant mode of nucleation is dependant on the particle configuration. Particle fracture is observed for particles elongated along

the main loading direction. Interface decohesion is observed when the loading direction is perpendicular to the long dimension of the particle. This effect of particle orientation has been addressed by finite element (FE) simulations on complex multiparticle cells [41]. Fig. 1.5 shows the principal stress distribution inside and at the interface of a particle, normalised by their respective fracture criteria, just before nucleation. High interface stresses are observed on a large part of the interface in the perpendicular configuration compared to the aligned configuration and interface decohesion mechanism is favoured.

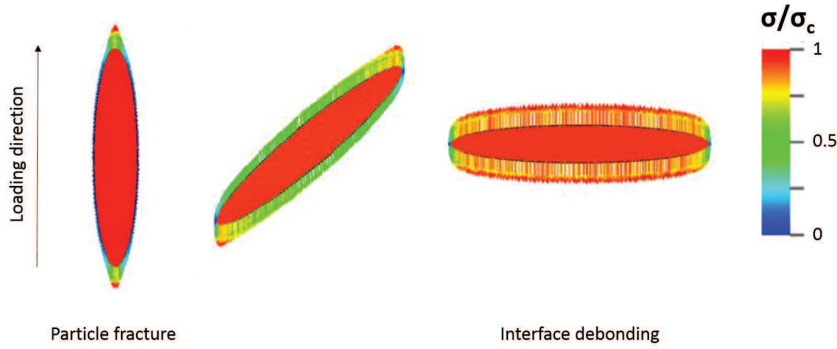


FIGURE 1.5: Principal stress distribution inside and at the interface of a particle, normalised by their respective fracture criteria, just before nucleation. Adapted from [41].

Lee and Mear [42] used computational cell simulations to compute the stress concentration arising inside and at the interface of an ellipsoidal elastic inclusion embedded in elasto-plastic strain-hardening matrix subjected to axisymmetric loadings. The effect of stress triaxiality on the void nucleation mechanism can be rationalized in terms of stress concentration factors inside the particle (κ_P) and at the interface (κ_{Int}):

$$\kappa_P = \frac{\sigma_{princ}^{max,Part}}{\Sigma_{princ}^{max}} \quad \text{or} \quad \kappa_{Int} = \frac{\sigma_{princ}^{max,Int}}{\Sigma_{princ}^{max}} \quad (1.7)$$

where Σ_{princ}^{max} is the remote axial stress, $\sigma_{princ}^{max,Int}$ is the maximum normal stress along the matrix-particle interface and $\sigma_{princ}^{max,Part}$ is the maximum principal stress in the particle volume. The two stress concentration factors κ_P and κ_{Int} depend on different factors, such as the rigidity mismatch, the flow properties of the matrix, the particle aspect ratio and

1.1. Damage and fracture

the stress triaxiality. The analysis showed that both stress concentration factors κ_P and κ_{Int} are reduced by an increase in remote triaxiality. Furthermore, the ratio κ_P/κ_{Int} decreases as the remote triaxiality is increased, but only significantly for elongated particle. These results indicate that void nucleation will have a greater tendency to occur by interfacial debonding than by particle fracture as the triaxiality is increased.

The stress concentration factors κ_P obtained by Lee and Mear analysis can be compared with the Beremin analysis⁴. However, in the Lee and Mear analysis, the stress concentration factor κ_P is a function of remote axial strain and closed form expressions are not given. Values have thus been extracted from graphical data and as the large strain asymptotic values⁵. Fig. 1.6 compare the stress concentration factor κ_P (points) with the stress concentration factor k_b' (surface) as a function of the particle shape and the stress triaxiality. It is obvious that there is a really good agreement between the two analyses.

Since both stress concentration factors κ_P and κ_{Int} are reduced by an increase in remote triaxiality, Lee and Mear proposed based on their theoretical work that the onset of void nucleation would be delayed with respect to remote strain if the triaxiality was increased. This conclusion is in contradiction with other experimental results [34, 43, 31]. In fact, close inspection of the results of Lee and Mear reveals the opposite. Indeed, since the matrix is subjected to axisymmetric loadings, eq. (1.7) becomes :

$$\sigma_{princ}^{max,Part} = \kappa_P \left(T + \frac{2}{3} \right) \sigma_{eq} \quad (1.8)$$

In eq. (1.8), κ_P decreases with T but the second term increases with T . Globally, $\sigma_{princ}^{max,Part}$ increases with T , for a same remote strain (or same σ_{eq}), and nucleation will take place more easily for the highest stress triaxiality.

Based on a micromechanical analysis of void nucleation from a rigid particle in an elastic-viscoplastic matrix, Needleman [31] suggested a modification of the Argon criterion. The nucleation stress is,

⁴The modified k_b' is used instead of k_b

⁵Note that κ_P does not always exhibit a perfect asymptotic behaviour. In this case, κ_P is taken at the largest given remote strain.

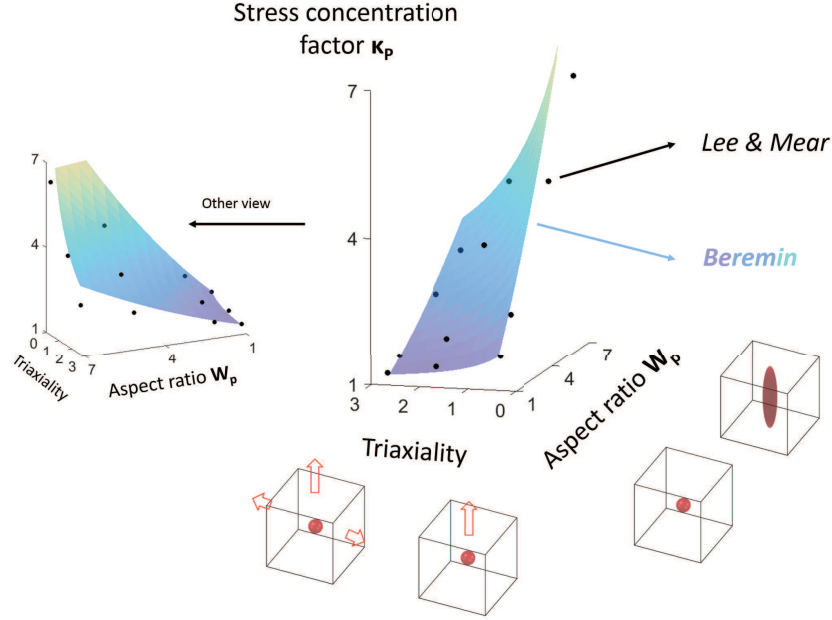


FIGURE 1.6: Comparison between the stress concentration factor obtained by the Beremin model (k_b') and obtained by the Lee and Mear analysis (κ_p).

similarly to the Beremin and the Argon criteria, a linear combination of the effective stress σ_{eq} and the hydrostatic tension σ_m , with the proportion of each term being an adjustable parameter c :

$$\sigma_{eq} + c\sigma_m = \sigma_c \quad (1.9)$$

This criterion was first shown to provide a good characterization of void nucleation by interface decohesion [31] for the adjustable parameter c equal to 0.4. Later, Shabrov and Needleman [43] have shown that this criterion is also suitable for the case of particle cracking, but in this case the optimal value of the parameter c was found to be 0.6. The value of c smaller than one indicates that only some fraction of the far-field hydrostatic tension is effective in increasing the stress acting across the interface or within the particle. The value of c depends on the particle volume fraction, particle shape, and the material properties [43]. In their conclusion, Shabrov and Needleman [43] raised the question of whether there is any physical basis for the optimal value of the parameter c or whether it is only a data fitting parameter. In order to

1.1. Damage and fracture

discuss this question, it is interesting to rewrite eq. (1.6) as :

$$\frac{\left(\sigma_{\text{princ}}^{\text{max}}\right)^{\text{part}}}{\frac{2}{3} + k_b} = \frac{1}{\frac{2}{3} + k_b} \sigma_m + \sigma_{eq} \quad (1.10)$$

It is now explicit that c should depend on the shape of the particle (through k_b) and be equal to $\frac{1}{\frac{2}{3} + k_b}$. In their experimental work, Shabrov and Needleman used a Ti-modified 4330 steel and observed cracking of cuboidal titanium nitrides (TiN inclusions). Even if these inclusions are cuboidal, they do not have a longer dimension oriented along a preferred orientation. It is thus a reasonable assumption to treat them as spheroidal ($W_p=1$) and the stress concentration factor k_b is thus equal to 1 (see Fig. 1.3). For $k_b = 1$, the expression for $c = \frac{1}{\frac{2}{3} + k_b}$ takes the value 0.6, i.e. the optimal value found by Shabrov and Needleman, and this would tend to suggest that the value of c has a physical meaning.

In conclusion, this short review of void nucleation criteria clearly shows that there is room for improving simple models, such as the Beremin approach. Nevertheless, this simple approach provides an adequate description of the void nucleation condition for a variety of particles of any ellipsoidal shape and for different loading directions. Indeed, even if some work has been accomplished, e.g. the analysis by Lee and Mear [42], results are usually not translated into simple expressions which can be easily used. Furthermore, the complexity of the nucleation process at that scale is such that critical stress σ_c have to be identified from experiments [24]. This best calibrated value of σ_c does not directly represent the fundamental interface cohesive strength or ideal particle strength, but rather some effective measures of strength [25] incorporating all the approximations inherent with the nucleation models. The next paragraph discuss a little more the calibration of σ_c .

Effects of particle size and distribution on void nucleation

The onset of void nucleation by inclusion cracking depends on the inclusion size, with larger inclusions cracking at lower stresses [43]. This is explained by the fact that larger particles have a greater probability of containing a critical volume defect [44].

Furthermore, the critical stress for nucleation must be distributed in order to represent the experimentally observed nucleation kinetics [45]. This heterogeneity can be due to a statistical distribution of the critical stress for nucleation due to a distribution of particle size [46], local microplasticity effects related to microstructure details and local crystallography [47] or clustering of the second phase particles [48]. The heterogeneity of the nucleation process has been introduced through the application of Weibull statistics to describe the fracture process :

$$P(\sigma_{part}^{max}, V_{part}) = 1 - \exp \left(- \frac{V_{part}}{V_0} \left(\frac{\sigma_{princ}^{max, Part}}{\sigma_w} \right)^m \right) \quad (1.11)$$

where σ_w and m are the Weibull stress and modulus, V_0 is the volume of particle for which the probability of survival is 37% at σ_w . For σ_w and V_0 fixed, void nucleation is confined in a narrower strength range when m increases. In principle, a single, material dependent, set of Weibull parameters (m, σ_w, V_0) should describe the probability of void nucleation within the material at all levels of straining [46].

Using FEM results to compute the particle stress within a 2618-T4 Al alloy reinforced with SiC particles, Maire *et al.* [49] found the following set of Weibull parameters: $m = 3.8$, $\sigma_w = 1700$ MPa and $V_0 = 125 \mu m^3$. Lewis and Withers [46] have studied particle cracking within a particulate $ZrO_2/2618$ Al alloy metal matrix composite. Using an Eshelby style analysis including a relaxed plastic strain component, they found that the best set of Weibull parameters was ($m=7$, $\sigma_w=1550$ MPa, $V_0=1780 \mu m^3$). Babout *et al.* have studied particle cracking within a "model" composites (Al matrix-oxides spherical particles) using 3-D tomography and FE cell calculations to estimate the local stress inside the particle [50]. Since the particle size distribution was relatively narrow, they found void nucleation to be well described by a simpler Weibull statistic without size effect (i.e. $V_{part} = V_0$), with $m=7$ and $\sigma_w=1550$ MPa.

It is important to note that the Weibull parameters (m, σ_w, V_0) de-

1.1. Damage and fracture

pend strongly on the choice of micromechanical model used to compute the stress inside the particle [51]. Fig. 1.7 shows all the calibrated values for σ_c which were found in the literature, for studies in which particle fracture is assumed to occur when the maximum principal stress in an elastic inclusion reach a critical value. In the few cases for which a distribution of σ_c was used, the averaged value is plotted. Furthermore, the value of σ_c was not always explicitly given. The values of σ_c is given in function of the average particle size. A small arrow on the point indicate that the value was calibrated for the first nucleation event, but is not representative of the average value of the distribution of σ_c . Note that there are many informations missing, such as the particle size distribution, the particle chemistry and the flow properties of the matrix. Nevertheless, it is clear that there is a general trend, and that the average value of σ_c decreases if the particle size is increased.

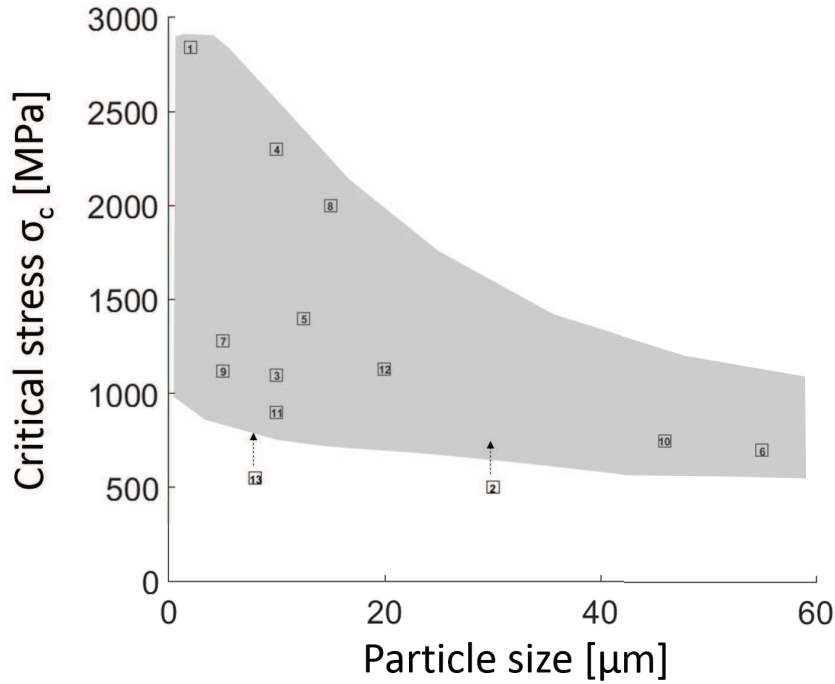


FIGURE 1.7: Review of the critical stress found in the literature for void nucleation by particle fracture. Points 1 to 13 come, respectively, from Refs.[52, 53, 34, 43, 54, 50, 55, 46, 56, 57, 58, 59, 60]

There have been a limited number of micromechanical studies on the effects of particle distribution on void nucleation [61]. The effect of the particle spatial distribution on void nucleation by interfacial debonding was studied by Shabrov and Needleman [62]. A series of particle distributions, with fixed inclusion volume fraction, were subjected to plane strain tension with a superposed tensile biaxial stress (characterized by the stress ratio $\rho = \Sigma_1/\Sigma_2$, see Fig. 1.8). A few selected results are shown in Fig. 1.8. The void nucleation strain is identified with the stress drop occurring at the initiation of debonding. For low values of stress triaxiality (low ρ), void nucleation occurs after extensive plastic straining. Regular distributions (Fig. 1.8 (a) and (b)) have a higher value of the void nucleation strain compared to random distributions (Fig. 1.8 (c) and (d)). For larger values of stress triaxiality, void nucleation occurs at relatively small plastic strains, the effect of particle distribution becomes less important and the effect of particle size becomes more important.

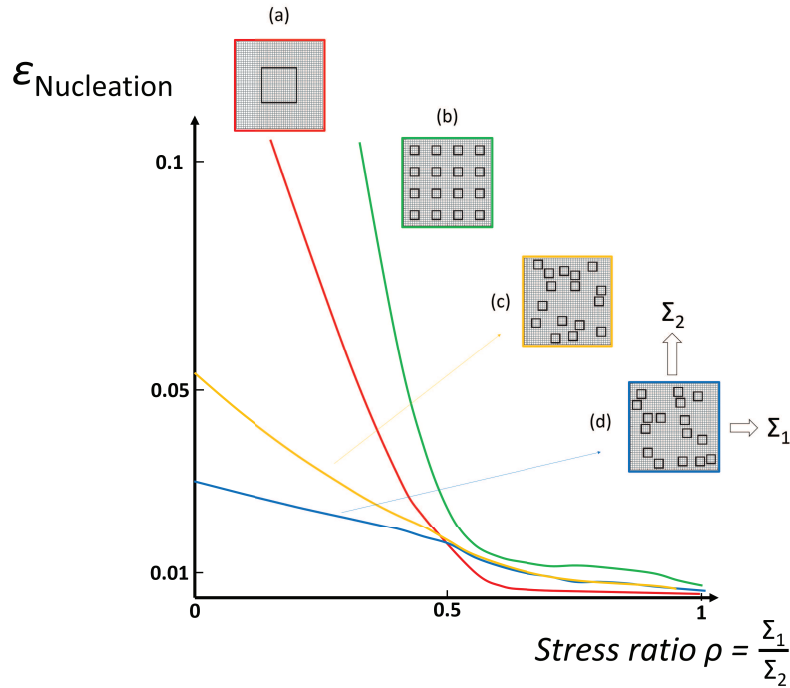


FIGURE 1.8: Effect of the particle spatial distribution on void nucleation by interfacial debonding. Adapted from [62].

1.1. Damage and fracture

Another study of the effect of particle clustering on void nucleation by particle fracture has been performed more recently by Abedini *et al.* [63]. A few particle distributions with different cluster geometries have been studied in uniaxial tension with multi-particle unit cells and for a fixed particle volume fraction (1%). Fig. 1.9(b) shows the evolution of the maximum principal stress in the central particle for the linear cluster geometry shown in Fig. 1.9(a). The severity of the clustering is quantified with the ratio of the inter-cluster spacing D over the particle spacing within cluster d . It is clear that the maximum principal stress in the particle increases as the degree of clustering is increased (i.e. when D/d is increased). In other words, void nucleation by particle fracture is more likely to occur inside clusters which lead to local stress concentration.

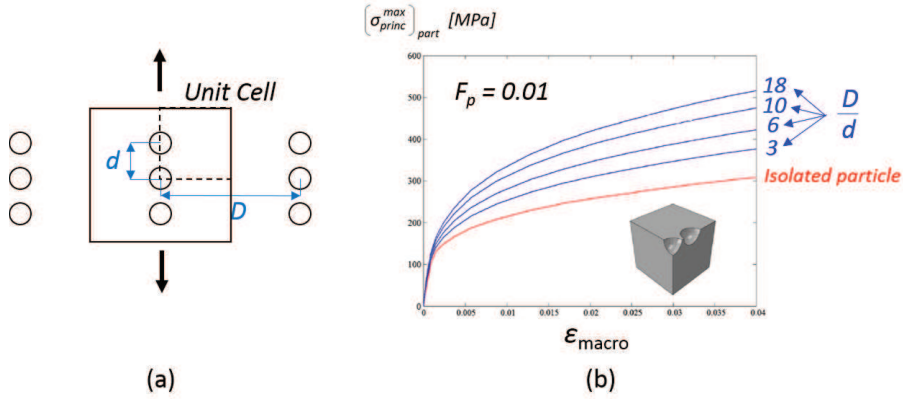


FIGURE 1.9: Effect of the particle spatial distribution on void nucleation by particle fracture. Adapted from [63]

1.1.3.2 Void growth

Micromechanical-based models for describing void growth can be divided into two groups, namely uncoupled and coupled models.

The Rice and Tracey (R&T) void growth model [64] :

The original version was developed for a spherical void in an infinite, rigid perfectly plastic material under uniform remote strain ϵ_{ij} and stress σ_{ij} . The volume change is a function of the hydrostatic stress

only while the change of shape is dictated by the deviatoric stress component. The strain rate field is expressed in terms of the principal components $\dot{\epsilon}_1 \geq \dot{\epsilon}_2 \geq \dot{\epsilon}_3$. A version of the model taking into account the aspect ratio evolution of an ellipsoidal void, with the main axes aligned with the principal loading directions $(\Sigma_1, \Sigma_2, \Sigma_3)$, has been elaborated by Thomason [65]. The rate of change of the three radii in the three directions of space ($R_i, i=1,2,3$) were shown to have the form :

$$\dot{R}_k = \left((1 + E)\dot{\epsilon}_k + \sqrt{\frac{2}{3}}\dot{\epsilon}_I\dot{\epsilon}_I D \right) R_{mean} \quad (1.12)$$

where $R_{mean} = \frac{R_1 + R_2 + R_3}{3}$ is used because the void is not spherical.

The dilatational factor D is given by $D = 0.558 \sinh\left(\frac{3}{2}T\right) + 0.008 \nu \cosh\left(\frac{3}{2}T\right)$ for non-hardening materials and $D = 0.75T$ for linear hardening materials. The Lode variable is defined as $\nu = \frac{-3\epsilon_2}{\epsilon_1 - \epsilon_3}$. For axisymmetric loadings, the Lode variable ν is equal to 1 and the dilatational factor for non-hardening materials becomes :

$$D = \alpha \exp\left(\frac{3}{2}T\right) \quad (1.13)$$

The pre-exponential parameter α , equal to 0.283 in the original work of R&T⁶, was later re-evaluated by Huang [66] using a more accurate numerical method :

$$\alpha = \begin{cases} 0.427 T^{1/4} & \text{for } T \leq 1, \\ 0.427 T & \text{for } T > 1. \end{cases} \quad (1.14)$$

Furthermore, the incompressible extension factor $(1 + E)$ was initially equal to 5/3 in the original work of R&T. Later, Worswick and Pick [67] proposed a more elaborated expression based on FE void cell calculations. For instance, for $f_0=0.01$, $n=0.2$ and for low stress triaxialities (<1.2), the result of Worswick and Pick leads to (see [68]) :

$$1 + E = \frac{(1.91 - 0.725T) \exp\left(\frac{3}{2}\epsilon_{eq}\right)}{2 \exp\left(\frac{3}{2}\epsilon_{eq}\right) - 1} \quad (1.15)$$

⁶ $a*\sinh(x) + b*\cosh(x) \approx \frac{a+b}{2} \exp(x)$

1.1. Damage and fracture

Using the incompressibility equation ($\dot{\varepsilon}_1 + \dot{\varepsilon}_2 + \dot{\varepsilon}_3 = 0$) and the definition of the Lode variable, integration of eq. (1.12) gives the following general expressions for the three principal radii of an ellipsoidal void :

$$R_1 = \left(A + \frac{3+\nu}{2\sqrt{\nu^2+3}} B \right) R_0 \quad (1.16)$$

$$R_2 = \left(A - \frac{\nu}{\sqrt{\nu^2+3}} B \right) R_0 \quad (1.17)$$

$$R_3 = \left(A + \frac{\nu-3}{2\sqrt{\nu^2+3}} B \right) R_0 \quad (1.18)$$

where $A = \left(\frac{2\sqrt{\nu^2+3}}{3+\nu} D \varepsilon_1 \right)$ and $B = \frac{1+E}{D} (A - 1)$. This integrated form provides useful estimates of the changes in volume and shape of the void in various plastic flow fields. However, it should be emphasised that the integration is strictly valid only when the principal axes of the strain-rates ($\dot{\varepsilon}_1, \dot{\varepsilon}_2, \dot{\varepsilon}_3$) remain fixed in direction throughout the strain path [65], including uniaxial tension ($\nu=1$), pure shear ($\nu=0$) and biaxial tension ($\nu=-1$). Furthermore, the integrated form is only correct if the triaxiality does not change during the loading, which is not true after the onset of necking.

The R&T model has been successfully used by many authors [69, 70, 71, 72, 73, 74], provided that the pre-exponential parameter α is taken as a free parameter adjusted to account for the approximations in the model. Marini *et al.* have shown that the parameter α increases with the initial void volume fraction. Indeed, the Rice and Tracey analysis describes the growth of a single void in an infinite perfectly plastic matrix. If the initial void volume fraction increases, the dilute void growth approximation becomes less accurate because the strain fields around the voids interact. This explains why the parameter α was found higher than the theoretical value when adjusting the model toward experimental measurements [69, 70, 71, 72, 73, 74]. Furthermore, the R&T model re-evaluated by Huang is developed for a perfectly plastic matrix, while the real material presents a strain hardening which tend to accelerate void growth. Lecarme *et al.* [74] have discussed several possible origins for the high value of the average factor α when compared to the one theoretically predicted by Huang (0.43).

The R&T model is now compared to the results of Tvergaard [75],

who performed void growth analysis using axisymmetric cell containing a single spherical void. The cell calculations are ideally performed at a constant imposed stress triaxiality and the integrated form of the R&T model given in eq.(1.18) can be used (with $\nu=1$ for axisymmetric loading). Fig. 1.10(a) and (b) shows the evolution of the void volume fraction and of the aspect ratio, respectively, for different stress triaxialities and for initial void volume fraction $f_0 = 10^{-8}$. Globally, Fig. 1.10(a) shows that the void growth predicted by the R&T model (dotted line) remains in good agreement with the cell analysis (plain line), irrelevant of the triaxiality.

Fig. 1.10(b) shows that in the cell simulations (plain line), the initial spheroidal void elongates along the loading direction for low triaxiality ($T < \frac{2}{3}$) and this is well captured by the R&T model. At larger triaxialities ($T > \frac{4}{3}$), the void tends to grow faster in the transverse direction and the void grows oblate, i.e. W_{void} decreases to values smaller than 1. In the R&T model, the void will always grow faster along the loading direction ($R_1 > R_2, R_3$ in eq. (1.18)) and the initial spheroidal void cannot become oblate. The spherical void grows and the shape factor W_{void} predicted by the R&T model approaches a constant value at large strain (see Fig. 1.10(b), dotted line). In other words, the void remains almost spherical at very large triaxiality in the R&T model (i.e. W_{void} remains ≈ 1 for $T = \frac{8}{3}$).

Furthermore, void growth analysis using axisymmetric cell indicates that the aspect ratio decreases smoothly (except for $T = \frac{2}{3}$) and then starts to decay abruptly (see Fig. 1.10.(b)). This abrupt change is associated with elastic unloading in most of the unit cell, resulting in the localized plastic flow between neighbouring voids that develops into void coalescence. This phenomenon is obviously not captured by the R&T model which describes the growth of an isolated void.

Growth of penny-shaped void

When voids nucleate by particle cracking, the void is initially penny-shaped and the mode of deformation is dominated by opening and blunting of the void/crack tip [75]. Unit cells have been used to simulate the growth and coalescence of initially penny-shaped voids under axisymmetric loading condition [76, 75]. Fig. 1.11 shows the evolution of the void aspect ratio W_{void} for a void initially very flat (W_{void}^0

1.1. Damage and fracture

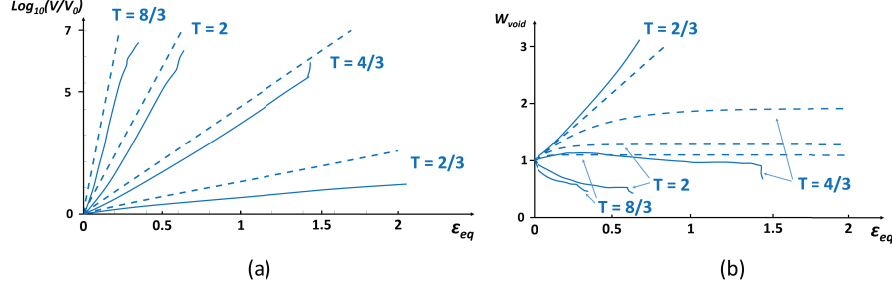


FIGURE 1.10: Evolution of the (a) void volume fraction and of (b) the void aspect ratio for different stress triaxialities and for initial void volume fraction $f_0 = 10^{-8}$ and cell aspect ratio = 1. The plain line indicate the unit cell results of Tvergaard (Redrawn from [75]) and is compared with the evolution predicted by the R&T model (dotted line, see eq. 1.18).

=0.091), with constant stress triaxiality and for a cell aspect ratio $\lambda_0=1$. The void grows mostly by opening in the loading direction for a large range of deformation and there is a limited effect of the stress triaxiality on the void aspect ratio W_{void} compared to the case of an initially spheroidal case (see Fig. 1.11.(b)).

Based on this observation, Lassance *et al.* have proposed a simple mapping between the evolution of the void aspect ratio W_{void} and the cell aspect ratio evolution λ :

$$\begin{aligned} W_{void} &= \frac{R_z}{R_x} = \frac{R_z^0 + \Delta R_z}{R_x^0 + \Delta R_x} = \frac{\Delta R_z}{R_x^0 + \Delta R_x} \\ &= \frac{\Delta L_z}{L_x^0 + \Delta L_x} = \lambda_0 \frac{\exp(\epsilon_{eq}) - 1}{\exp(-\epsilon_{eq}/2)} \end{aligned} \quad (1.19)$$

The evolution law given in eq. (1.12) is also shown in Fig. 1.11 and is in good agreement with unit cell calculations.

Void locking

At low triaxiality, the void elongates along the loading direction and contracts in the transverse direction. In reality, in the case of particle fracture, the particle fragments constraint the transverse contraction of the penny-shape void [76, 75]. The same behaviour occurs in the case

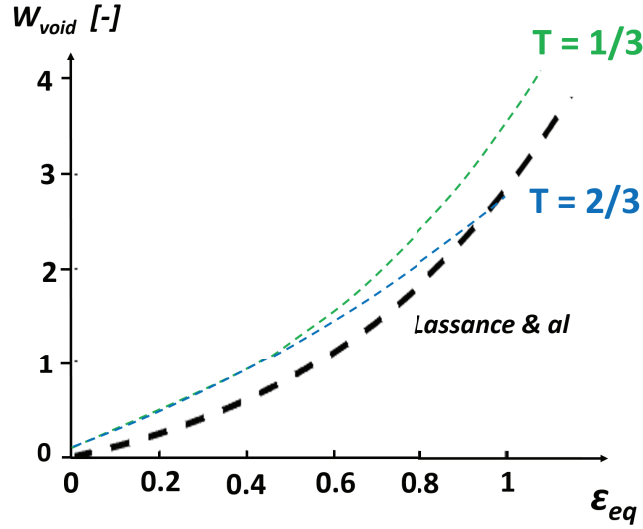


FIGURE 1.11: Unit cell results for the evolution of an initially penny-shaped crack (blue, green line) from Ref. [75]. The black line shows the void shape evolution given in eq. (1.19).

of void nucleation by interface decohesion, with the particle inside the cavity hindering the transverse contraction of the void [77, 78]).

Siruguet and Leblond [77] performed FE simulations of the growth of a void nucleated from a spherical rigid particle with no cohesion with the matrix. Fig. 1.12 shows the evolution of the void volume fraction with (solid line) and without (dashed line) particle inside the void, for $T = 0, 1/3$ and $2/3$ and initial porosity $f_0=0.01$. Note that the void volume fraction predictions for an empty void are based on an extended Gurson model and not on cell computations. At very low triaxiality ($T=0$), if no inclusion is present, the void collapses and the void volume fraction decreases. If there is an inclusion inside the void, void locking hinders such a collapse and the porosity increases slightly. For uniaxial tension ($T=1/3$), the increase of porosity remains limited because the void elongation along the rolling direction is partly offset by the void contraction in the transverse direction. If an inclusion is present, the inclusion hinders the void contraction and the void growth is more significant. As the stress triaxiality increases ($T=2/3$), the effect of the particle on the void growth becomes smaller because the contrac-

1.1. Damage and fracture

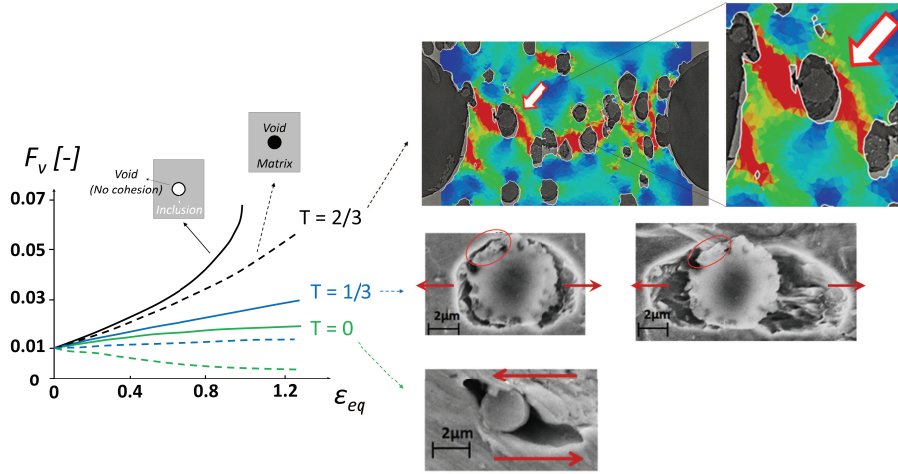


FIGURE 1.12: Left : FE simulations [77] of the growth of a spherical void (dotted line) and of a void nucleated from a spherical rigid particle with no cohesion with the matrix (plain line) for $T=0, 1/3$ and $2/3$. Right : Experimental observation of void locking by a particle inside a void at low (from [79]), intermediate (from [79]) and high triaxiality (from [80]).

tion of the void in the transverse direction gets weaker. Siruguet and Leblond [77] observed that void locking no longer occurs for a stress triaxiality larger than $2/3$.

Similarly, Bordreuil *et al.* [78] have studied FE voided cells containing or not a rigid spherical inclusion undergoing an axisymmetric uniaxial straining ($T=1/3$). Fig. 1.13 shows the evolution of the void radii along the loading direction (R) and in the transverse direction (r). As already explained, the void elongates along the rolling direction (R increases) and contracts in the transverse direction (r decreases) in the absence of a particle. When contact takes place between the void and a debonded rigid inclusion, the inclusion stops the matrix displacement and the corresponding radius is constant (i.e. r is unchanged with straining).

Furthermore, it is observed that the evolution of the void radius along the rolling direction (R) is not strongly modified by the contact with an inclusion. In other words, the radius change in the loading direction is not strongly coupled to the radius change in the transverse direction. Based on this observation, Bordreuil *et al.* have proposed

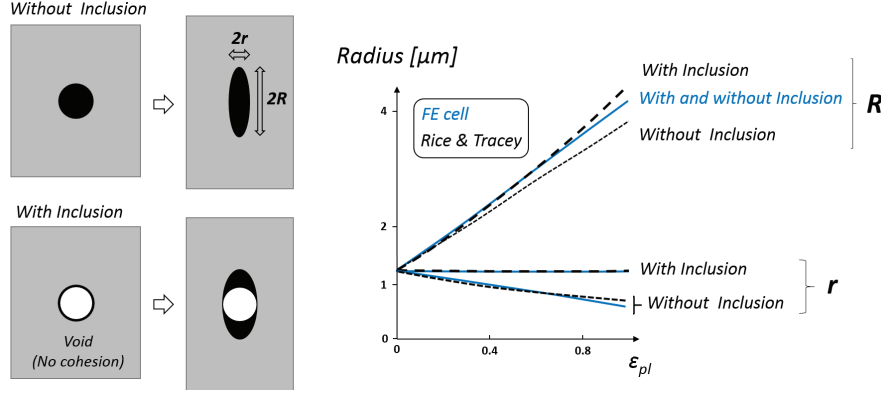


FIGURE 1.13: FE simulations of the growth of a spherical void and of a void nucleated from a spherical rigid particle with no cohesion with the matrix, for $T=1/3$. Adapted from [78].

to use the R&T model (see eq. (1.12)) with a simple modification of the model. The inclusion stops the matrix displacement and the corresponding radius may not diminish (i.e. $\dot{r} \geq 0$). Fig. 1.13 shows that this modified version of the model reproduce quite well the evolution of the void radii.

Similarly, Tvergaard [75] has studied the effect of particle fragments on the growth of initially penny-shaped voids using axisymmetric cell (see Fig. 1.14(b)). The effect of void locking by the broken particle is quite similar with what has been previously observed in the case of interface decohesion. Fig. 1.14(a) shows the evolution of the porosity for a void initially very flat ($W_{void}^0 = 0.091$) with (plain line) and without (dotted line) a cracked particle. The effect of void locking is more important at low triaxiality ($T=1/3$). For stress triaxiality larger than $2/3$, the void growth is unchanged with and without the cracked particle, as previously observed in the case of decohesion of the particle–matrix interface by Siruguet and Leblond [77]. It is interesting to note that this critical triaxiality is quite well predicted by the R&T model (dotted line). Indeed, void locking effect is expected only in the case of void contraction and the R&T growth model given by eq. (1.12) predicts a decrease of the void size in the transverse directions (i.e. $\dot{R}_2 \geq 0$) for $T < 0.567$ (for $1 + E=2$ in eq. (1.12)).

In Fig. 1.14(c), the penny-shaped void growth has been modelled

1.1. Damage and fracture

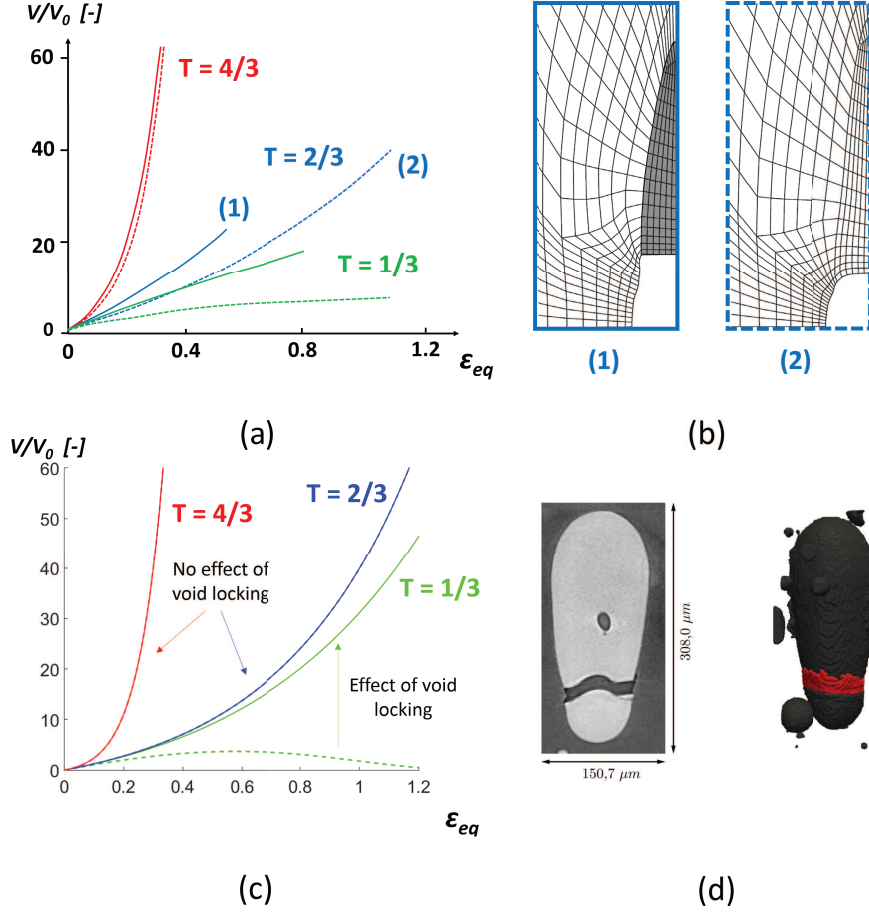


FIGURE 1.14: Effect of particle fragments on the growth of initially penny-shaped voids : (a) unit cell results of Tvergaard (redrawn from [75]) compared with the evolution predicted by the (c) R&T model. The unit cell used with/without the particle fragments is shown in (b). The initial void aspect ratio W_{void}^0 is 0.091 and the particle aspect ratio W_p is 5. In both (a) and (c), plain/dotted line corresponds to void growth with/without void locking effect. Figure (d) shows void locking observed by X-ray tomography (reproduced from [81]).

(dotted line) with the evolution law of Lassance *et al.* given in eq. (1.12) for the opening of the void along the loading direction and the R&T model for the void growth in the transverse direction ($R_2 = R_3$ in eq. (1.18)). This simple model (Fig. 1.14(c)) reproduces well the poros-

ity evolution from the cell computations (Fig. 1.14(a)). In analogy with the modification of the R&T model proposed by Bordreuil *et al.* (see Fig. 1.14,[78]), it is proposed to constraint the transverse void growth : $R_2 \geq R_2^0$ in eq. (1.18). Fig. 1.14(c) shows the evolution of the porosity with the modified model (solid line). The void growth is only modified at low triaxiality ($T=1/3$) since the R&T growth model predicts a contraction of the void only for $T < 0.567$, as explained previously. Nevertheless, at low triaxiality, the effect of void locking is quite well captured with this simple modification of the R&T growth model.

The Gurson model

The void growth must be coupled with a yield criterion to capture material softening and its influence on the yield behaviour of the material. Gurson [3] was the first to propose a damage based yield criterion and flow rules for a porous ductile material. The analysis is based on the upper bound theory of plasticity. In this model, the real void distribution within the material is replaced with an equivalent void located at the center of a cylindrical or spherical unit cell. Gurson obtained the following upper bound approximation for the yield surface :

$$\Phi = \frac{\sigma_{eq}^2}{\sigma_y^2} + 2f \cosh\left(\frac{3\sigma_h}{2\sigma_y}\right) - 1 - f^2 = 0, \quad (1.20)$$

where f is the void volume fraction, σ_{eq} is the equivalent von Mises stress, σ_y is the yield stress and σ_m is the hydrostatic stress. The behaviour of the matrix is described by the von Mises yield criterion and the Gurson model reduces to the von Mises yield criterion for a damage-free material (i.e. when $f=0$). A von Mises material is incompressible and does not yield plastically under pure hydrostatic stresses. In the presence of a porosity ($f>0$) and under large hydrostatic stress, tensile hydrostatic stress will expand the voids, softening the material and resulting in plastic yielding whatever the value of the deviatoric stress. This is why constitutive models for porous media, such as the Gurson model, are also called dilatant plasticity models.

Significant efforts have gone into refining the model and many extensions have been proposed in order to improve global accuracy [82] and incorporate more physics into the model, such as void shape effects [83], strain hardening of the matrix [5] or plastic

1.1. Damage and fracture

anisotropy [84]. However, the format of the model remains basically the same and these models assume the existence of a periodic arrangement of voided unit cells. In other words, damage is treated as a global process and the Gurson model (and extensions) is not suited to study the role of the void distribution on fracture.

1.1.3.3 Void coalescence

According to Thomason [65], the physical interpretation of void coalescence is the competition between a stable, homogeneous deformation mode and an unstable, highly localized deformation mode in the ligament matrix.

In the early stages of deformation, the influence of the voids on the material response is negligible. In other words, the voids are small, widely spaced and the value of the stress which is required to initiate localised plastic flow σ_n is considerably greater than the macroscopic plastic flow σ_z . The transition to a localized deformation mode within the void ligaments would thus be energetically unfavourable and the diffuse flow is better predicted by a constitutive model for porous media, such as the Gurson model (see Fig. 1.15).

With increasing plastic flow, the energy required to transition to a localized deformation mode within the void ligaments decreases due to two different contributions. First, the local stress transferred on the matrix ligament from the macroscopic plastic flow σ_z increases because the effective area fraction of the ligament decreases with void growth. Second, the value of σ_n is changing with the geometry of the void because the ligament "neck" increase the ligament stress triaxiality. Thomason [85] has shown that this geometrical effect can be rationalized by the use of a "plastic constraint factor", C_f , which is a function of the void aspect ratio (W_{void}) and the void spacing ratio (χ).

The plastic limit-load of the ligament, i.e. the transition to the localized deformation mode, is thus reached when the following constraint is satisfied :

$$\frac{\sigma_1}{(1 - \gamma\chi^2)} \geq C_f(W_{void}, \chi) \sigma_0 \quad (1.21)$$

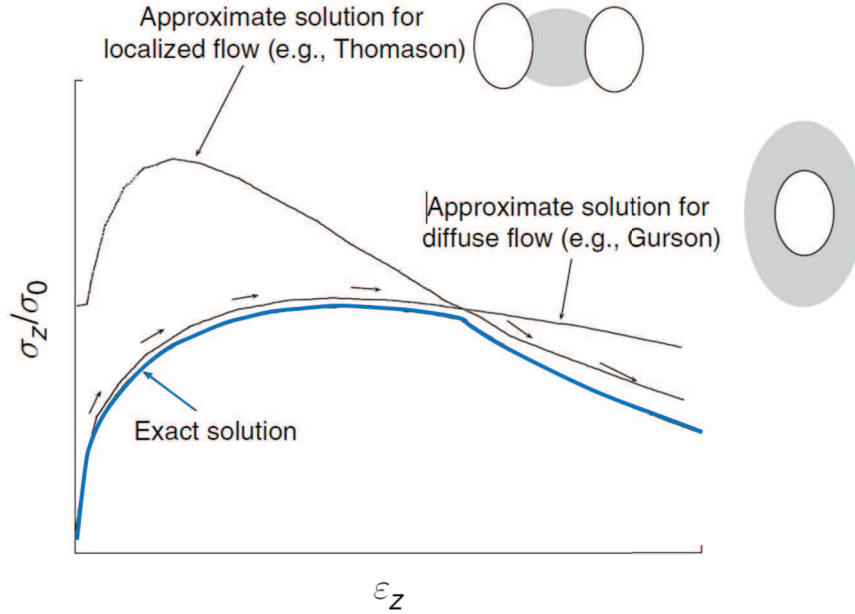


FIGURE 1.15: Schematic description of the competition between the two modes of plasticity. In the early stages of deformation, the growth is stable. At some point, the deformation localized within the void ligament. Reproduced from [24].

where $\gamma=1$ for an axisymmetric unit cell and $\gamma = \pi/4$ for a cubic cell.

Thomason [85, 86] has used two different unit cells, namely a square-prismatic unit cell containing a square-prismatic void and a cylindrically symmetric unit cell containing circular-cylindrical void, in order to assess the sensitivity of the model to the detailed void shape. The matrix material was assumed to be perfectly plastic in both cases. The two different unit-cell models gave very similar dependence of the constraint factor with W_{void} and χ . In the case of the axisymmetric cell, the following expression gives an empirical expression for C_f which is a close approximation of the upper-bound analysis [85] :

$$C_f = \left[\alpha \left(\frac{1-\chi}{\chi W_{void}} \right)^2 + \beta \frac{1}{\sqrt{\chi}} \right], \quad (1.22)$$

1.1. Damage and fracture

with $\alpha=0.1$ and $\beta=1.2$. The constraint factor C_f decreases as the voids open (W_{void} increases) and get closer to each other (χ increases), see Fig. 1.16.(a). From a physical point of view, this translates a lower ligament stress triaxiality which increases the rate of plastic deformation and favours void coalescence. Thomason has proposed to use the same constraint factor for ellipsoidal voids by identifying the void aspect ratio W_{void} with the height-to-diameter ratio of the cylindrical void. He claimed that this approximation should not lead to any serious inaccuracy as long as the void volume fraction is smaller than about 0.2.

Several variants of the plastic constraint factor C_f have been proposed in order to generalize and enhance the model, but the essence remains the same.

The expression for the constraint factor C_f was improved by Pardoen and Hutchinson [5] in order to incorporate material hardening. The parameter α is replaced by a function of the strain-hardening exponent n ($\alpha=0.1+0.217n+4.83n^2$) and σ_0 is replaced by the current mean yield stress σ_y of the matrix.

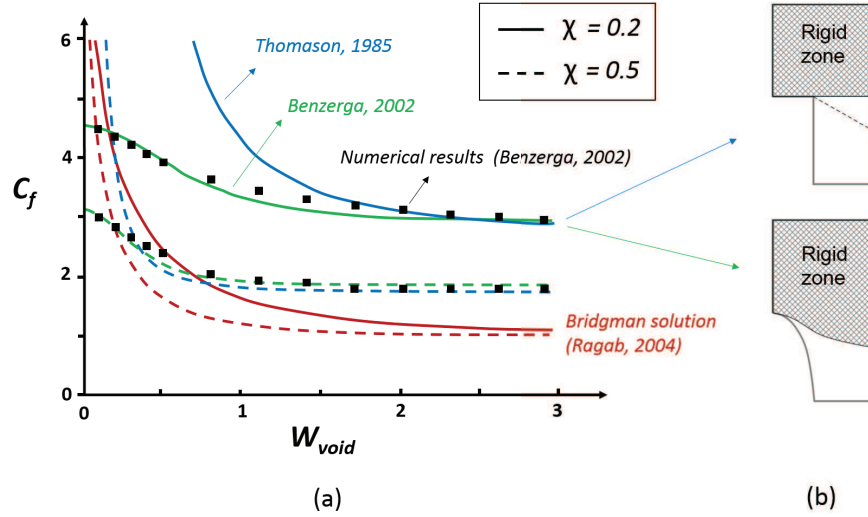


FIGURE 1.16: (a) Effect of the voids configuration (W_{void} and χ) on the plastic constraint factor C_f . (b) Unit cells used by Thomason [85, 86] and by Benzerga [25].

Thomason's coalescence model does not predict any coalescence

between very flat voids. Indeed, the constraint factor C_f given by eq. (1.16) goes to infinity in the limit of very flat voids, i.e. W_{void} approaches 0. This behaviour is due to the axisymmetric velocity fields used by Thomason which diverge for penny-shaped cracks. In other words, eq. (1.21) will never be satisfied and the Thomason's model predicts that no coalescence would occur for very flat voids. To remove this limitation, Benzerga [87] used improved trial velocity fields in the limit-analysis procedure (see Fig. 1.16.(b)) and the numerical results are shown in Fig. 1.16.(a). Benzerga proposed a new form of the plastic constraint factor based on these numerical results :

$$C_f = \left[0.1 \left(\frac{\chi^{-1} - 1}{W^2 + 0.1\chi^{-1} + 0.02\chi^{-2}} \right)^2 + 1.3 \frac{1}{\sqrt{\chi}} \right], \quad (1.23)$$

In practice, however, the quantitative predictions obtained with the two models are very close for most cases analysed [25]. This can be explained by the fact that penny-shaped voids rapidly open in the loading direction, get more rounded and W_{void} increases rapidly to values for which both models give similar results (i.e. W_{void} larger than about 1, see Fig. 1.16(a)). Furthermore, the highly constrained and localized character of plastic flow in the intervoid ligament during void coalescence swamps the effect of void shape (W_{void}) and it is the ligament size ratio χ which is the key parameter controlling the transition to the coalescence mode.

A generalization has been proposed by Scheyvaerts *et al.* [88] in order to account for the orientation of the inter-void ligament. Indeed, the void axes are not necessarily aligned with the principal loading directions and the ligament where coalescence takes place is not necessarily transverse to the maximum principal stress. The two voids are transformed into an effective configuration made of two identical voids (defined by W_{eff} and χ_{eff}) in order to allow the evaluation of the coalescence model and σ_1 is taken as the stress normal to the ligament. This extension of the coalescence criterion reduces to its original definition when the voids are aligned and have the same orientation.

In many ductile metallic alloys, the growth and coalescence of primary voids (typically larger than 1 μm) is affected by the growth of a second population of much smaller voids. These secondary voids do not affect the stable growth of the primary voids but strongly accelerate

1.1. Damage and fracture

the void coalescence process by softening the material in the ligament between the voids where the strains are very large [22]. The softening induced by the growth of secondary voids is heuristically introduced in the coalescence criterion [22] by multiplying the yield stress of the matrix material by $(1-f_2)$, with f_2 being the maximum value of the secondary voids volume fraction over the ligament.

The coalescence criterion given by eq. (1.22) can be used to define a simple indicator of the propensity of a void configuration to initiate the coalescence process. The coalescence indicator I_{coal} is given by :

$$I_{coal} = \frac{\cos(\phi)^2}{(1 - \chi^2) C_f} \quad (1.24)$$

where ϕ is the angle between the loading direction (σ_1) and the inter-void ligament. The propensity of a pair of void to enter a coalescence process increases if the indicator I_{coal} increases.

Fig. 1.17.(a) [89] shows the deformation sequence of a laser drilled model material made of an Al 5052 plate (with thickness around 100 μm) containing a random array of holes (with diameter around 10 μm). These pictures are taken in situ at far-field true strains equal to 0, 0.204 and 0.206. Fig. 1.17.(b) shows the initial void distribution and the corresponding Voronoi cells. The coalescence indicator I_{coal} has been evaluated for each pair of neighbouring voids, i.e. sharing a common Voronoi cell facet. In Fig. 1.17.(b), a red line has been drawn between 20% of void pairs which have the highest I_{coal} . The fracture path after void linkage shown in the last macrograph of Fig. 1.17(a) is in good agreement with Fig. 1.17(b).

A similar comparison has been made with a random multivoid cell computation carried out by Shakoor *et al.* [90], see Fig. 1.17(b),(c),(d). Again, the largest values of I_{coal} (see Fig. 1.17(a)) compares well with the FE cell at the early stage of void coalescence shown in Fig. 1.17(c). This is interesting to note that the Thomason model was not used in [90] because it did not seem to be applicable to the authors.

Effect of a secondary void population on the coalescence process

After the onset of the coalescence process, the merging of the voids

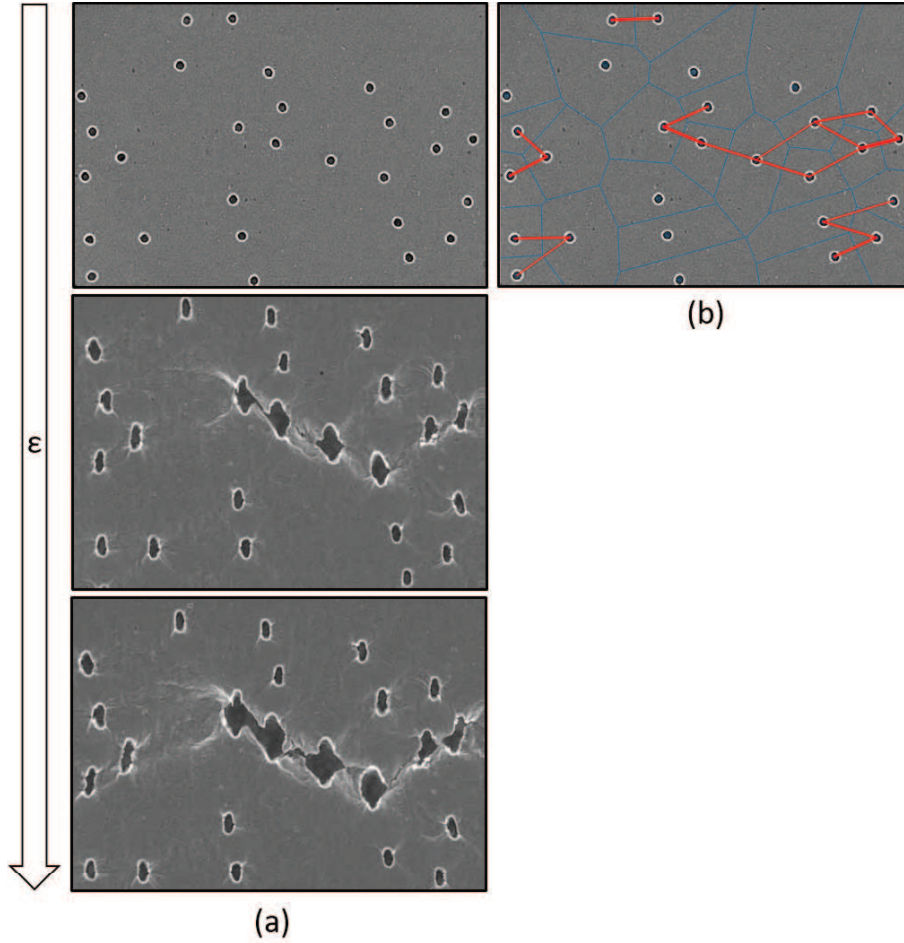


FIGURE 1.17: (a) Deformation sequence of a laser drilled model material made of an Al 5052 plate (with thickness around $100\ \mu\text{m}$) containing a random array of holes (with diameter around $10\ \mu\text{m}$). Reproduced from [89]. (b) A red line indicates location where void coalescence is likely to occur first, according to the coalescence indicator I_{coal} .

usually consists in the failure of the remaining ligament, for example with the help of a second population of smaller voids, rather than radial void growth until impingement [24]. Two modelling strategies have been used to address the effect of a second population of voids on the coalescence process. In both cases, the primary voids are simulated numerically using FE void simulations. In the first strategy, the secondary

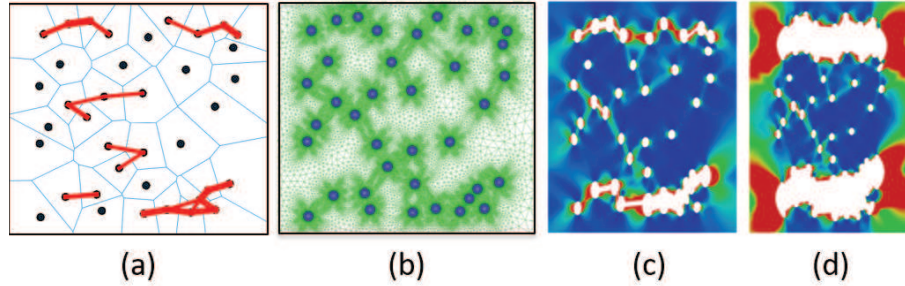


FIGURE 1.18: *(b, c, d)* Deformation sequence for a random multivoid cell. Reproduced from [90]. *(a)* A red line indicates location where void coalescence is likely to occur first, according to the coalescence indicator I_{coal} .

population of voids is explicitly taken into account by incorporating them in the FE mesh of the matrix ligament between the primary voids [23, 91]. In the second strategy, the softening effect of the second population of void is implicitly taken into account by using a Gurson-type model for the behaviour of the matrix ligament [22].

1.1.4 Simple fracture strain model

The micromechanical models that were reviewed in previous sections are generally applied to structural materials. However, more phenomenological models (which have not been included in this literature review) are commonly employed in sheet metal forming [27]. In this section, a very simple model is developed based on the micromechanical models that were proposed in the last sections. The objective is to illustrate that micromechanics based models, whose applicability in metalworking remains to be demonstrated [27], provides a fertile ground for modelling failure in metal forming.

Phenomenological plasticity models provide quite accurate predictions of the multi-axial stress-strain response of metals up to the point of strain localization. This is probably due to the fact that the void growth, and thus the void volume fraction, remains limited before up to this point [92]. The Mohr-Coulomb (MC) failure criterion is commonly used for describing ductile fracture even if it was initially developed for rock and soil mechanics. This criterion was extended

by Mohr and Marcadet [92] to the Hosford-Coulomb criterion (HC) by replacing the Tresca stress in the MC model by the Hosford equivalent stress. The HC-model writes :

$$\sigma_{HF} + c_{HF}(\sigma_1 + \sigma_3) = \sigma_{coh} \quad (1.25)$$

$$\sigma_{HF} = \left(\frac{1}{2}((\sigma_1 - \sigma_3)^a + (\sigma_1 - \sigma_2)^a + (\sigma_2 - \sigma_3)^a) \right)^{\frac{1}{a}} \quad (1.26)$$

where $a_{HC}, c_{HF}, \sigma_{coh}$ are calibration parameters which are used to fit the experimental data.

This models is based on the assumption that the onset of fracture is imminent with the formation of a primary localization band. The validity of this assumption depends strongly on the failure scenario, as discussed in the following section.

Localized plastic flow prior to damage : If failure is caused by localized plastic flow prior to damage (mechanism [b] of Fig. 1.1), phenomenological models should thus work quite well.

Failure by localization induced by the accumulated porosity: In this mechanism (mechanism [b] of Fig. 1.1), voids appear prior to macroscopic localization and reduce the load-bearing area so much that flow localizes in a narrow band. In other words, the macroscopic localization is driven by the softening due to the growth of the voids. The width of this localization band is expected to be of the order of one or more inter-void spacing [5].

A very simple model is now developed to compare the HC-criterion with a fracture strain prediction based on void nucleation, growth and coalescence. The results are developed for plane stress condition. Indeed, there is a one-to-one relationship between the triaxiality T and the Lode parameter ν in plane stress condition (see Ref.[93] for details). This relationship allows easier transformation from the stress state representation (Σ_1, Σ_2) to the mixed strain-stress state (ϵ, T) , which is the what is typically measured experimentally.

Void nucleation is modelled with a simple Beremin approach, see section 1.1.3.1, $\sigma_{Nucleation} = \Sigma_{max}^{Princ} + (\Sigma_{eq} - \Sigma_0)$. The critical stress void nucleation σ_c is equal to $1.8\sigma_0$. For each loading condition (i.e. each pair of T, ν), void growth is modelled with the R&T model, see sec-

1.1. Damage and fracture

tion (1.1.3.2). The initial void volume fraction f_0 at void nucleation is equal to 0.05 and the voids are initially spheroidal. The void coalescence is predicted with the Thomason criterion, see eq. (1.22). The onset of void coalescence is taken as the indicator of the fracture strain of the material.

As already explained in section 1.1.3.2, the R&T growth model given by eq.(1.12) predicts a decrease of the void size in the transverse directions (i.e. $R_2 \geq 0$) for low triaxiality. At low triaxiality, there is thus a critical void volume fraction at initiation (f_0) under which the transverse contraction of the voids is so important that coalescence is never taking place [76]. The critical triaxiality T_{crit} is shown in Fig. 1.19(a) and (b). For this case, the choice of f_0 of 0.05 gives a critical triaxiality around 1/3. The evolution of the critical T_{crit} with f_0 is shown in Fig. 1.19(c). Obviously, if the initial porosity f_0 increases, T_{crit} decreases.

In reality, fracture is also observed for $T < T_{crit}$. At very low triaxiality, fracture involves the catastrophic linkage of voids in a shear band. A very simple model, proposed by Teirlinck *et al.* [15] considers that unstable void growth (i.e. void coalescence) will occur if the work hardening of the matrix is insufficient to compensate for the increase of shear stress within the localization band. It is then easy to show that the instability condition can be expressed as:

$$n\left(\frac{K}{\sigma_{eq}}\right)^{(1/n)} = V_0 N_v \quad (1.27)$$

where K and n are work hardening constants of a power hardening law, and V_0 and N_v are the volume of one void and the number of voids per unit volume. A low triaxiality, voids do not grow much and the right-hand side term depends only on the number of voids which have nucleated at a given stress. The left-hand side depends only of the flow properties. In this case, failure corresponds thus to the attainment of a number of voids per unit volume large enough to trigger plastic localization. Void nucleation must thus be distributed and a Weibull statistic ($m=5$, $\sigma_w=2\sigma_0$) is used for the evolution of void nucleation (see section 1.1.3.1).

A comparison between the HC-Coulomb criterion and the fracture strain evolution given by these simple models (see Fig. 1.19(b))

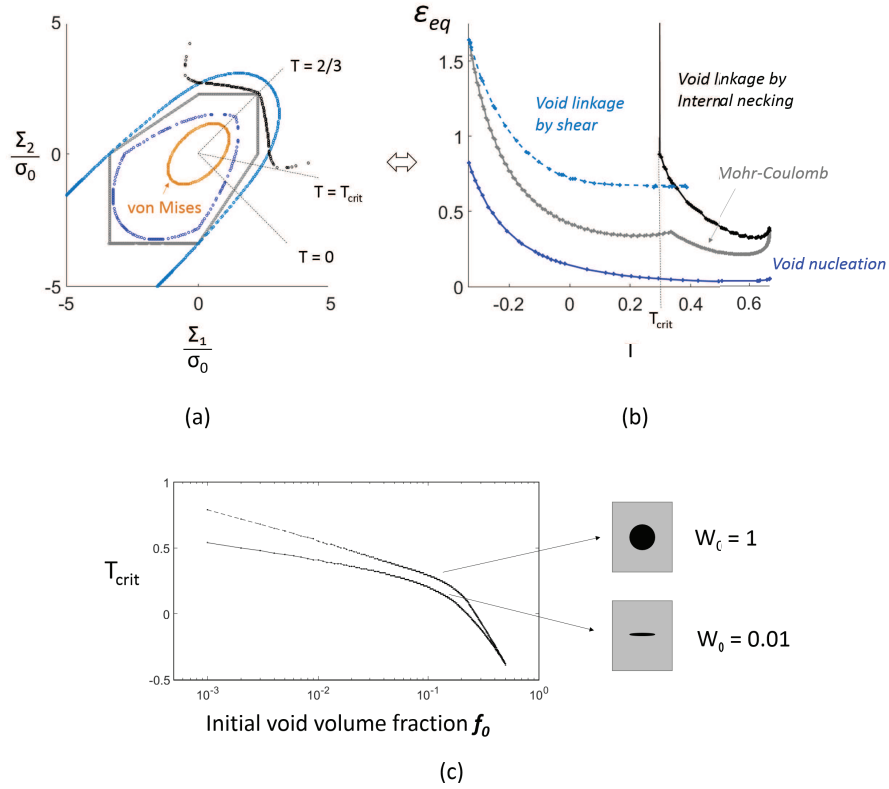


FIGURE 1.19: (a), (b) Comparison between the HC-criteria (with randomly picked parameter $a_{HC}=1, c_{HF}=0.2, \sigma_{coh}=2.7\sigma_0$) and the void nucleation, growth and coalescence approach in plane stress condition. (c) Evolution of T_{crit} with the volume fraction of void at nucleation f_0 for a penny-shaped void ($W_0=0.01$) and spheroidal void ($W_0=1$).

shows that the shape of the fracture locus might be linked to the damage process. This also contributes to explain why good predictions of ductile fracture can be achieved with a HC-criterion, even though it is only a phenomenological model.

Failure by cluster localization : In this case (mechanism [e] of Fig. 1.1), coalescence occurs over a limited number of neighbouring voids. The assumption that the onset of fracture is imminent with the formation of a primary localization band does not correspond to the physics of the fracture process. In this case, failure is related to the

1.1. Damage and fracture

percolation of microcracks which are not necessarily concomitant with plastic localization. As already explained, this fracture scenario is at the core of the present work.

1.2 Tomography

1.2.1 Short history of the tomography

The word "tomography" comes from the Greek word "tomos" meaning "slice". The technique has been initially developed for medical applications in the 1970s. Cormack [94] was the first to demonstrate the feasibility to reconstruct the cross-section image of an object with a limited number of X-ray radiographs. As a theoretical physicist, Cormack was not concerned about the practical application of his research [95] and the construction of the first CT scanner was achieved by Hounsfield [96]. Hounsfield developed a new approach for the image reconstruction using the computers available at that time. In this way, the concept of computed tomography was born⁷. Computed tomography improved with the development of digital computers [97] and industrial X-ray computed tomography for non-destructive materials characterization were marketed in the 1980's.

1.2.2 The principle of X-ray tomography

The principle of X-ray tomography basically consists in an extension of classical X-ray radiography. A series of radiographs is recorded for different angular positions of the sample, which rotates around an axis perpendicular to the beam. Each radiograph provides only a projection of the variation of X-ray absorptivity within the object. The set of radiographs (a scan) is then combined to reconstruct the three-dimensional distribution of the local attenuation coefficient of the material. The reconstructed volumetric image consists of a 3D matrix of voxels (analogous to pixels in a 2D digital image), for which the grey-level of each voxel describes the X-ray attenuation at that position.

In the case of a monochromatic beam (constant energy E) and in the photoelectric domain ($E < 200$ keV), the local attenuation coefficient

⁷Cormack and Hounsfield, generally credited with inventing computed tomography, were awarded the Nobel Prize in Medicine in 1979.[95]

1.2. Tomography

is commonly described by [98] :

$$\mu_E(x, y, z) = \frac{K\rho Z^4}{E^3} \quad (1.28)$$

where K is a constant, ρ and Z are the local density and atomic number, respectively. This equation translates the fact that the attenuation of the beam is highly dependent on the atomic number of the material and the X-ray energy E . This dependence is due to the fact that the absorption of X-rays is caused by the interactions of the photons with bound electrons in the atom [99]. If the photon energy exceeds the binding energy of each particular shell, then it may be absorbed and the energy is used to liberate the electron. This means that in Al alloys, for example, the heavier iron-rich particles absorb more X-rays than the lighter Al matrix.

The voxel size is dependent on the field of view and the number of detector elements. Indeed, the specimen must remain in the field of view for all rotations in order to ensure a good volume reconstruction. For a fixed voxel size, the maximum specimen diameter is thus limited by the number of elements of the detector (usually between 1000 and 4000 pixels across the width for a CCD detector [100]) multiplied by the voxel size. Recently, commercial nanoCT systems have started to appear in research laboratories. Spatial resolutions substantially below $1\text{ }\mu\text{m}$ can be achieved, but much smaller specimen diameters than in microCT are thus required. For example, for the Zeiss Xradia systems [101], the microXCT-400 provides pixel size down to about $0.5\text{ }\mu\text{m}$ with a field of view of 0.65 mm and the typical sample size required is 2 mm . The XRadia Ultra-810 offers a pixel size of $\approx 65\text{ nm}$, but a limited field of view of $65\text{ }\mu\text{m}$ and requires samples smaller than $\approx 0.8\text{ mm}$. This is the reason why 3D imaging is increasingly considered as part of a multi-scale strategy, involving the characterization of matter across multiple length scales using complementary techniques.

1.2.3 X-ray set-ups

Modern laboratory projection micro-XCT systems operate in cone beam geometry (Fig. 1.20(a)). With the use of a cone beam, the spatial

resolution is changing with the source-to-object distance. Synchrotron X-ray microtomography uses a parallel beam (Fig. 1.20.(b)) where the source-to-object distance is very large (e.g. 145m on ID19 at the European Synchrotron Radiation Facility (ESRF) [102]). In this case, the spatial resolution can be tuned using different types of detector [98]. The parallel beam geometry facilitates in-situ experiments since a large distance between sample and detector can be maintained without loss of intensity [101]. Synchrotron facilities offer X-rays with high flux, and high coherence, with the ability to tune the X-ray spectrum from a broad spectrum to a monochromatic beam [101]. The high flux also facilitates in-situ experiments as it enables rapid tomography data collection [103]. Furthermore, monochromatic X-rays are advantageous since their wavelength can be specifically tuned to achieve greater absorption contrast compared to a broad spectrum source (such as laboratory sources) [101].

Nowadays, multiple manufacturers offer turnkey microCT systems for routine, day to day laboratory characterisation [104]. However, commercial laboratory microCT employs polychromatic radiation and provide thus lower contrast than synchrotron microCT with monochromatic radiation. A recent review of commercial laboratory microCT systems can be found in Ref. [104]. The high flux, high coherence, the wide range of energies, imaging modalities and spatial resolutions available with good time resolution [103] makes of synchrotron X-ray tomography the most attractive approach to investigate the impact of heterogeneities in the physics of ductile failure.

1.2.4 Recent developments in X-ray tomography

The evolution of the number of publications involving X-ray microtomography and nanotomography related to materials science applications is shown in Fig. 1.21. Microtomography is usually associated with at least $50\mu\text{m}$ spatial resolution and nanotomography for spatial resolutions substantially below $1\mu\text{m}$ [104]. It shows a rapid increase in publication rate since the mid 1990s, which is concomitant with large improvements of the systems and the increase in accessibility that have occurred since that period [104]. Improvements of the systems are mainly due to the use of 3rd generation synchrotron

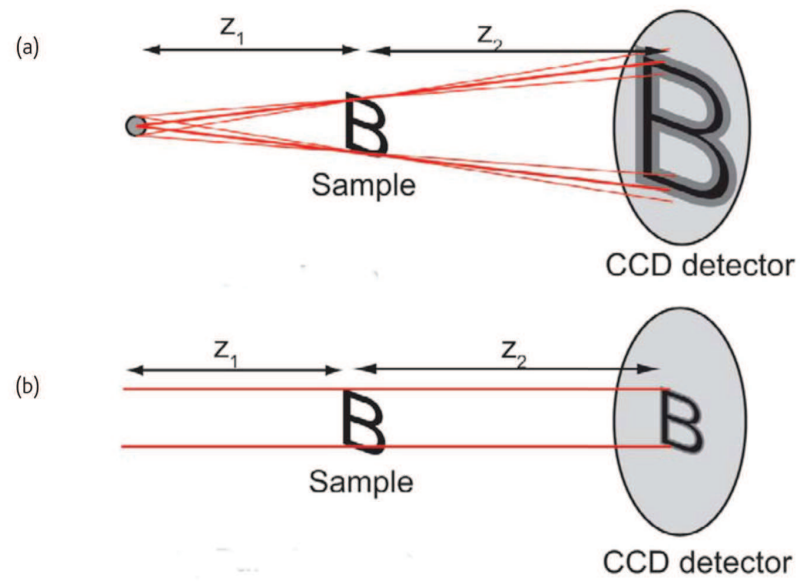


FIGURE 1.20: Schematics illustrating (a) cone-beam and (b) parallel-beam projection systems. Reproduced from [102].

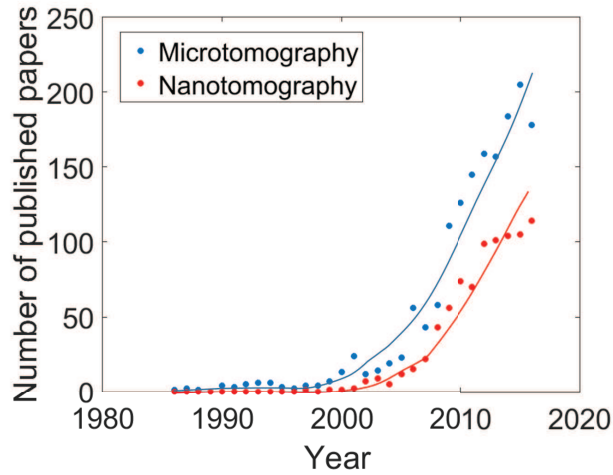


FIGURE 1.21: Evolution of the number of publications involving X-ray tomography related to materials science applications (according to Elsevier Scopus, www.scopus.com, consulted in July 2017).

radiation sources [98] and the development of high-tech detectors (linked with the demand for improved area detectors for consumer electronics [104]).

Recently, multi-scale correlative methods, which involve the coordinated use of X-ray tomography combined with other characterization techniques have been developed ("coordinated" refers to the fact that all analyses are performed for the same 3D region). This type of approach allows a more complete understanding than either method could have provided separately. Such a 3D correlative approach has been used recently to provide greater insight into the mechanisms of ductile fracture in low alloy steel [105].

1.2.5 Laminography

It is difficult to image flat, laterally extended specimens using tomography because the X-ray transmission varies strongly with the rotation during a scan and this is prone to generate imaging artefacts [19]. For laminography, the rotation axis of the specimen is tilted at an angle

1.2. Tomography

with respect to the beam direction which makes it possible to observe objects that are extended in two directions and thin in the third one [106].

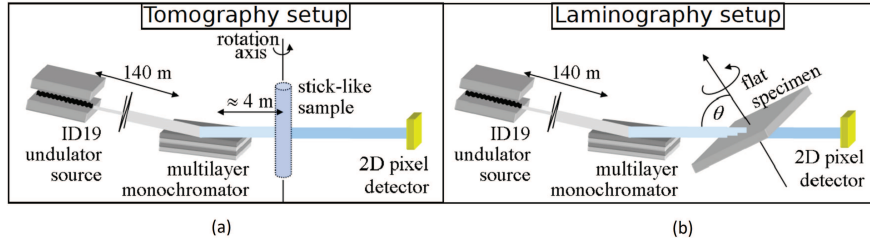


FIGURE 1.22: Tomography and laminography experimental setup. Reproduced from [106].

Fig. 1.22 compares typical tomography and laminography experimental setups. In the case of tomography, the largest dimension of the specimen is perpendicular to the beam direction (i.e. $\theta=90^\circ$). For laminography, the rotation axis of the specimen is tilted at an angle $\theta<90^\circ$ with respect to the beam direction. A series of radiographs is then recorded while the specimen turns about the normal vector of the sheet plane. A drawback of laminography is that the sampling of the 3D Fourier domain of the region of interest is incomplete and leads to imaging artefacts [106].

1.2.6 Human vision and impacts for the thesis

In this section, a few comments are made on some characteristics of human vision and their implications for this thesis. A very good introduction to human vision which does not go too deeply into the anatomical details of the visual system can be found in [99].

It is well known from CT users that the perceived volume fraction of objects, such as particles, in a 3D perspective is exaggerated. As explained by Hibbard [107], all 3D graphics suffer from ambiguity because many 3D points are projected onto the user's 2D retina. Produced images may look nice, but it can be really hard to extract spatial relationships or quantities based on these 3D perspectives. Part of this difficulty can be explained by the human preference for simple shapes

and continuous surfaces (also called the "illusion of preference" [99]) and is illustrated in Fig. 1.23. The black sphere is obviously on top of what seems to be a blue ellipsoid. However, when observed from a different angle, it becomes clear that this object is not an ellipsoid and has a smaller volume.

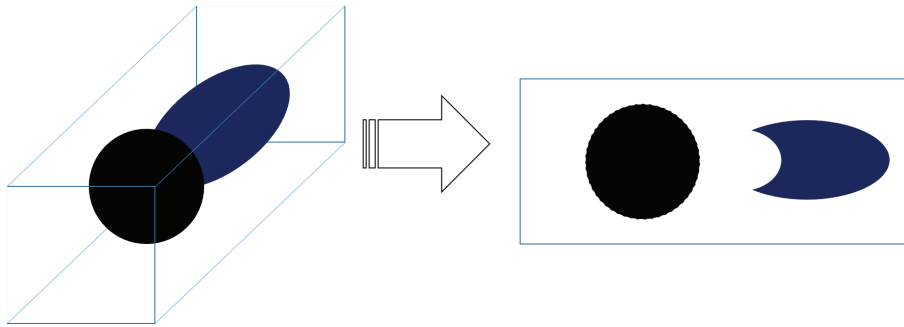


FIGURE 1.23: The black sphere is obviously on top of the blue ellipsoid. However, a view from another angle shows that the blue object is actually not an ellipsoid.

While some measurements (such as object size and location) are much more accurate even with simple computer algorithms than with any visual estimates, human vision remains extremely efficient for some other characteristics, such as grouping objects together to form structures. For example, in Fig. 1.24, it is easy to perceive the circumferential or radial alignment within the points patterns. Visual inspection helps to understand that this pattern can be captured by connecting each point to its nearest neighbours. However, visual inspection should always be supplemented with a more rigorous analysis in order to provide an objective classification of the pattern.

This capacity of grouping objects together into space is also true in time (a phenomenon called "aliasing" [99]). An object appearing in one time frame is associated with an object in the next if they are nearly of the same shape and if they are close to one another. In other words, humans are very good at tracking moving objects and predicting their path.

In this thesis, detailed characterization of particles sizes and spatial distributions will be performed. This short section illustrates that

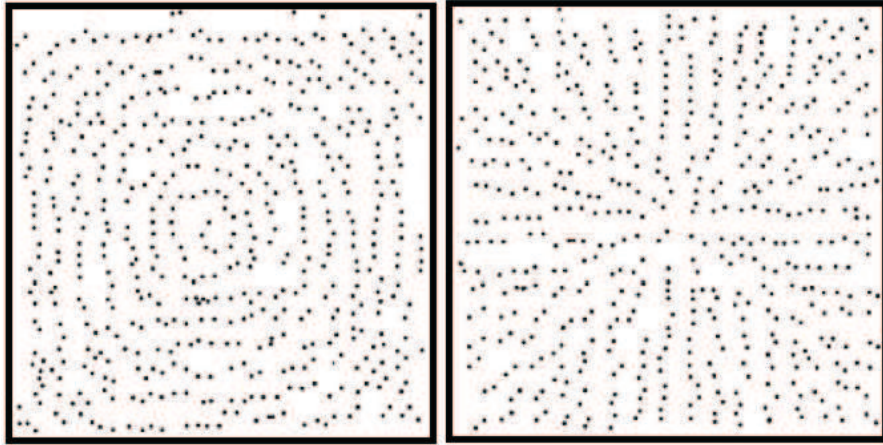


FIGURE 1.24: Example of mental interpretation of structures and objects. Reproduced from [99].

even though 3D visualization is a powerful tool to guide the analysis, it should always be supplemented by a quantitative image analysis. Furthermore, tracking algorithms will be used in order to identify the particles and the cavities from one tomographic scan to the other. Visual inspection will be used in order to choose the parameters (such as size, shape, ...) used for identifying the same objects in successive images, and to assess the quality of matching.

1.3 Aluminium alloys

The attractiveness of aluminium⁸ (Al) and its alloys are their high strength-to-weight ratios, resistance to corrosion by many chemicals, high thermal and electrical conductivities, non-toxicity, reflectivity, appearance [109]. Furthermore, it is one of the most easily fabricated high-performance materials, which usually correlates directly with lower costs [110]. Aluminium was first produced in 1825 [111]. It is the most abundant metallic element, making up about 8% of the Earth's crust, and its production is only exceeded by that of iron [109]. The principal ore for aluminium is bauxite⁹, which is an aluminium rich mineral in the form of aluminium hydroxide.

1.3.1 Standard nomenclature: alloy and temper

The Aluminium Association Wrought Alloy Designation System consists of four numerical digits. An alphabetic prefix or suffix is occasionally added, although it is rarely the case [112]:

- The first digit defines the major alloying class of the series starting with that number.
- The second defines variations in the original basic alloy: that digit is always zero (0) for the original composition, one (1) for the first variation, and so forth.
- The third and fourth digits designate the specific alloy within the series. There is no special significance to the values of those digits

Different series of Al alloys are employed in different applications [113]:

- Pure aluminium and solid solution alloys (1xxx, 3xxx and 5xxx series) are used in kitchen foil, drinks cans and formable sheet products.

⁸The American spelling (Aluminum instead of the "ium" common to metals) comes from an advertisement of the Pittsburgh Reduction Company (today known as Alcoa) [108]. It is unclear whether the omission was intended or if it was a mistake.

⁹The rock was named bauxite after Les Baux, the area where it was found [111].

1.3. Aluminium alloys

- Precipitation-hardened alloys (2xxx, 6xxx and 7xxx series) are employed in rocket motor tubes, in extrusions, struts and panels in land-based transportation vehicles; and in wings, fuselage and undercarriages for aircraft.
- Wide use is made of as-cast components (4xxx series) .

The heat treatable alloys (2xxx, 6xxx, 7xxx) contain elements whose solid solubility decreases with decreasing temperature, and whose concentrations exceed their equilibrium solid solubility at room temperature and moderately higher temperatures [110]. This provide significant solid solution and precipitation strengthening during solution heat treatment and subsequent ageing.

The temper designation is used to indicate the sequences of mechanical and thermal treatments which have been applied on the Al product. The first character in the temper designation is a capital letter indicating the general class of treatment (see [112] for more details). The general class of T temper is further detailed as it is widely studied in this work. The T temper indicates that the product has been thermally treated to produce stable tempers [112]. The T is always followed by one digit which indicates the specific combination of basic operations [114] :

- **T4:** Precipitation hardening results from natural ageing. Alloys in T4-type tempers are usually characterized by high ratios of tensile to yield strength and high fracture toughness.
- **T6:** Precipitation heat treatment following solution heat treatment and quenching. Alloys in T6-type tempers generally have the highest strengths practical without sacrifice of the minimum levels of other properties and characteristics found by experience to be satisfactory and useful for engineering applications.
- **T7:** Precipitation heat treatment following solution heat treatment and quenching. Alloys in T7 tempers are overaged, which means that some degree of strength has been sacrificed or "traded off" to improve one or more other characteristics. Strength may be sacrificed, for example, to lower residual stresses in order to reduce warpage or distortion in machining. In this work, T7 temper will be simply called overaged state.

Wrought aluminium alloys are produced in ingot or billet form and subsequently worked by any of a number of processes such as rolling, extruding, or other metalworking process to produce semifinished products from which end-use products are subsequently made. The standard orientations the semifinished products are :

- Longitudinal: The axis of the specimen is parallel to the longitudinal axis of the product.
- Transverse: The axis of the specimen is normal to the longitudinal axis of the product and it is within the major plane of the product.
- Short transverse: The axis of the specimen is normal to the major plane of the product, and thus normal to both the longitudinal and long transverse directions.

1.3.2 Fracture toughness of age-hardenable Al alloys

The "toughness tree" presented by Starke and Staley (see Fig. 1.25 [115]) is an effective representation of microstructural features that affect toughness in age-hardenable aluminium alloys. The microstructural features on the right-hand side are categorized as extrinsic effects because they are controlled rather by defects due to processing than by composition. On the other hand, microstructural features on the left-hand side are categorized as intrinsic effects because they are characteristic of the composition and microstructure of the alloy. Naturally, intrinsic features also depend on the response of the material to deformation processing and heat treatment.

Non-metallic impurities, hydrogen and porosity, i.e. extrinsic effects, are mainly associated with the casting and solidification processes [116]. Hydrogen content is kept low by minimizing exposure to moisture, filtering and direct-chill processing [115]. In general, hydrogen levels trapped in aluminium alloys ranges from 3 to 4 orders of magnitude larger than hydrogen solubility, due mainly to the existence of a hydrogen-solubility gap at the melting temperature of aluminium. As a consequence, micropores are formed in the aluminium matrix by the precipitation of molecular hydrogen [117]. Closing of these micropores

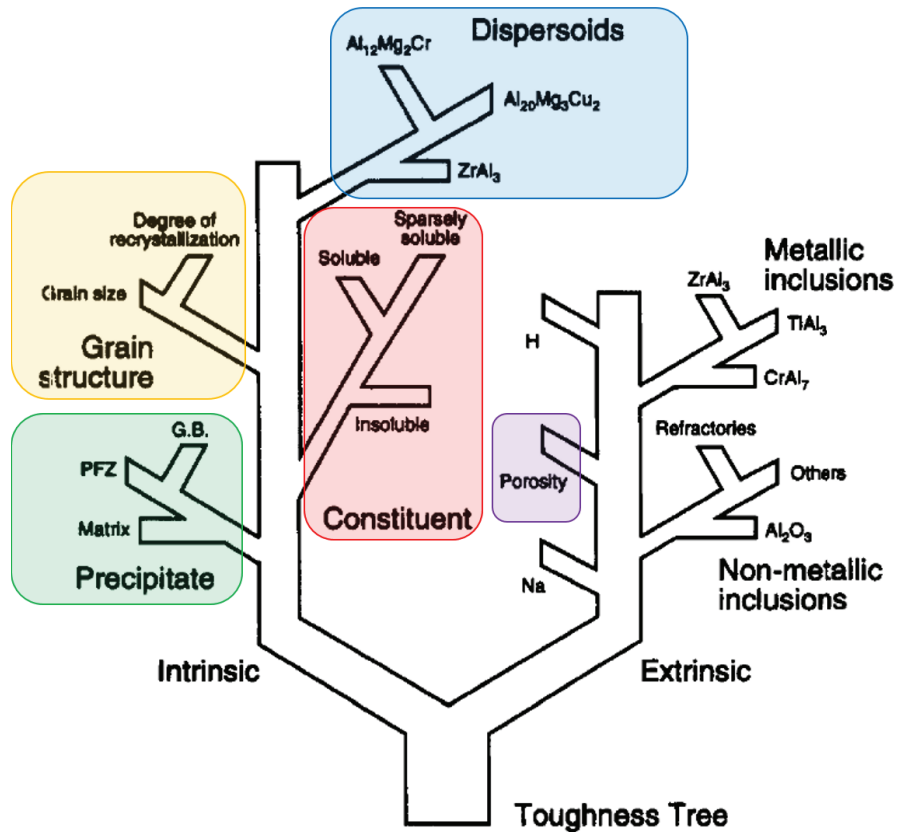


FIGURE 1.25: Toughness tree representation of microstructural features that affect toughness in age-hardenable aluminium alloys [115].

is usually achieved by deformation processing. However, some micropores remain even after large plastic deformation [118] and micropores have been observed by X-ray microtomography in many wrought aluminium alloys with volume fractions of the order of a few tenths of one percent and for an average micropore size typically of a few μm [119], which corresponds to a relatively high density of pores ranging between 1000 up to 10^6 per 1 mm^3 . Nevertheless, the density of micropores observed in conventional cross-sectional observations is usually quite low because they easily fill with the abrasion powders when samples are cut and polished [117].

Large constituent particles affect the fracture toughness of alu-

minium alloys through the void nucleation, growth and coalescence processes, as extensively discussed in section 1.1. The main inclusions in aluminium alloys detrimental to fracture toughness are principally intermetallic particles formed from iron and silicon impurities. A variety of brittle intermetallic particles of the form $Fe_xSi_yAl_z$ may be present, typically ranging between 1 and 10 μm and generally form in the melt. They often have angular form and, in the larger sizes, could be polycrystalline [113]. The 6xxx serie aluminium alloys contain a volume fraction of various intermetallic particles typically around 1%. The most typical are the plate-like $\beta - Al_5FeSi$ particles which can be transformed into the more rounded meta-stable cubic rounded $\alpha - Al_{12}(Fe, Mn)_3Si$ particles by a long homogenization heat treatment [40].

Dispersoids are intermetallic particles, typically ranging between 0.02 and 0.5 μm , introduced deliberately in the matrix to achieve grain-size control during solution treatment. Sometimes, undesired dispersoids particles may also arise from the interaction of minor alloying additions or impurities in the solid state [113]. Depending on their size and on their interface with the matrix, a second population of void nucleated around dispersoids may also play an unfavourable role in the fracture process. Specifically, coalescence of voids nucleated by fracture or decohesion of large constituent particles occurs then due to the formation of void sheets associated with the nucleation of fine voids at dispersoids (see section 1.1.3.3)

Strengthening precipitates increase the resistance of the matrix to plastic deformation, i.e. increase the yield strength of the alloy. In general, precipitates do not directly take part in the failure process. However, they have an indirect influence on the resistance to fracture. From a macroscopic point of view and if all other parameters, including strain hardening, could be kept constant, then ageing leading to a higher yield stress (σ_0) would increase the fracture toughness [24]. However, an increase of σ_0 is accompanied by a decrease of the strain-hardening capacity in age-hardenable aluminium alloys which has the opposite effect on toughness. From a microscopic point of view, precipitation-hardening alloys may exhibit heterogeneous precipitation of coarse phases at grain boundaries. As a result, the formation of a fine dispersion of precipitates is inhibited in the grain boundary environment [120]. These precipitate free zones (PFZ) remain very

1.3. Aluminium alloys

soft while the interior of the grains is highly hardened. The combination of these soft zones, which promote strain localization, and grain boundary precipitates, which may promote void nucleation, may result in intergranular fracture mode decreasing the fracture toughness of the material [121].

The grain structure, i.e. the grain size and the degree of recrystallization may also have a significant effect on the fracture toughness. The fracture of thin metallic sheets, under plane stress, is associated with a localized necking zone in the near-crack-tip region [24]. Fracture is thus more controlled by plasticity and a small recrystallized grain size is preferable [113]. On the other hand, for fracture of thick plates under plane strain, an unrecrystallized grain structure is preferable because fracture is usually controlled by void nucleation, growth and coalescence from coarse particles [113]. Furthermore, grain boundaries are dislocations barriers and a smaller grain size is usually associated with a higher yield strength (Hall–Petch effect). However, the contribution of the grain size to the strengthening is negligible compared with the effect of the solute and small precipitates in heat treatable aluminum alloys [122, 123].

1.4 Friction stir processing (FSP)

Friction stir welding (FSW) is a solid-state welding process developed to bypass limitations associated with local melting and solidification involved in most conventional welding processes. It is particularly suitable for high strength Al alloys [124, 125]. Friction stir processing (FSP) is a process derived from FSW, involving a non-consumable tool with complex features to locally modify the microstructure through solid state mixing. The rotating tool (see Fig. 1.26(a)) penetrates the material until the shoulder gets into contact with the upper surface (see Fig. 1.26(b)) and is then displaced along the surface at a controlled velocity and rotation speed (see Fig. 1.26(c)).

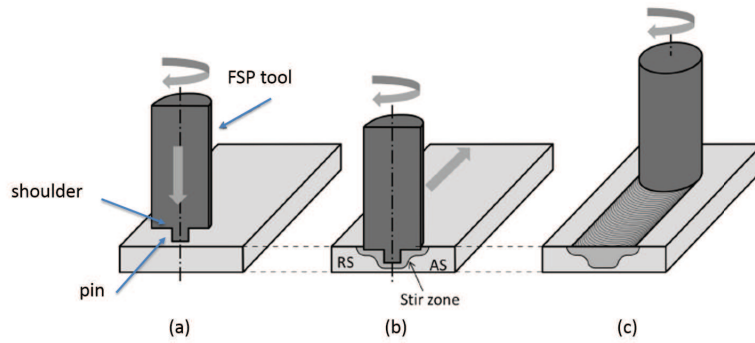


FIGURE 1.26: A schematic drawing of friction stir processing. Courtesy of N.J. Mena.

The development of friction stir processing as a generic metallurgical tool for microstructural modification and a broader manufacturing technology is connected to the material flow and microstructural evolution [126]. An overview of the microstructural evolutions due to FSP and their link to various emerging FSP-based technologies is shown in Fig. 1.27. More details will be given in section 3.1.

1.4. Friction stir processing (FSP)

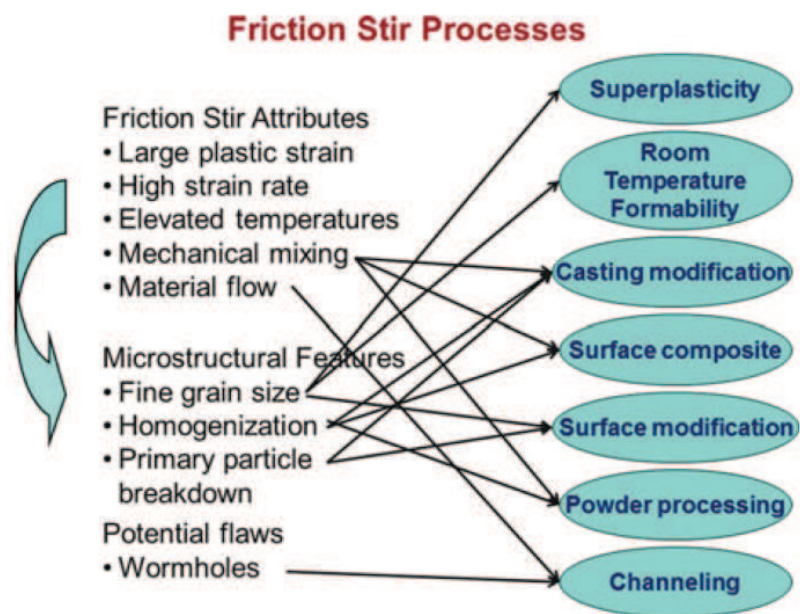


FIGURE 1.27: An overview of the evolution of microstructural features and its linkage to various emerging FSP technologies. Reproduced from Ref.[126].

Chapter 2

Characterization and micromechanical modelling of microstructural heterogeneity effects on ductile fracture of 6xxx aluminium alloys

This chapter is based on the published article *Characterization and micromechanical modelling of microstructural heterogeneity effects on ductile fracture of 6xxx aluminium alloys*, F. Hannard, T. Pardoen, E. Maire, C. Le Bourlot, R. Mokso, A. Simar, *Acta Materialia* 103(2016) 558-572.

2.1 Introduction

Ductile fracture results from the nucleation, growth and coalescence of small internal cavities. Microvoid nucleation is usually associated with the fracture of brittle particles or interface decohesion [29, 33]. After nucleation, voids grow by plastic deformation of the surrounding matrix [25]. During the last four decades, ductile fracture has been a subject of intense investigation in the solid mechanics and material science communities [25]. The first micromechanical treatments due to McClintock [127] and Rice and Tracey [64] considered isolated voids. Stable growth proceeds until plasticity localizes within the ligament between closely spaced cavities setting the onset of the coalescence mechanism [65]. The accumulation of void linkages leads to the formation of a dominant macroscopic crack that finally propagates into the entire material. The understanding and modelling of the different steps of the process of nucleation, growth, and coalescence of voids have been enhanced over the years, as covered in several recent review papers [25, 24, 7]. However, major questions remain about the understanding and proper modelling of ductile fracture in metals involving significant microstructure heterogeneities. This introduction proceeds with a review of the main issues related to heterogeneities and how they affect damage evolution and complexify the application of existing models.

Void nucleation is inherently a discontinuous process made of a succession of discrete nucleation events [24]. The effect of particle size, associated with the distribution of internal or interfacial defects, can be a dominant feature of the inhomogeneity of the damage process. Void nucleation becomes increasingly difficult with decreasing particle size since the largest defects are more likely to be found in the largest inclusions. The particle shape combined to the loading orientation can also contribute to the heterogeneity of the nucleation process [34, 40]. Furthermore, the interfacial resistance between second phase particles and surrounding matrix is dependent on the local chemical composition [24]. Nucleation can be inhomogeneous also because the load transfer in the particles depends on local microplasticity effects related to microstructure details and local crystallography, see e.g. [47]. Heterogeneities in the spatial distribution of the second phases which can be spatially clustered lead to local stress concentrations [48]. All these effects could in principle be rationalized using micromechanics

based nucleation criteria. The Beremin group proposed a void nucleation model valid for a range of particle sizes formulated within a continuum approach [34], based on a critical value of maximum principal stress in the particle or at the interface. The model accounts for plastic strain incompatibility between the matrix and inclusion and for particle shape. The effects of inclusion orientation and of complex loading path on nucleation have been addressed by finite element (FE) simulations on complex multiparticle cells [41].

Voids nucleated by particle fracture are initially flat and become more rounded with plastic deformation. Voids nucleated by interface decohesion are initially rounded and tend to elongate in the principal loading direction under low stress triaxiality. Heterogeneities in the void distribution do not significantly affect the void growth rates [128, 129, 130, 5] and the interaction between voids during the growth phase is usually weak in industrial alloys (i.e. with low volume fraction of constituent particles). However, these interactions become significant in void clusters of highly heterogeneous materials (e.g., [131]). For instance, small voids in the neighbourhood of large cavities grow much faster than in a single size population [23, 22]. The void growth rate depends also on the local crystallographic orientation [132]. Aside from the simple analytical models for isolated voids, constitutive models for porous plastic materials were developed based on homogenization theory but without interaction effects, see in particular the Gurson model [3] and a realm of its extensions [83, 5, 133, 134, 25, 135, 136, 137, 138, 40, 88, 139].

The dominant parameter controlling the transition from stable growth to coalescence is the relative void spacing which can be highly heterogeneous if particles are clustered. If coalescence proceeds after significant amount of void growth, the failure is said "void controlled". When nucleation is rapidly followed by void coalescence with limited amount of void growth, the fracture mechanism is said to be "nucleation controlled" such as observed for instance in high strength Al alloys [21]. Significant progress has been made over the past two decades in the micromechanical modeling of coalescence, mainly along the lines of the approach developed by Thomason [65] with several generalizations [5, 22, 140, 25, 141, 142, 143, 144]. The proximity within clusters of voids, see e.g. [130], has a huge effect on coalescence because the dominant parameter is the relative void spacing.

2.1. Introduction

The impact of heterogeneities on damage accumulation until cracking can be modelled in many different ways, see review [7]. One approach consists in averaging the response on large RVE through statistical treatment of void nucleation. This statistical treatment can be connected to the Beremin model [34] based on a weakest link approach through a Weibull distribution for particle fracture stress [145] or based on more simple distributions, see [146, 60]. A mean average porosity is assumed for the void growth phase and coalescence is treated by accounting for a nearest neighbours void spacing [147]. More advanced approaches consist in representing the microstructure and property heterogeneities in a systematic way. For instance, damage percolation models have been developed to capture cluster effects on coalescence and cracking [21, 8]. Heterogeneous damage nucleation can also be taken into account in FE calculations with different void nucleation rates in each element [148]. Probably the most sophisticated approach is to generate a large ensemble of particles, following physical distribution, and to simulate all steps of the damage process without the need for approximate nucleation, growth and coalescence laws [149].

From an experimental view point, 3D X-ray tomography is perfectly suited to study the effect of microstructure heterogeneity on all steps of ductile failure. It allows non-destructive local characterization of the microstructure and quantitative analysis of the morphology of the particles associated to void nucleation [145]. The results of in situ tensile tests in X-ray tomography have lead to a quantitative characterization of the initiation [150], growth [73] and coalescence [151] in high strength steels, Al and Ti alloys.

The goal of the present study is to understand and model the effect of microstructure heterogeneities on damage accumulation in three 6xxx series Al alloys, but with a methodology that can be directly extended to any other metallic alloy. These alloys, i.e. Al 6005A, Al 6061 and Al 6056, exhibit a volume fraction of iron rich particles close to 1 %. However, the variation of fracture strain as a function of yield stress shown in Fig. 2.1.a (and explained further in this paper) is very different, involving a factor 10 in the reduction of area between the extreme cases (i.e. factor 3 in true "log" fracture strain). In a preliminary study [152], a recent advanced extension of Gurson's model for porous ductile metals has been applied in order to reproduce this effect. However, because of the very small difference in volume fraction of Fe based

particles, the model was not rich enough to allow capturing the differences between the three alloys. This negative outcome motivated the formulation of a physical model which does not rely on an average description of the microstructure. Furthermore, damage models are rarely challenged in applications involving wide datasets encompassing several materials and heat treatments. This is the purpose of the paper to describe the key experimental results regarding damage evolution motivating the need for a model that accounts for microstructural heterogeneities.

The outline of the paper is the following. Section 2.2 introduces the experimental methods used for the microstructural and mechanical characterization of the three alloys. The damage model is described in section 2.3. Subsequently, section 2.4 starts with the experimental results which are used for the identification of the damage model. The model is then validated by comparison with additional experimental data.

2.2 Experimental methods

2.2.1 Materials

The fracture of 6005A, 6061 and 6056 Al alloy, is investigated under a wide range of heat treatment conditions in order to allow cross-comparisons at identical yield strength. Table 2.1 presents the chemical composition as measured by inductively coupled plasma – optical emission spectroscopy. The alloys are provided in extruded plates (6005A alloy) or cold rolled plates (6061 and 6056 alloys), the rolling or extrusion directions are referred as the "primary" processing direction. The thickness of the plates is equal to 6 mm for the 6005A and 6056 alloys and 4.7 mm for the 6061 alloy.

The alloys are initially in T4 (6056 alloy) or T6 conditions (6005A and 6061 alloys). Some plates provided in T6 condition are transformed into T4 condition by a solution heat treatment of 1 hour at 535°C for the 6061 alloy and at 530 °C for the 6005A alloy. Heat treatments at 180°C are performed on samples in the T4 state. Some 6056 plates in T4

2.2. Experimental methods

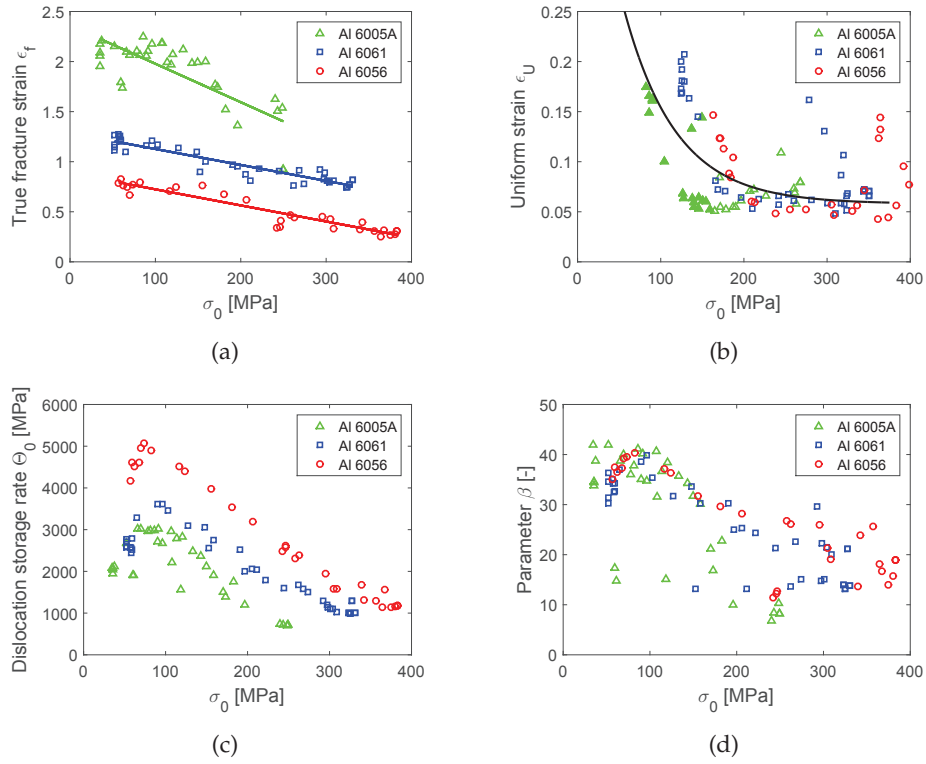


FIGURE 2.1: Variation of the different parameters extracted from the uniaxial tensile tests on the three alloys as a function of the yield stress corresponding to the different heat treatments. (a) True fracture strain of three Al alloys; (b) true strain at the onset of necking; (c) dislocation storage rate θ_0 of the Voce law; (d) dynamic recovery parameter β .

Chap. 2: Ductile fracture of 6xxx aluminium alloys

<i>Alloy</i>	<i>Mg</i>	<i>Si</i>	<i>Cu</i>	<i>Fe</i>	<i>Mn</i>	<i>Cr</i>	<i>Zn</i>	<i>Ti</i>	<i>Al</i>
<i>Al 6005A</i>	0.47	0.60	0.09	0.23	0.11	< 0.01	0.07	0.02	98.73
<i>Al 6061</i>	0.88	0.58	0.23	0.39	0.05	0.18	0.03	0.02	98.44
<i>Al 6056</i>	0.75	0.95	0.64	0.11	0.61	< 0.01	0.18	0.01	97.5

TABLE 2.1: Chemical composition measured by ICP-OES of the 6005A, 6061 and 6056 Al alloys [*wt.%*] Al content is the balance.

state are transformed into T6 by a heat treatment at 180°C for 8 hours. Heat treatments at 300°C and 350°C for various times ranging from 10 seconds to 20 days are performed on samples initially in the T6 state. Heat treatments are systematically followed by water-quenching and natural aging for at least one week before further artificial aging.

2.2.2 Microstructure characterization of base alloys

After standard polishing, the intermetallics and dispersoids particles are characterized using a Field Emission Gun Scanning Electron Microscope (FEG-SEM) operated at 15 kV under electron backscattered mode. X-ray microtomography are performed at the TOMCAT beam-line of the Swiss Light Source [153]. The sample is probed by X-ray beam tuned to 20 keV using a double crystal multilayer monochromator. 1800 projections are recorded in absorption mode using the 16 bit PCO.edges CMOS detector coupled with a 20X optical magnifying lens to a 20 micrometer thick LuAG:Ce scintillator. The observed region is a cube of 260 μm side length with an isotropic voxel size of 325 x 325 x 325 nm³.

2.2.3 Characterization of mechanical properties and damage

2.2.3.1 Macroscopic tensile tests

Uniaxial tensile tests are performed on an universal Zwick 50 kN testing machine under displacement control at constant velocity (1 mm/min). The cylindrical samples, 4 mm in diameter, are extracted perpendicular to the primary direction. The initial gauge length is

2.2. Experimental methods

equal to 30 mm. The initial yield stress σ_0 and the strain hardening parameters are extracted from the stress-strain curves based on an extended Voce law, i.e. $\partial\sigma/\partial\epsilon_p = \Theta_0 - \beta\sigma + \Theta_{IV}$, where σ is the true stress and ϵ_p is the true plastic strain. The strain hardening parameters are the dislocation storage rate Θ_0 , the dynamic recovery rate β and the stage IV hardening Θ_{IV} following the extended Voce law, see [152]. The strain hardening exponent at necking is defined as $n_{necking} = \frac{\epsilon}{\sigma} \frac{\partial\sigma}{\partial\epsilon} \big|_{\epsilon=\epsilon_u}$, where ϵ_u is the true strain at necking. The local true strain is defined as $\epsilon_{loc} = \ln\left(\frac{A_0}{A}\right)$, where A_0 and A are the initial and current cross-section area, respectively. The true fracture strain is thus defined as $\epsilon_f = \ln\left(\frac{A_0}{A_f}\right)$, where A_f is the final cross-section area.

2.2.3.2 Metallographic observations on broken samples

Three broken tensile samples per alloy are polished down to mid-thickness along transverse section near the fracture surface: one in T4 state, one in T6 state and one in overaged state. FEG-SEM images at a magnification of 1500x are taken along a line perpendicular to the fracture surface every 0.2 mm, see Fig. 2.2. Particle fracture is the dominant nucleation mechanism with almost no interface decohesion. The surface and best fitting ellipse is recorded for every unbroken particle. If the particle is broken, it is reconstructed by merging all fragments and the best fitting ellipse and surface is recorded. About 10,000 particles have been analysed.

2.2.3.3 In-situ tensile testing in X-ray microtomography

A dedicated in-situ tensile testing machine described in [154] has been mounted on the rotation stage of the 3D tomography setup [155] of the ID15A beam line at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The stage rotates at a constant speed while the tensile test is continuously performed at a displacement rate equal to $2 \cdot 10^{-7} \text{m.s}^{-1}$ for the 6061 and 6056 alloys and at $5 \cdot 10^{-4} \text{m.s}^{-1}$ for the 6005A alloy. The tensile force F is recorded. Three mini notched tensile specimens, with yield stress close to 120 MPa, outer diameter of 3 mm,

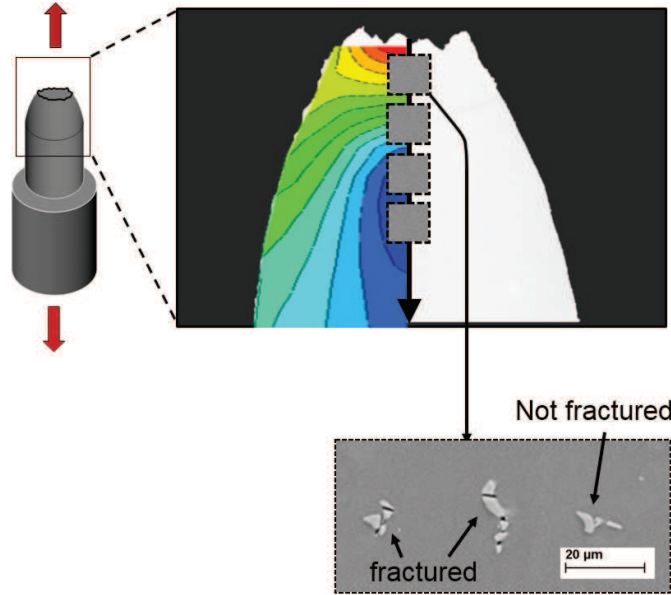


FIGURE 2.2: Schematic representation of the method used to analyse the probability of fracture of intermetallic iron-rich particles at various distances from the fracture surface of broken tensile samples polished down to mid-thickness; for each particle, record position, dimensions and broken or not broken (here a 6056 alloy specimen), micrograph obtained by FEG-SEM. The color map shows the finite element simulation predictions of the tensile test.

reduced diameter of 1 mm and notch radius of 1 mm are deformed up to fracture.

The data acquisition is made at regular discrete time steps, with a voxel size of $1.06\mu\text{m}^3$. The procedure used to separate the voids from the Al matrix is described in [73]. Only the central zone of the specimen is selected for the quantitative analysis of damage because it undergoes the largest stress triaxiality and thus the fastest damage rates [155]. A cube of $200\mu\text{m} \times 200\mu\text{m} \times 200\mu\text{m}$ is analyzed which is large enough to involve a sufficient number of voids and to be representative of the damage process, see further.

For each scanning step, the voids are distinguished from the Al matrix and the geometrical characteristics are recorded. A tracking procedure follows the cavities from one step to the next. The results of the tracking carried out on the Al 6056 specimen provides the experimen-

2.3. Cellular automaton micromechanics based damage model

tal void growth rate. Experimental data were analysed by automatic tracking following the algorithm in [74], and double-checked by manual tracking. Only the cavities tracked for a minimum of 8 consecutive steps are used. The cavities leaving the analyzed box during straining are not considered. About one hundred tracks have been analyzed.

2.3 Cellular automaton micromechanics based damage model

2.3.1 Synopsis of the model

A cellular automaton model has been developed based on an initial distribution of n particles described by their position, dimensions (and thus shape) and orientation in a volume of material. A nucleation stress is associated to each particle following a distribution law which can be identified experimentally or predicted theoretically. The material is deformed under a prescribed stress and strain history which can be homogeneous or not at the scale of the volume. Nucleation, growth and coalescence of voids are described using analytical micromechanical models presented in sections 2.3.2 to 2.3.4. The model assumes no interactions between particles and/or voids during the nucleation and growth stages. The presence of the voids do not affect the applied stress and strain. At each deformation step, the nucleation, growth and coalescence of cavities are updated until a final failure criterion is verified (see section 2.3.5). The material is modeled using isotropic J_2 flow theory. Since the volume fraction of second phase particles f_p is close to 1 %, the reinforcement effect of these particles is weak as well as the loss of strength resulting from particle fracture. The matrix stress state is thus accurately described with the strain hardening parameters identified on the overall experimental flow stress (see section 2.2.3.1).

2.3.2 Void nucleation model

Stress within the particle

Particle fracture depends on the stress transfer in the particle and on the interface which itself depends on microstructural parameters such as particle size, shape and relative orientation to the loading direction [34]. Void nucleation via interface decohesion is not taken into account as particle fracture is the dominant failure mechanism [152], see also later.

The stress in the particle is decomposed as the sum of the stress in the matrix and of an additional stress transfer arising from the deformation mismatch between particle and matrix, which is calculated using Eshelby's theory with a plastic equivalent modulus for the matrix [36] as in the Beremin approach [34]. Hill [156] developed a self-consistent scheme for the problem of an ellipsoidal elastoplastic inclusion embedded in an infinite elastoplastic matrix. The elastoplastic behavior of the matrix is described by the effective moduli L^{eff} which links the macroscopic stress rates $\dot{\Sigma}$ and the macroscopic strain rates $\dot{\epsilon}_{macro}$ as

$$\dot{\Sigma} = L^{eff} : \dot{\epsilon}_{macro} . \quad (2.1)$$

The homogeneous stress rate in the inclusion $\dot{\sigma}$ is then written as

$$\dot{\sigma} = \dot{\Sigma} + L^{eff} : (S^{-1} - I) : (\dot{\epsilon}_{macro} - \dot{\epsilon}_{inc}) \quad (2.2)$$

where S is Eshelby's tensor and $\dot{\epsilon}_{inc}$ is the strain rate in the inclusion. The isotropic approximation of the effective moduli proposed by Berveiller and Zaoui [36] with the isotropic approximation of the effective moduli called L' writes

$$L'_{ijkl} = \mu^* \left[\frac{2\nu^*}{1 - 2\nu^*} \delta_{ij} \delta_{kl} + \delta_{ik} \delta_{jl} - \delta_{il} \delta_{jk} \right] \quad (2.3)$$

where δ_{ij} is the Dirac delta function. The expression for the secant "elasto-plastic" shear modulus μ^* and Poisson ratio ν^* are given in [36].

Introducing these secant elasto-plastic moduli into equation (2.2) gives the resulting interaction law relating finite quantities instead of

2.3. Cellular automaton micromechanics based damage model

their rates :

$$\sigma = \Sigma + L' : (S^{-1} - I) : (\epsilon_{macro} - \epsilon_{inc}) . \quad (2.4)$$

Void nucleation model

Particles are treated as brittle solids and fracture is assumed to occur when the maximum principal stress σ_{part}^{max} (eq.(2.4)) reaches the critical stress σ_c^i associated to this particle. The particle fracture stress distribution is assumed to follow a Weibull statistics given by

$$P(\sigma_{part}^{max}, V_{part}) = 1 - \exp \left(- \frac{V_{part}}{V_0} \left(\frac{\sigma_{part}^{max}}{\sigma_w} \right)^m \right) \quad (2.5)$$

where P is the probability of particle fracture for a given σ_{part}^{max} and volume of particle V_{part} , see Babout *et al.* [145] for example; V_0 is the reference particle volume and σ_w and m are the parameters of the distribution.

The nucleated void is assumed to be penny-shaped and its location is given by the intersection of the particle with a plane perpendicular to the maximum principal stress direction, see Fig. 2.3(a). The newly nucleated void is assumed to be ellipsoidal with main axis a, b and c . The crack is assumed to appear at the middle of the particle. The initial void aspect ratio W_0 is set equal to 0.01 (with $c = W_0 \sqrt{ab}$, see Fig. 2.3(a)). Lassance *et al.* [76] have shown that penny-shaped voids rapidly open and that the predicted void growth rate is independent of the choice of the initial porosity when imposing a value for W_0 sufficiently small.

2.3.3 Void growth model

The Rice and Tracey (R&T) void growth model [64] has been selected. The original version was developed for a spherical void in an infinite, rigid perfectly plastic material under uniform remote strain ϵ_{ij} and stress σ_{ij} . The volume change is a function of the hydrostatic stress only while the change of shape is dictated by the deviatoric stress component. The strain rate field is expressed in terms of the principal components $\dot{\epsilon}_1 \geq \dot{\epsilon}_2 \geq \dot{\epsilon}_3$. A version of the model taking into account the aspect ratio evolution elaborated by Thomason [65] links the rate of

change of the radii in the three directions of space (R_i , $i=1,2,3$) to the magnitude of $R_{mean} = \frac{R_1+R_2+R_3}{3}$:

$$\dot{R}_k = \left((1 + E)\dot{\epsilon}_k + \sqrt{\frac{2}{3}}\dot{\epsilon}_I\dot{\epsilon}_I D \right) R_{mean} \quad (2.6)$$

where D is given by $D = \alpha T^{0.25} \exp(\frac{3}{2}T)$ for $T \leq 1$, and $D = \alpha \exp(\frac{3}{2}T)$ for $T \geq 1$, with the stress triaxiality T and α equal to 0.427 as corrected by Huang [66]. Here α can be taken as a free parameter that can be adjusted to account for the approximations in the model such as the absence of strain hardening. The factor D characterizes the dilatational contribution and $(1 + E)$ characterizes the change of aspect ratio. The factor $(1 + E)$ is fixed and equal to $\frac{5}{3}$ as initially predicted by Rice and Tracey [64], see [67] for a more elaborated expression not used in this study.

This void growth model has been initially developed for voids with the main axes aligned with the principal loading directions ($\Sigma_1, \Sigma_2, \Sigma_3$). An heuristic procedure is proposed in order to estimate the void growth rates in the case of an ellipsoidal void under general loading conditions. The principal radii of the ellipsoidal void are replaced by the three intersections of the void with the principal stress directions. This equivalent void is then assumed to follow the growth model given by equation (2.6), see Fig. 2.3(b). The void growth procedure is detailed in the Appendix.

Since voids nucleate by particle cracking rather than by decohesion, a freshly nucleated void is initially penny-shaped and the mode of deformation is dominated by opening and blunting of the void/crack tip [75]. At low T , the growth model given by equation (2.6) predicts a decrease of the void size in the transverse directions Σ_2 and Σ_3 while, in reality, the particle constraints the transverse contraction same as for the case where the particle is inside the cavity as analysed by [76]. A slight modification of the model has thus been made. The term controlling the transverse contraction due to the shape changing component of the remotely applied deformation field is deleted for the transverse directions ($1 + E = 0$ for $\dot{R}_2, \dot{R}_3$). This version of the model will be referred next as the "constraint" growth model.

2.3.4 Void coalescence model

The void coalescence condition developed by Thomason is used in its extended form [65, 5, 88]:

$$\frac{\Sigma_n}{\sigma_y} = (1 - \chi_{eff}^2) \left[\alpha_{Th} \left(\frac{1 - \chi_{eff}}{W_{eff} \chi_{eff}} \right)^2 + 1.24 \frac{1}{\sqrt{\chi_{eff}}} \right] \quad (2.7)$$

where Σ_n is the overall stress component normal to the localization plane; χ_{eff} and W_{eff} are the effective void aspect ratio and effective void spacing respectively defined in eq. (2.8) and (2.9), see later. The parameter α_{Th} depends on the strain hardening exponent n as proposed by Pardoen and Hutchinson [5] : $\alpha_{Th} = 0.1 + 0.217n + 4.83n^2$.

The coalescence model has been developed for identical neighboring voids having the ligament joining their centers normal to the principal loading direction Σ_1 . This is not representative of an actual microstructure. A generalization has been proposed by Scheyvaerts *et al* [88] in order to account for the orientation of the inter-void ligament. The equivalent maximum in-plane tensile stress Σ_{Plane}^1 is found using a simple procedure detailed in [157]. To apply this procedure, the two voids are transformed into an effective configuration made of two identical voids (Fig. 2.3 (c)) in order to define W_{eff} and χ_{eff} .

The later is determined from the center-to-center distance L_{center} and $R_{center,1}$ and $R_{center,2}$ the radii measured along the center-to-center direction (Fig. 2.3 (c)) as

$$\chi_{eff} = \frac{R_{center,1} + R_{center,2}}{L_{center}}. \quad (2.8)$$

The effective void aspect ratio W_{eff} is taken as a mean of the aspect ratio of the two neighboring voids and is defined as :

$$W_{eff} = \frac{1}{2} \left(\frac{R_{perp,1}}{\sqrt{a_1 b_1}} + \frac{R_{perp,2}}{\sqrt{a_2 b_2}} \right) \quad (2.9)$$

where (a_1, b_1) and (a_2, b_2) are the half-axes of the two ellipses resulting from the cross section of each void taken in the transverse direction to the in-plane stress direction. The equivalent voids resulting from this procedure are shown in Fig. 2.3(d), where $R_{center} = \chi_{eff} \frac{L_{center}}{2}$ and R_{perp}

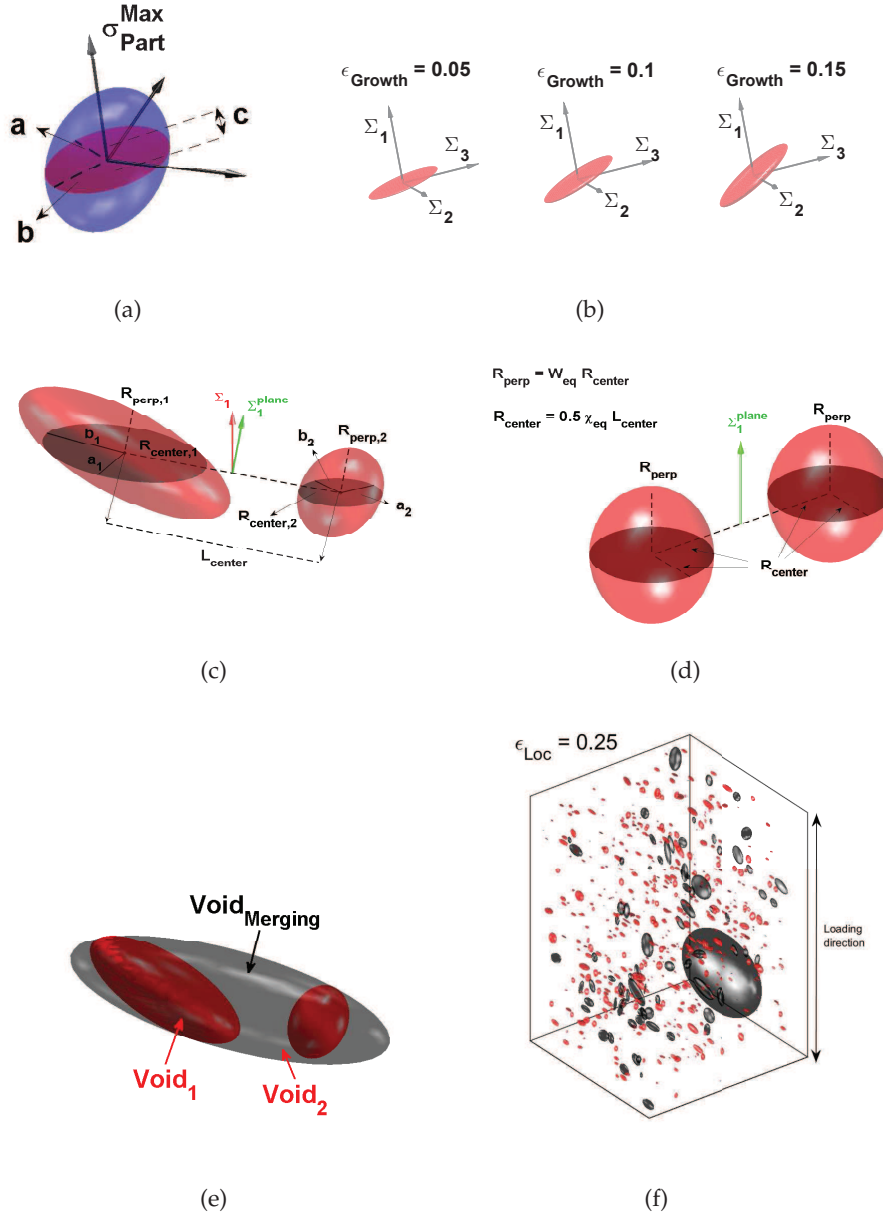


FIGURE 2.3: (a) Void nucleation on particle. (b) Result of the growth model at different $\epsilon_{Growth} = \epsilon_{Loc} - \epsilon_{Nucleation}$. (c) Definition of the effective parameters used to apply the coalescence criterion. (d) Equivalent geometry. (e) Result of the merging process: minimum ellipsoid that envelops both coalescing voids. (f) Example of a whole RVE at a local strain $\epsilon = 0.25$. (a – f) The particle is in blue, the voids in red and the coalesced voids in grey.

2.3. Cellular automaton micromechanics based damage model

$= W_{eff} R_{center}$. Furthermore, the minimum distance between each void is checked to be larger than zero in order to ensure no overlapping. This heuristic extension has been partly assessed by [88], although only for spheroids of identical dimension. This extension is certainly an open area for improvement. The local displacements of the centroid of the particles and cavities during a strain increment are calculated by interpolation of the FE displacement field.

Once the coalescence criterion is satisfied for two voids, failure of the inter-void ligament is modelled by merging the two voids and replacing them by the minimum ellipsoid that envelops them both (Fig. 2.3 (e)). In this case, the axis of the cavity can be significantly out of the principal loading direction justifying the correction for non aligned ellipsoid (see Appendix). The coalescence criterion is then checked for this new void and each neighboring void, allowing the coalescence process to locally flourish. This procedure neglects the small additional strain needed to bring the two voids from the onset of coalescence to final coalescence (see [143] for discussion of details). More advanced versions of a coalescence model accounting for the shear component along the localization plane [142] will be considered in future extensions.

2.3.5 Final failure criterion

Several authors [158, 72, 5] have shown the inadequacy of models based on a critical porosity value to predict the strain to failure. Indeed, the porosity at coalescence is found to depend on the initial void volume fraction f_0 , the flow properties and stress state and is thus not a "universal" value. The onset of coalescence in a small material volume as predicted by eq.(2.7) is not a sufficient condition for fracture in the case of heterogeneous materials. Thomson et al. [130] have shown that coalescence of voids within a cluster consisting of a few particles is a stable event while the collapse of inter-cluster ligaments leads to imminent material failure. This inter-cluster ligaments collapse is associated to a significant and sudden increase of the porosity (i.e. a large rate of porosity increase). An empirical failure criterion has thus been chosen here to be a critical rate of porosity increase $\left. \frac{\partial f}{\partial \epsilon^p} \right|_c$. The physical meaning of this critical rate of porosity increase is associated to voids linkage

over a macroscopic scale that triggers catastrophic ductile failure. In a more sophisticated model, crack propagation from the most critical coalesced patch of voids would be the most correct approach but this goes out of the scope of the current state of this work.

2.3.6 Summary of the parameters of the model

The following set of parameters is needed to run a simulation with the cellular automaton model (see Fig. 2.4). The microstructural parameters, i.e. particles and initial cavities parameters¹ :

- the positions x_i, y_i, z_i ;
- the dimensions $R_{x,i}, R_{y,i}, R_{z,i}$;
- the three angles which define the orientation in space $\theta_{1,i}, \theta_{2,i}, \theta_{3,i}$.

The flow properties of the matrix :

- the initial yield stress σ_0 ;
- the extended Voce law parameters : $\Theta, \beta, \Theta_{IV}$.

The damage model parameters :

- the Weibull distribution parameters² : V_0, σ_w, m ;
- the α parameter of the *R&T* model which is experimentally measured to correct for the limitations of the model : α_{growth} ;
- the critical rate of porosity increase $\left. \frac{\partial f}{\partial \epsilon^p} \right|_c$.

2.3.7 Numerical procedures

A multi-scale approach combines FE simulations and the use of the cellular automaton model at a local scale, see Fig. 2.4. FE simulations of the tensile specimens are conducted with the general purpose software Abaqus [159] using J_2 flow theory and hardening law prescribed in section 2.2.3.1. The aim is to transfer to the cellular automaton the local stress and strain variations accounting here for specimen necking. The FE procedures are fully standard and convergence of the results was carefully checked. A Matlab [160] program has been written for the cellular automaton (CA). For each deformation step, one

2.4. Results and discussion

iteration of the CA is performed, see Fig. 2.4. Fig. 2.3(f) shows the full representative volume element (RVE) used in the cellular automaton for a local strain $\varepsilon_{loc} = 0.25$. It appears from this figure that the size of the RVE must be large enough to capture the local variation of spatial distribution of voids and particles inducing local coalescence processes (voids in grey in Fig. 2.3(f)) and to enable damage percolation over a macroscopic scale that triggers catastrophic ductile failure.

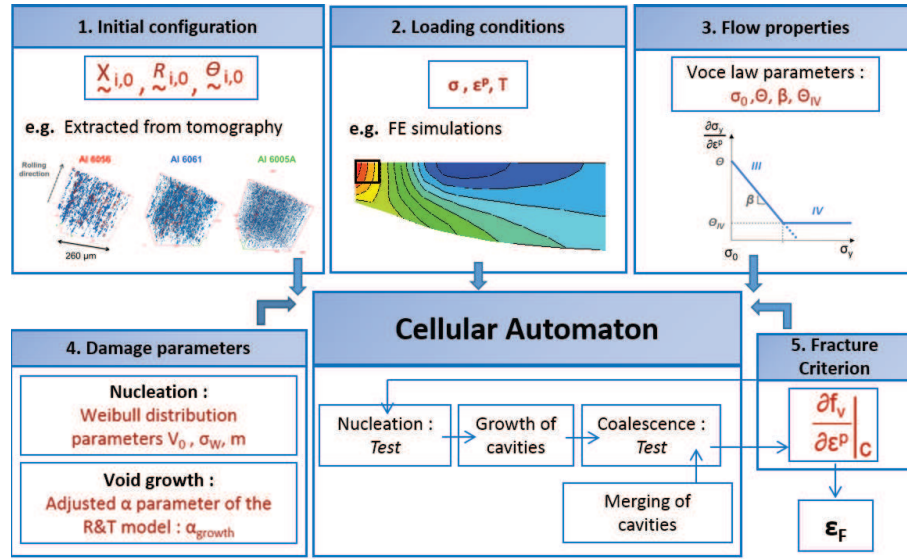


FIGURE 2.4: Overview of the modelling strategy

2.4 Results and discussion

2.4.1 Microstructure and mechanical properties

The reconstructed X-ray microtomography volume of the three alloys in their initial states (as observed with $0.3 \mu\text{m}$ voxel size microtomography at the TOMCAT beamline in SLS) are illustrated in Fig. 2.5. Particles and initial porosities are respectively highlighted in blue and

¹Identified from X-ray microtomography data of the base alloys (section 2.2.2)

²Identified in order to satisfy the Weibull distribution (section 2.2.3.2)

red. Fig. 2.5(b,c) presents the size distribution for the intermetallic particles based on SEM micrographs and on 3D X-ray microtomography images. The equivalent diameter D_{eq} corresponds to the diameter of a sphere with the same volume as the particle. Table 2.2 gives the corresponding averaged parameters. The differences between the two characterization techniques are probably due to the difference of resolution. The SEM micrographs were taken at a magnification of 2000X allowing a statistical analysis of particles larger than about 500 nm. The X-ray microtomography images have a voxel size of 325x325x325 nm³ (section 2.2.2) allowing to reliably identify particles larger than about 700 nm (i.e. 2 voxels). Indeed, it will be shown subsequently (section 2.4.2.1, Fig. 2.7) that the particles that will actually lead to damage are essentially the largest one ($D_{eq} > 1 \mu m$). Thus, the exact identification (size, position, number density) of the smallest particles is of secondary importance for the prediction of the fracture strain by the cellular automaton model. Furthermore, the SEM micrographs allow the observation of the near surface of the polished sample only. As the particles are not necessarily intersected close to the equator, the value of the equivalent diameter measured in the plane section is smaller than the actual 3D value and the 2D equivalent diameter has thus been multiplied by a stereological factor $\frac{4}{\pi}$. Globally, Fig. 2.5(b,c) shows that the trends remain in good agreement, irrelevant of the characterization technique. In particular Table 2.2 indicates that the particle volume fraction of all three alloys is close to 1 % (the value for the 6005A is underestimated due to the small size of the particles in that alloy). All alloys have a particle size shape factor close to 2 that is measured as slightly lower by SEM, but 2D imaging is not ideal for the identification of this parameter. The mean equivalent diameter is $\sim 1 \mu m$ for the 6005A alloy, $\sim 2 \mu m$ for the 6061 alloy and slightly above $2 \mu m$ for the 6056 alloy which includes a high fraction of very large particles ($D_{eq} > 5 \mu m$) as demonstrated in the inset of Fig. 2.5(b).

Initial porosities are observed in the Al 6056 and Al 6061 both in the X-ray microtomography images and SEM micrographs. None of these porosities are observed in the Al 6005A at the resolution of the X-ray microtomography images. The initial porosity f_0 , equivalent diameter and shape factor are provided in Table 2.3. Some of these cavities originate from the gathering during Al solidification of the hydrogen easily dissolved in liquid Al [117] while others originate from the fracture of intermetallic particles during the forming process.

2.4. Results and discussion

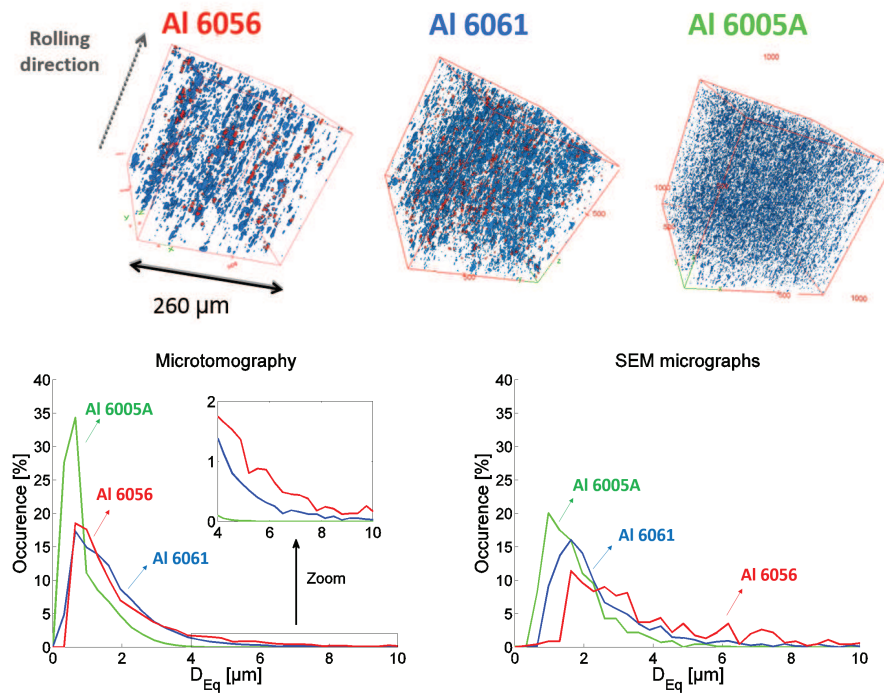


FIGURE 2.5: Characterization of intermetallic particles sizes and initial porosity by X-ray microtomography and SEM. (a) 3D microtomography perspective of particles (blue) and pores (red) distribution in all three alloys. Distribution of equivalent diameter for the intermetallic particles; (b) based on the 3D X-ray microtomography images (c) based on the SEM micrographs.

Chap. 2: Ductile fracture of 6xxx aluminium alloys

Technique	Parameter	Alloy		
		Al 6005A	Al 6061	Al 6056
SEM	Surface fraction	1.2%	1.2%	1.0%
Microtomography	Volume fraction	0.6 %	1.1 %	0.85 %
SEM	Shape factor	1.7 ± 0.7	1.6 ± 0.6	1.7 ± 0.9
Microtomography		1.8 ± 0.7	2 ± 0.8	2.4 ± 1.4
SEM	Equivalent diameter	1.3 ± 0.7 μm	2.1 ± 1.3 μm	3.1 ± 2.1 μm
Microtomography		0.9 ± 0.7 μm	1.8 ± 1.3 μm	2.1 ± 1.8 μm

TABLE 2.2: Mean volume fraction, shape factor, diameter (based on the surface area) of the intermetallic particles in the three aluminium alloys; results based on FEG-SEM image analysis; observations performed at mid-thickness viewed from the top of the plates.

Parameter	Alloy	
	Al 6061	Al 6056
Volume fraction	0.11%	0.12%
Equivalent diameter	2.4 ± 1.1	3.7 ± 2.2
Shape factor	1.7	1.85

TABLE 2.3: Mean volume fraction and equivalent diameter (based on X-ray microtomography images) of the initial porosities.

Fig. 2.6 presents the yield stress σ_0 of the three alloys as a function of the various heat treatments durations. In each alloy, the yield stress peak corresponds to the T6 state. Fig. 2.1 summarizes the results for the

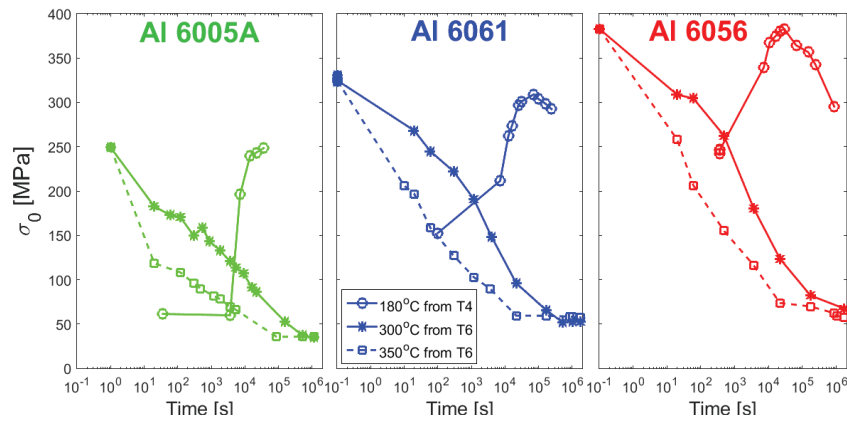


FIGURE 2.6: Yield stress σ_0 corresponding to the various heat treatments performed on the three alloys.

2.4. Results and discussion

three alloys in terms of true fracture strain (Fig. 2.1.a) and of true strain at onset of necking (Fig. 2.1.b) as a function of the yield stress. While the fracture strains are very different for the three alloys, the necking strains follow the same "banana" shaped trend with limited exceptions. This clearly indicates that uniform elongation is absolutely not representative of the true fracture strain. Fig. 2.1(c,d) provide the dislocation storage rate θ_0 and parameter β . These parameters are needed to account for strain hardening in the model (Fig. 2.4, input 3).

2.4.2 Damage evolution: experimental results and model parameter identification

2.4.2.1 Void nucleation

The magnitude and distribution of particle fracture stress can be identified in the following way. Metallographic observations of the polished sections of broken specimens (cf. section 2.2.3.2) are used instead of in-situ microtomography tests because the void nucleation kinetics is not accurately captured with the resolution of about $1 \mu m^3$ (cf. section 2.2.3.3) when compared to the particle size (1 to $4 \mu m$). The maximum principal stress σ_{Part}^{max} in each particle can be estimated using equation (2.4) based on the overall stress and strains calculated by standard FE simulations at the location of the particles. If a given observed particle is broken, the critical stress at which void nucleation has occurred lies between 0 and σ_{Part}^{max} . The nucleation stress of the broken particles is said to be interval-censored and falls in the interval $(0, \sigma_{Part}^{max}]$. On the other hand, if a given particle is not broken, it can only be concluded that the critical stress at which void nucleation would occur is larger than σ_{Part}^{max} . The nucleation stress of the unbroken particles is said to be right-censored and falls in the interval $(\sigma_{Part}^{max}, \infty]$. The exact nucleation stress of the i^{th} particle is thus not exactly known but is associated to an interval $(0, \sigma_{Part}^{max}]$ or $(\sigma_{Part}^{max}, \infty]$. A mathematical iterative procedure is needed in order to retrieve from these data, the nucleation stress distribution. A set of Weibull parameters is selected and for all the particles used to generate the curve of Fig. 2.7, a critical stress σ_c^i is assigned to each particles. σ_{Part}^{max} is calculated for each particle and if the critical stress σ_c^i is larger than σ_{Part}^{max} , the particle is considered as broken. The criterion is checked for all particles and the prediction of "broken" versus

"unbroken" is compared to the experimental record. This procedure is repeated for several sets of Weibull parameters and the best parameters are found to be $\sigma_w = 1200$ MPa, $m=7$, and V_0 corresponding to an equivalent diameter $d_0 = 2.2 \mu m$.

A very strong assumption is made regarding the void nucleation stress: the magnitude and distribution of void nucleation stress is taken identical for the three alloys, depending only on the particle size. Alternatively, different critical stress distribution could have been identified but, for the sake of simplicity, and mainly to focus on the primary issue of particle size and spatial distribution, it was decided to aggregate all data together. This implicitly assumes that the intermetallic particles in the three alloys have a similar structure and mechanical behaviour.

Fig. 2.7 shows the evolution of the fraction of broken particles as a function of particles size as observed experimentally and as obtained with the best Weibull parameters ($\sigma_w = 1200$ MPa, $m=7$, and V_0 corresponding to an equivalent diameter $d_0 = 2.2 \mu m$) found in section 2.3.2. The predictions agree with the experimental evolution. Consequently, a Weibull distribution using the same parameters for the 3 alloys seems to be a reasonable approximation partly validating our initial choice. The fraction of broken particles increases with particle size. Fig. 2.7 also shows that smaller particles give rise to void nucleation in the 6005A alloy (Fig. 2.5(b)), but at relatively large equivalent strains (as it will be shown in section 2.4.3) corresponding to sufficiently large stress needed to break small particles. The alloy 6061 presents an intermediate behavior consistent with its reduced proportion of large particles (Fig. 2.5(b)).

2.4.2.2 Void growth

The results of the tracking carried out on the Al 6056 specimen provides the experimental void growth rate for each cavity (Fig. 2.8(a)), with clear dispersion in the growth kinetics.

Fig. 2.8(b) shows a comparison of the experimental and predicted evolution of the relative rates of change $R_{eq}/R_{eq,0}$ of the equivalent radius of the voids. These rates have been averaged over all voids. The predictions of the R&T (cf. eq 2.6, section 2.3.3) in the original and "constraint" versions are provided in Fig. 2.8(b) for the void growth, and in

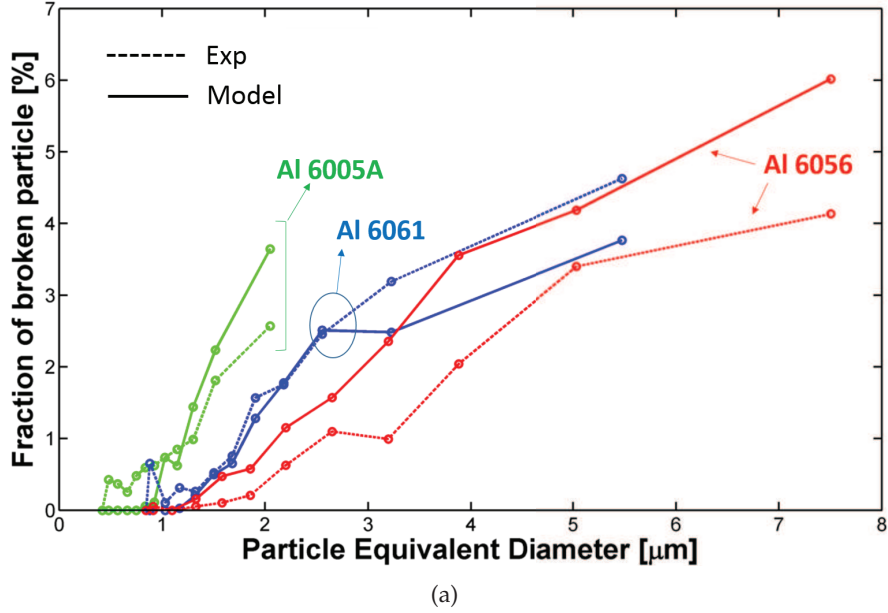


FIGURE 2.7: Comparison between the experimental and modelled (best identification) fraction of broken particles as a function of the particles equivalent diameter in the three alloys for all heat treatment conditions.

Fig. 2.8(c) for the void aspect ratio evolution. The void aspect ratio W_{void} is defined as $W_{void} = \frac{R_1}{\sqrt{R_2 R_3}}$, see section 2.3.3. Knowing the values of T and of the strain around the cavity for each microtomography measurement, an inverse procedure is carried out in order to calculate the value of α (eq. 2.6) which best corresponds to the experimental relative growth rate for each version of the model. The best value associated to the void growth model is $\alpha \approx 0.84$. With $\alpha \approx 0.84$, one can observe that the rate of change of the void aspect ratio is then significantly over predicted (Fig. 2.8(c)). The same procedure has been carried out for the "constraint" growth model. In this case, the best value for the parameter α is found to be 0.57, much closer to the theoretical value 0.427 [66], and the rate of change of the void aspect ratio is more consistent with the experimental data. Landron [73] and Lecarme et al. [74] have largely discussed several possible reasons that justify why α can be different from the theoretical value. Now, this value $\alpha = 0.57$ and constraint growth model are used for all other materials and all thermal treatments.

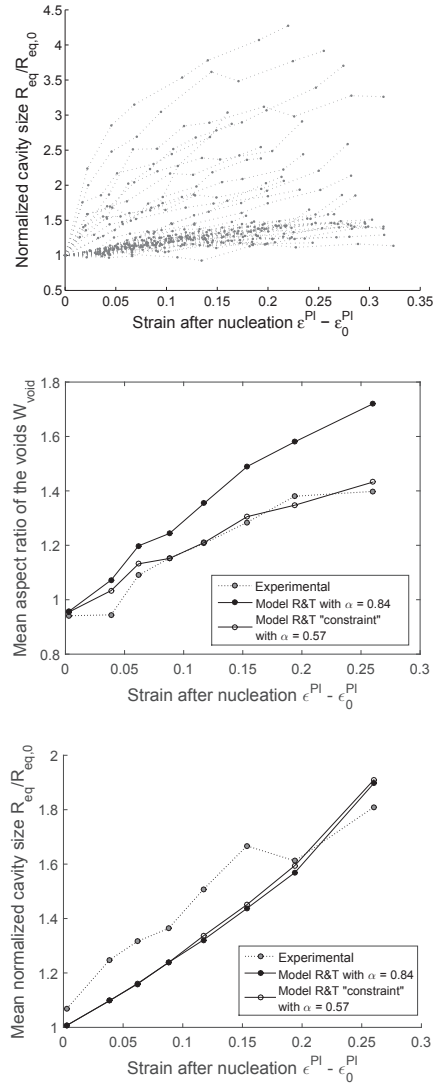


FIGURE 2.8: (a) Evolution of the rates of change of the equivalent radius of every void $\frac{R_{eq}}{R_{eq,0}}$ as a function of strain after nucleation; (b) Evolution of the mean rate of change $\frac{R_{eq}}{R_{eq,0}}$ of all voids; (c) Evolution of the mean void aspect ratio W_{void} .

2.4.3 Exploitation and assessment of the model

The model is assessed in two steps. First, by direct comparison of the full model with microtomography results obtained on the three alloys with a similar yield strength. Second, all heat treatment conditions are modelled and compared to the experimental ϵ_f , as provided in Fig. 2.1.a.

The growth model is now coupled to the nucleation and coalescence model, which was not the case in Fig. 2.8.

Fig. 2.9 shows the evolution of the void volume fraction f . The predicted evolution of f agrees with the experimental evolution. The microstructural parameters used to run these simulations (see section 2.3.6) are identified from the X-ray microtomography data of section 2.2.2, with a resolution of about $0.034 \mu m^3$. Indeed, the resolution of the first scan of the material (i.e. in its undeformed state) of in-situ microtomography tests is about $1 \mu m^3$ (cf. section 2.2.3.3) and is not sufficient to accurately capture the particle size distribution ranging mainly between 1 to $4 \mu m$. This explains why the initial void volume fraction predicted by the model is different from the initial void volume fraction measured during in-situ microtomography (see Fig. 2.9.b). However, the fact that the predicted f agrees with the experimental evolution is an indication that the RVE has a sufficient size from a statistical viewpoint to reproduce the experiments.

For the alloy 6005A, the rapid increase of f is predicted later than in the experiments. This could have been corrected by selecting different void nucleation Weibull parameters for each alloy. However, using the same nucleation stress distribution versus particle size, i.e. neglecting possible differences of particles properties between the three alloys, highlights the major effect of particle size distribution on the fracture strain.

Fig. 2.10 shows in more details results from the model applied to the Al 6056 alloy for three heat treatments (namely T6, T4 and overaged with $\sigma_0 \simeq 60$ MPa) and results for the three alloys in the T6 state. The point on each curve corresponds to the predicted fracture strain, i.e. the strain at which the critical rate of porosity $\dot{f}_c = \left. \frac{\partial f}{\partial \epsilon_{loc}} \right|_c$ is satisfied.

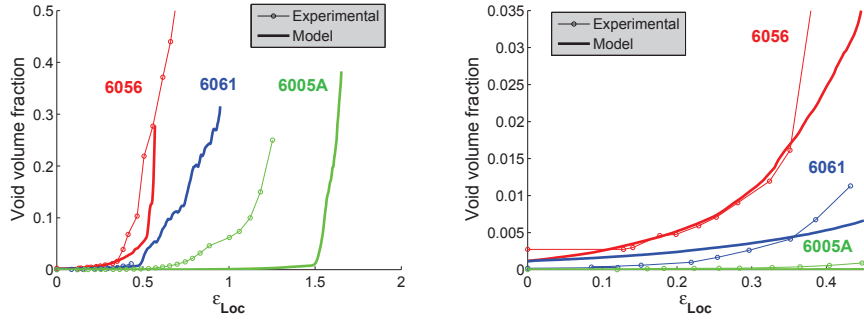


FIGURE 2.9: (a) Comparison between the variations of the void volume fraction (f) measured during in-situ microtomography and the void volume fraction predicted by the model as a function of the local strain in the necking zone; (b) Zoom in figure (a).

Results obtained with the model for the three alloys in the T6 state explain the large differences in ϵ_f . Fig. 2.10(b) shows a fast increase of the fraction of broken particles, i.e. a large nucleation rate, for the Al 6056 and Al 6061. This fast increase corresponds to very large particles which break at very low strain. Very large particles are present in larger proportion in the alloy 6056, see inset of Fig. 2.5(b), and it exhibits the highest fraction of broken particles. On the opposite, this initial fast increase of the fraction of broken particles is much lower for the Al 6005A due to the absence of large particles in this alloy, see inset of Fig. 2.5(b). After a few percents of deformation, the increase of the fraction of broken particles reaches a linear evolution. The rate of increase in this linear regime is the highest for Al 6056, and higher for Al 6061 compared to Al 6005A. This is consistent with the particle size distribution (see Fig. 2.5(c)), similarly to what was concluded from Fig. 2.7. Fig. 2.10(c) shows that the differences in the fractions of coalesced voids between the three alloys correlate with the trends in void nucleation. This is due to the fact that more void nucleation increases the void density, and this locally increases probability that two voids satisfy the coalescence criterion. Fig. 2.10(d) shows the variation of the total number of voids, i.e. the sum of the initial cavities and nucleated voids minus the number of coalescence events. In each case, the total number of voids exhibits first an increase, as it is dominated by void nucleation, up to some point where voids linkage spread over. At this point, the number

2.4. Results and discussion

of voids starts to decrease and the void volume fraction increases very fast (Fig. 2.10(a)).

Predictions of the model applied to the Al 6056 alloy for three heat treatments (T6,T4,OA) explain why, for a given alloy, ε_f decreases with increasing σ_0 . This well known evolution [152] is associated to the larger strains needed to reach the same stress level in particles surrounded by a soft matrix (Fig. 2.10(b)) and is consistent with a stress criterion for the fracture of the particles. The density of voids and the number of coalescence events, at a given strain, is thus lower if σ_0 decreases (see Fig. 2.10(c,d)).

If the final failure criterion is chosen as a given fraction of coalesced voids (10% for exemple, see Fig. 2.10(c)) or as the beginning of the fast decrease in the total number of voids (Fig. 2.10(d)), the predicted ε_f is different, but the differences between the three materials or between the heat treatments exhibit the same trend. This is due to the fact that the fast increase of void volume fraction is associated to voids linkage over a macroscopic scale that triggers catastrophic ductile failure.

The model is now applied to predict the evolution of ε_f with σ_0 for all heat treatments conditions. The critical $\dot{f}$, $\dot{f}_c$, was identified as equal to 24 ($\dot{f} = \frac{\Delta f}{\Delta \varepsilon_{Loc}}$, with $\Delta \varepsilon_{Loc}=0.01$) to capture one data point of one of the three alloys and then kept constant for all other conditions. The results are shown in Fig. 2.11. The model nicely captures the differences in ε_f among the three alloys as well as the decrease of ε_f with the increase of σ_0 . The difference between the alloys is thus primarily related to the particle size distribution. Once again, the cellular automaton model parameters have been identified directly from experimental data with a clear physical meaning with no additional fitting or adjustment of tuning parameters except for $\dot{f}_c$ and α (with for the last one a value that remains close to the theoretical one).

2.4.4 Sensitivity analysis

Fig. 2.12 presents the sensitivity analysis of the CA to the inclusion orientation and spatial distribution. The analysis also shows the

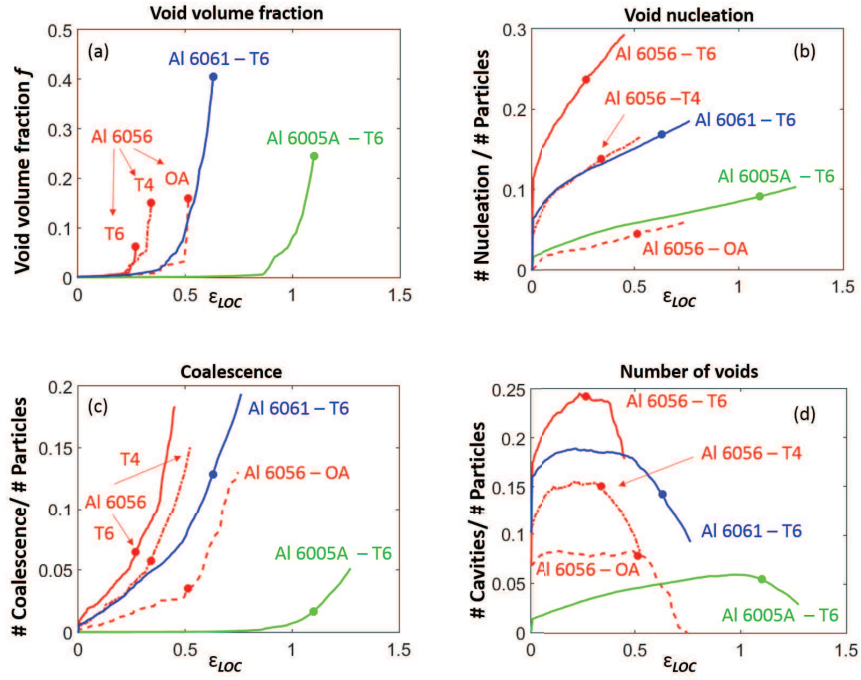


FIGURE 2.10: Results of the model applied to the Al 6056 alloy for three heat treatments (namely T6, T4 and overaged with $\sigma_0 \simeq 60$ MPa). Comparison between : (a) the variations of the void volume fractions; (b) fraction of broken particles extracted from the model; (c) number of coalescence events; (d) total number of voids. All the data are extracted from the model and their evolution are presented as a function of the local strain in the necking zone. The point on each curve corresponds to the predicted fracture strain.

effect of using the two versions of the void growth model and the effect of the void aspect ratio definition used in the Thomason criterion. The sensitivity analysis has been performed for the Al 6056 alloy under the three different heat treatments (namely T6, T4 and overaged with $\sigma_0 \simeq 60$ MPa) which have already been studied in Fig. 2.10. The version of the CA developed in section 2.3, and applied to the microstructure extracted from X-ray microtomography volume, will be referred as the "full model". Each effect has been tested independently.

Sensitivity to inclusion orientation

The sensitivity of the model to inclusion orientation has been anal-

2.4. Results and discussion

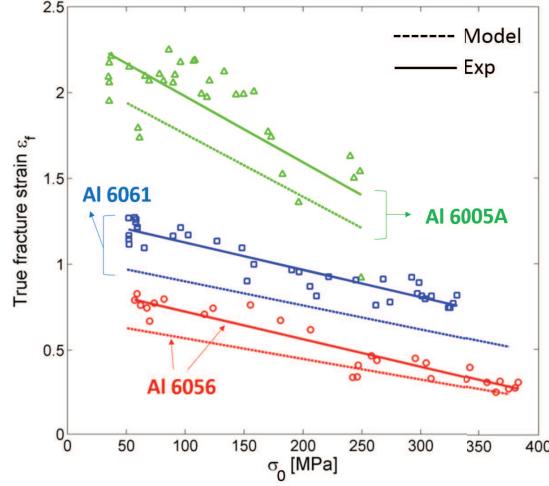


FIGURE 2.11: Comparison between the experimental fracture strain and the fracture strain of the model as a function of yield stress. The plain line corresponds to the linear regression of experimental values. The dotted line corresponds to the linear regression of the values from the model.

used by testing the extreme case for which all the particles have their major axis aligned with the main loading direction, keeping their volume and spatial distribution unchanged. Fig. 2.12 shows that for this configuration, the predicted ε_f is nearly unchanged in the T6 state, slightly modified in the T4 state and lower in the overaged case by 15 %. The orientation of the particles mainly affects the void nucleation stage. When the major axis of the particles is aligned with the main loading direction, the load transfer arising from the deformation mismatch between particle and matrix increases (see section 2.3.2), as already shown in Ref. [34]. In the T6 and T4 cases, void nucleation is already quite fast (see Fig. 2.10(b)) in any cases but in the overaged case the impact is more significant. The increased void nucleation rate due to the reorientation of the particles has thus a small to moderate impact on ε_f .

Sensitivity to spatial distribution of inclusions

The spatial distribution of the particles used in the full model has been directly extracted from microtomography. A periodic cubic arrangement of particles has been chosen to study the effect of particle

distribution on the damage process as a sort of extreme ideal case. The particles shape and orientation and the overall particles volume fraction remain unmodified compared to the full model. Fig. 2.12 shows that there is a very large effect of the particle distribution on the predicted ε_f . Spatial distribution of particles influences the void coalescence process due to its effect on the local void spacing. On the opposite, the spatial distribution doesn't impact void nucleation and growth since interactions are neglected. The increased ductility with the periodic distribution can be explained by the fact that the voids linkage percolating over a sufficient length scale to trigger catastrophic ductile fracture is significantly postponed. Indeed, as shown in Fig. 2.5(a), the actual spatial distribution of particles is not homogeneous, with some local clustering. The full analysis of the effect of particles spatial distribution, for instance using percolation indicator, goes beyond the scope of the present analysis but this result indicates the need for further investigation on this topic in order to define the right topological indicators to characterize the microstructure heterogeneity with respect to this specific mechanism.

Sensitivity to initial porosity

Fig. 2.12 shows that the initial porosity has a large effect on the predicted ε_f . Indeed, even though f_0 is very low (see Table 2.3), the cavities which are initially present in the material are quite large compared to the penny-shaped voids nucleated by particle fracture. The presence of an initial porosity facilitates voids linkage over a sufficient length scale to trigger earlier catastrophic ductile failure. The effect is more important in the overaged case since void nucleation is a dominant phase in the T6 and T4 cases (see Fig. 2.10(b)), attenuating the impact of the presence of an initial porosity. Now, looking back at the comparison between the 3 alloys tested experimentally, the initial porosity is also a factor that can justify the large difference in fracture strain among the three alloys as ε_f clearly decrease with increasing f_0 , see Table 2.3.

Sensitivity to the void growth model

As explained in section 2.3.3, the R&T void growth model has been slightly modified to take into account the effect of the presence of the particle on void growth. Furthermore, the best value for the parameter α_{growth} has been shown to be 0.57 instead of the theoreti-

2.4. Results and discussion

cal value 0.427. This modified void growth model has been shown to give a better agreement with the experimentally observed void growth (section 2.4.2.2). In order to assess the significance of this correction, the model has been applied with the original version of the R&T void growth model (i.e. $\alpha_{growth} = 0.427$ and no effect of the particle on the void growth). Fig. 2.12 shows that there is a strong increase of the predicted ε_f without the correction. Indeed, reducing the parameter α_{growth} from 0.57 to 0.427 induces a slower void growth rate retarding fracture. Furthermore, at low stress triaxiality, the original growth model predicts a decrease of the void size in the transverse directions while, in reality, the particle constraints the transverse contraction (see section 2.3.3). This delays the coalescence process as the relative void spacing is reduced and this shows that fracture in these alloys is also partly dominated by the void growth phase.

Sensitivity to coalescence model

A heuristic extension of the Thomason criterion has been used in order to define an effective void aspect ratio W_{eff} (see section 2.3.4). As mentioned previously, this extension has been partly assessed in [88], although only for spheroids of same dimension. To test the impact of the assumptions made to estimate W_{eff} , the effective void aspect ratio W_{eff} used in eq.(2.7) has been fixed equal to 1 as an extreme case. In other words, the coalescence criterion is evaluated as if the two voids were spherical. This extreme assumption is used to evaluate the void coalescence criterion only; the shape of the actual void is used in the rest of the model. Fig. 2.12 shows that the predicted ε_f remains nearly unchanged in the overaged case. In this ductile condition, a significant void growth occurs before the coalescence criterion is met with a shape that gets close to equiaxed, see Lassance et al. [76] for more detailed analysis of the void growth and coalescence of initially penny-shaped voids. In the T6 state, void nucleation is the dominant stage (see Fig. 2.10(b)) and coalescence events occurs at smaller strains (see Fig. 2.10(c)). Since the void growth stage is reduced, the opening of the penny-shaped voids is reduced and the aspect ratio is significantly smaller than one when coalescence starts.

As a conclusion to the sensitivity analysis, it shows first that the particle distribution is the most significant factor. It also shows that it is important to test the validity of the three elements of the model; void

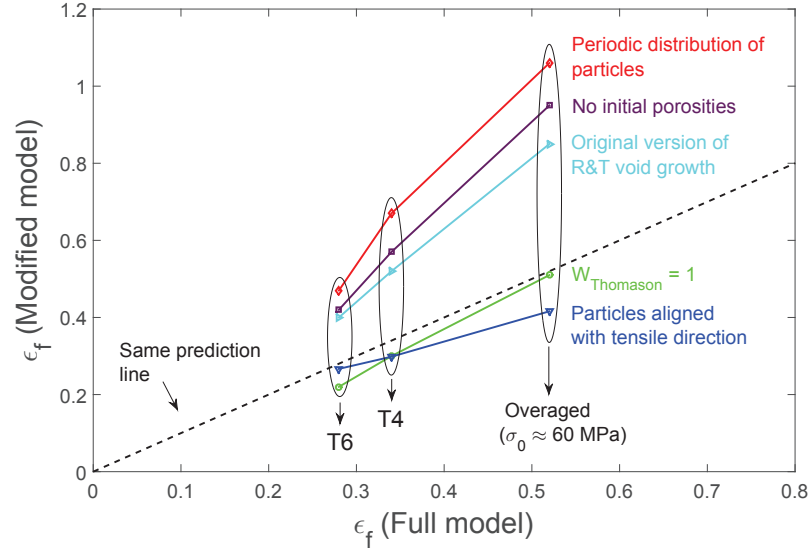


FIGURE 2.12: Sensitivity analysis on different contributions of the cellular automaton model. The "full model" corresponds to the predictions of the model derived in the present paper and used in Fig. 2.10 and Fig. 2.11 for comparison to experimental data. The "modified models" are degraded versions of the model.

nucleation, void growth and void coalescence as they can play a first order role depending on the hardening of the matrix.

2.5 Conclusion

The hardening and fracture properties of three 6xxx series aluminum alloys have been measured for a wide variety of heat treatment conditions. The tensile tests revealed major differences in the fracture strain for the same yield strength, even though the volume fractions of second phase particles are very similar ($\sim 1\%$). The fracture strain decreases with increasing yield strength with about the same slope for each alloy. A cellular automaton model, based on simple micromechanical models for void nucleation, growth and coalescence taking into account microstructural heterogeneities is presented. It provides

2.5. Conclusion

very good predictions regarding the different fracture strain variations with only two fitting parameters, out of which one is close to the theoretical estimate.

The main conclusions of the study are the following :

- the fracture strain is primarily dictated by the effect of particle size distribution with the alloy exhibiting the smallest particles being the most ductile;
- the use of the same magnitude of nucleation stress and distribution with particles sizes for the three alloys is sufficient to deliver adequate predictions indicating that the intermetallic particles properties must be relatively similar;
- particle spatial distribution when compared to periodic distribution plays also a first-order effect on controlling the ductility;
- the presence of pre-existing voids has also a first order effect on the predicted fracture strains;
- the experimental scattering in the void growth rate in aluminum is significant, such as recently concluded for a titanium alloy (see Lecarme *et al*[74]);
- a cellular automaton model involving simple damage criteria is a relevant approach to address the fracture strain prediction in the case of a metallic alloy exhibiting significant heterogeneity effects.

Appendix

The void growth model has been developed for voids with the main axes aligned with the principal loading directions ($\Sigma_1, \Sigma_2, \Sigma_3$). An heuristic procedure is proposed in order to estimate the void growth rates in the case of an ellipsoidal void under general loading conditions. The principal radii of the ellipsoidal void are replaced by the three intersections of the void with the directions of the principal stress (R_1, R_2, R_3 , see Fig. 2.13). This equivalent void is then assumed to follow the growth model given by equation (2.6). The problem can now be

treated as the stretching of an ellipsoid along three arbitrary directions (see Fig. 2.13) which requires an eigenvalue solution where the new void semi-axes and orientation is defined by the eigenvalues and the eigenvectors. This procedure for the arbitrary stretching of an ellipsoid is detailed in [157]. Using this void growth model, the void will grow and reorient itself to be aligned with the preferential loading direction. If the directions of the semi-axes and principal loading directions are aligned, the void will not rotate. The center of the ellipsoid remains unchanged.

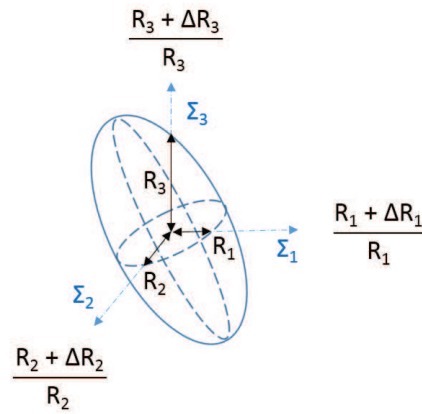


FIGURE 2.13: Definition of the effective void dimensions used in the growth model.

2.6. Additional comments on the onset of necking

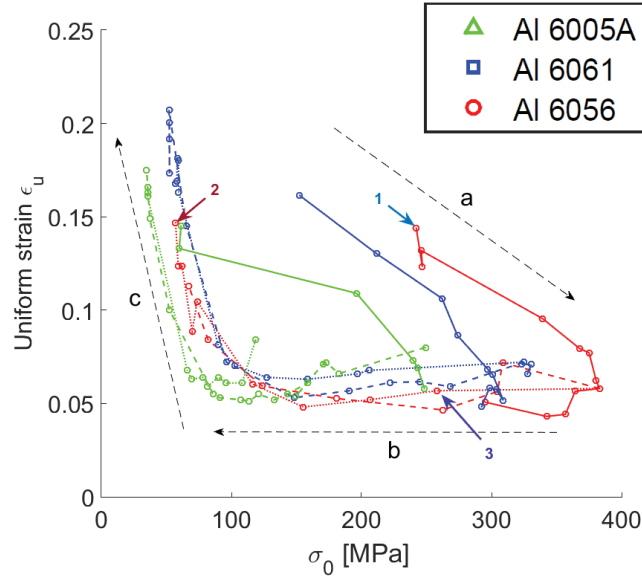


FIGURE 2.14: Variation of the true strain at the onset of necking ε_u extracted from the uniaxial tensile tests on the three alloys as a function of the yield stress corresponding to the different heat treatments. Plain line, dashed line and dotted line correspond to heat treatments which are performed at 180°C, 300°C and 350°C, respectively. Stage *a*, *b* and *c* indicate the evolution with increasing heat treatment time.

2.6 Additional comments on the onset of necking

In this section, the evolution of the uniform strain ε_u with the yield stress σ_0 shown in Fig. 2.1(b) is discussed in more details.

Fig. 2.14 shows the evolution of the uniform strain ε_u with the heat treatments. The plain line indicates heat treatments which are performed at 180°C on samples in the T4 state. The dashed line and the dotted line indicate heat treatments which are performed at 300°C and 350°C on samples in the T6 state. The evolution can be divided into three stages *a*, *b* and *c*. These three stages are linked to the evolution of the strain hardening parameters, as explained in the following.

The strain hardening parameters used are the dislocation storage rate Θ_0 , the dynamic recovery rate β and the stage IV hardening Θ_{IV}

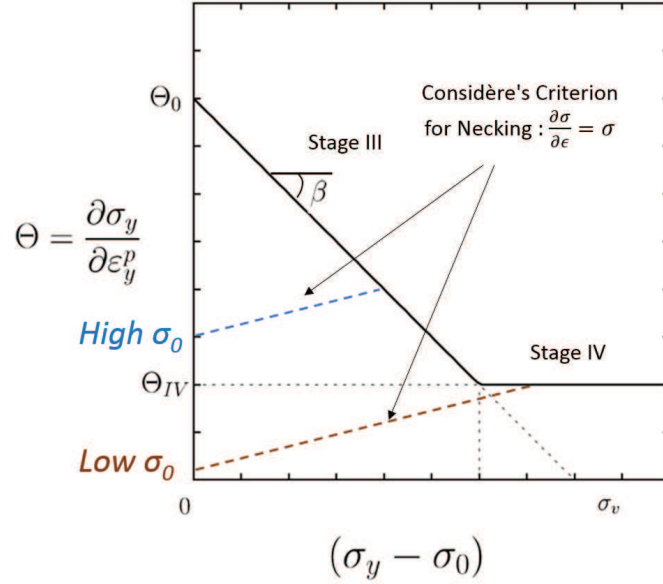


FIGURE 2.15: Definition of the strain hardening parameters (Θ_0 , β , Θ_{IV}) of the Voce law involving both stages III and IV based on a hardening rate versus flow stress plot.

following the extended Voce law which is used (see Fig. 2.15). The Considère condition which sets the strain at which necking starts in rate independent materials (and without imperfection) is also shown in Fig. 2.15.

The evolution is very similar for the three alloys and will be discussed in more details only for the alloy Al 6056. Fig. 2.16 shows the evolution of the parameters Θ_0 and β with the heat treatments.

The physical interpretation of these trends are discussed based on Simar *et al.* [152]:

Stage a : Starting from a supersaturated solid solution (T4 state), the 180°C heat treatment gives rise to the growth of β'' strengthening precipitates for durations lower than about 8h (T6 state). This leads to an increase in σ_0 , a drop of Θ_0 and a very limited change of the parameter β (see Fig. 2.16). This explains why the uniform strain ε_u decreases during stage a.

2.6. Additional comments on the onset of necking

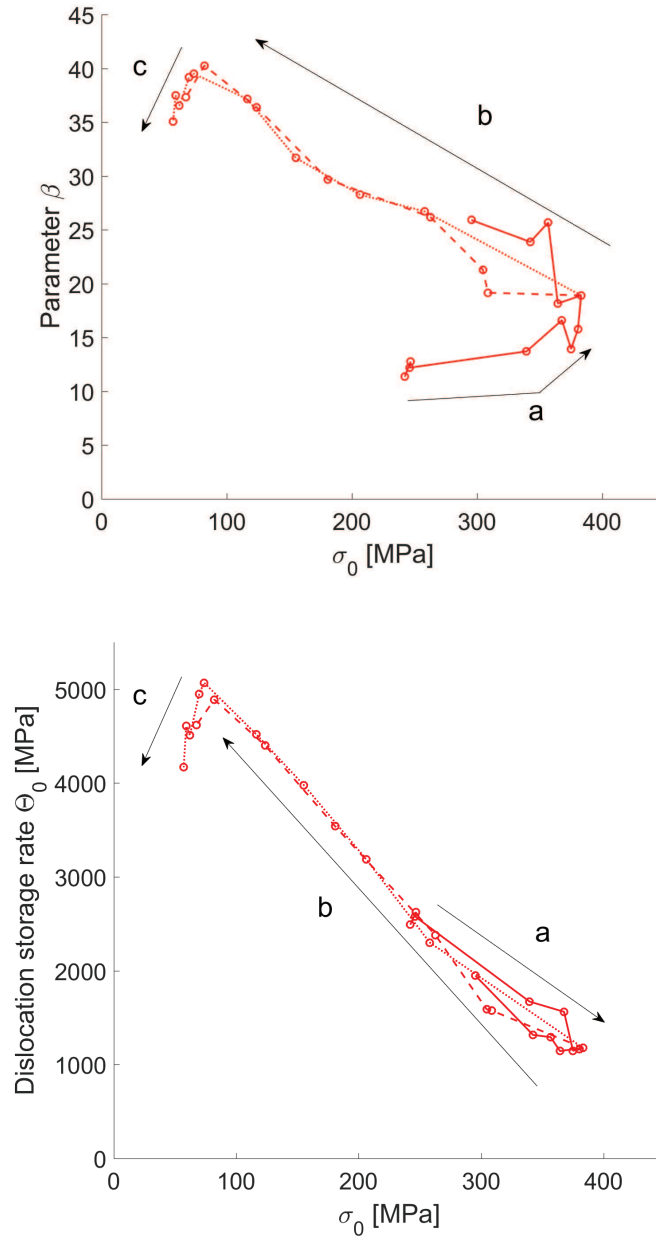


FIGURE 2.16: Variation of (a) Θ_0 and (b) β extracted from the uniaxial tensile tests on the alloy Al 6056 as a function of the yield stress corresponding to the different heat treatments.

Stage *b* : Further heat treatments at 180°C and higher temperatures (300°C, 350°C) were performed from the T6 state. This leads to the dissolution of the β'' precipitates and the formation of semi-coherent β' precipitates and a continuous decrease of σ_0 is observed. Θ_0 and β increase both strongly and the impact of these two factors cancel more or less each other in such a way that the uniform strain ε_u remains roughly unaffected during stage *b*.

Stage *c* : For very long heat treatments, the yield stress σ_0 becomes very low. Both Θ_0 and β decreases and this drop can be attributed to the efficient storage of Orowan loops around precipitates. Furthermore, a constant stage IV hardening is observed before necking takes place and the plastic slope deviates from the linear evolution of stage III. The yield stress σ_0 is low and the Considère's criterion is met after that the stage IV, which explains why ε_u increases significantly during stage *c*.

Fig. 2.17(*a*) shows the stress-strain curves corresponding to points (1), (2) and (3) in Fig. 2.14. The measured and fitted tensile curves are shown in plain and dashed lines, respectively, showing a good agreement. Fig. 2.17(*b*) shows the evolution of the plastic slope as a function of $(\sigma_{true}-\sigma_0)$ for points (1), (2) and (3) in Fig. 2.14. The Considère condition which indicates necking is also shown and is in good agreement with the measured tensile strength (σ_u).

2.6. Additional comments on the onset of necking

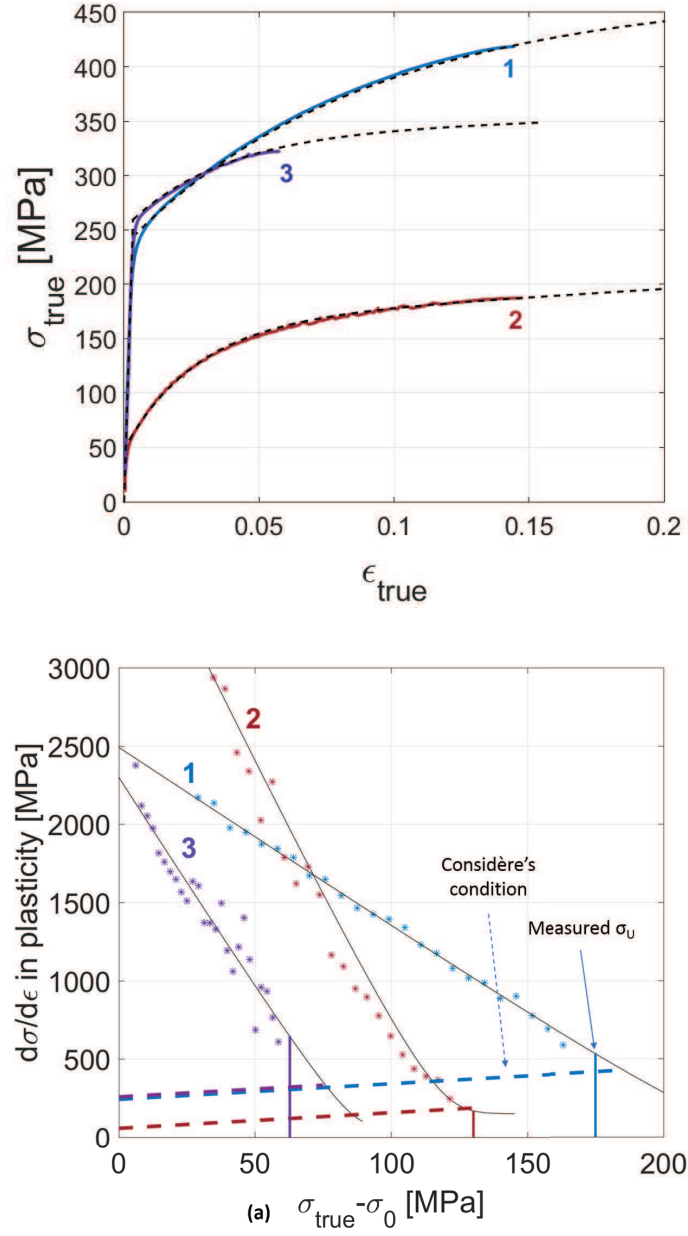


FIGURE 2.17: (a) Stress-strain curves corresponding to points (1), (2) and (3) in Fig. 2.14. (b) and hardening rate versus flow stress plots corresponding to these points.

2.7 Additional microtomography data

Fig. 2.18 and Fig. 2.19 shows 3D microtomography perspectives of the damage evolution observed by in situ tomography for the specimen Al 6056 and Al 6005A, respectively. The corresponding void volume fraction are shown in Fig.2.9(a).

2.7. Additional microtomography data

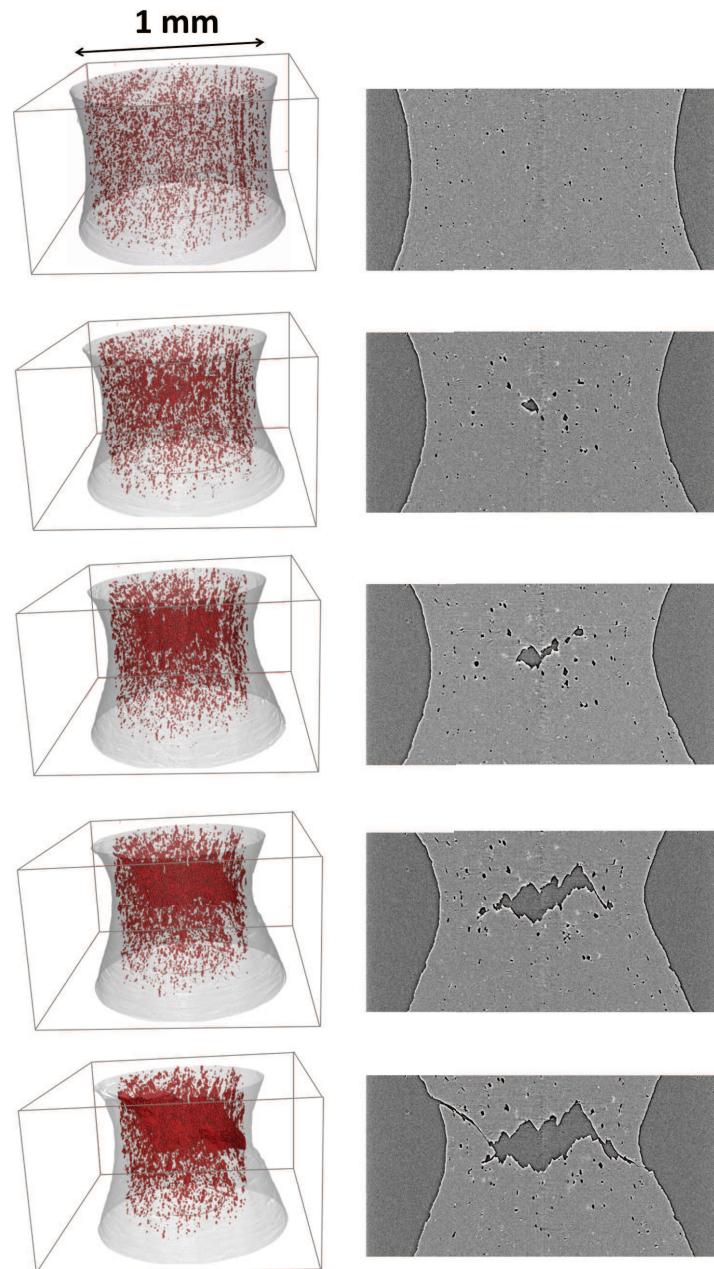


FIGURE 2.18: 3D microtomography perspectives of the damage evolution for the Al 6056.

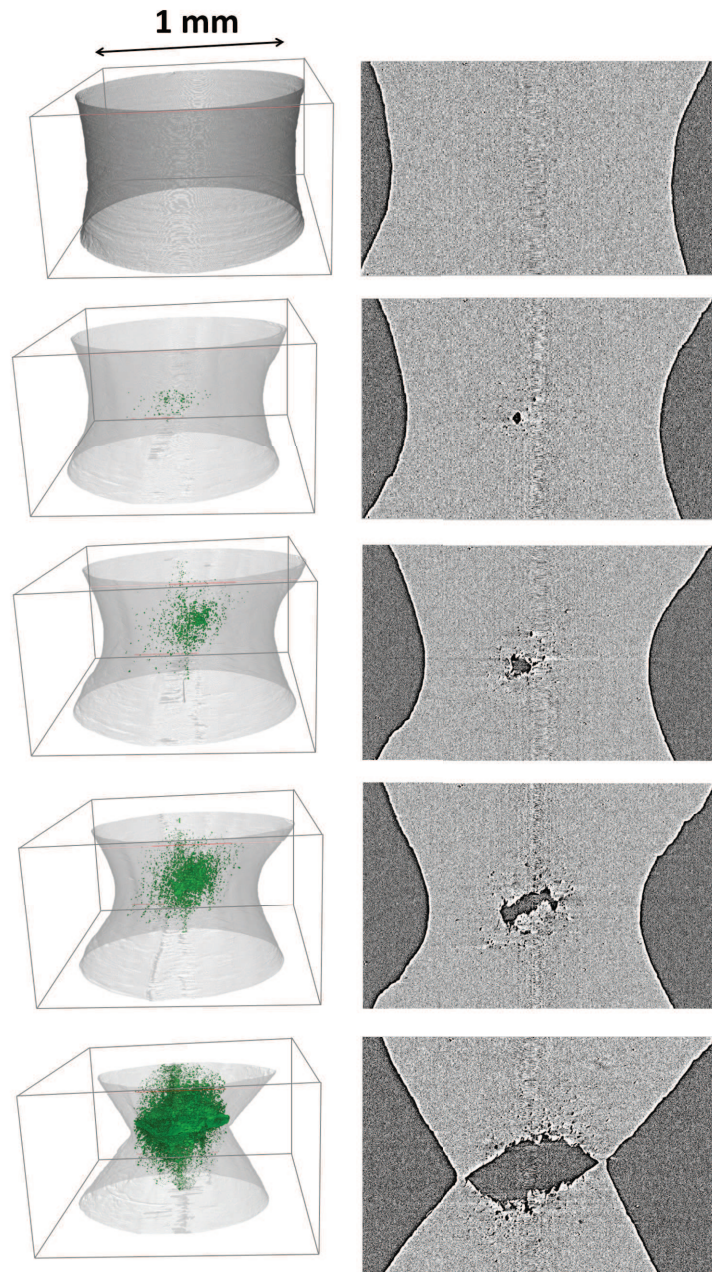


FIGURE 2.19: 3D microtomography perspectives of the damage evolution for the Al 6005A.

2.8 3D vizualisation of the automaton cellular

The comparison between the experiment and the automaton cellular is shown in Fig. 2.21 for the Al 6056 in the overaged state ($\sigma_0 = 120\text{MPa}$) and in Fig. 2.22 for the Al 6056 in the T6 state. The full representative volume element (RVE) used in the cellular automaton is shown in Fig. 2.20 ($\approx 1\text{mm} \times 1\text{mm} \times 0.15\text{ mm}$). In order to visualize easily the percolation process, the cavities which are found in the 3D RVE are projected on a 2D plane (see Fig. 2.20.)

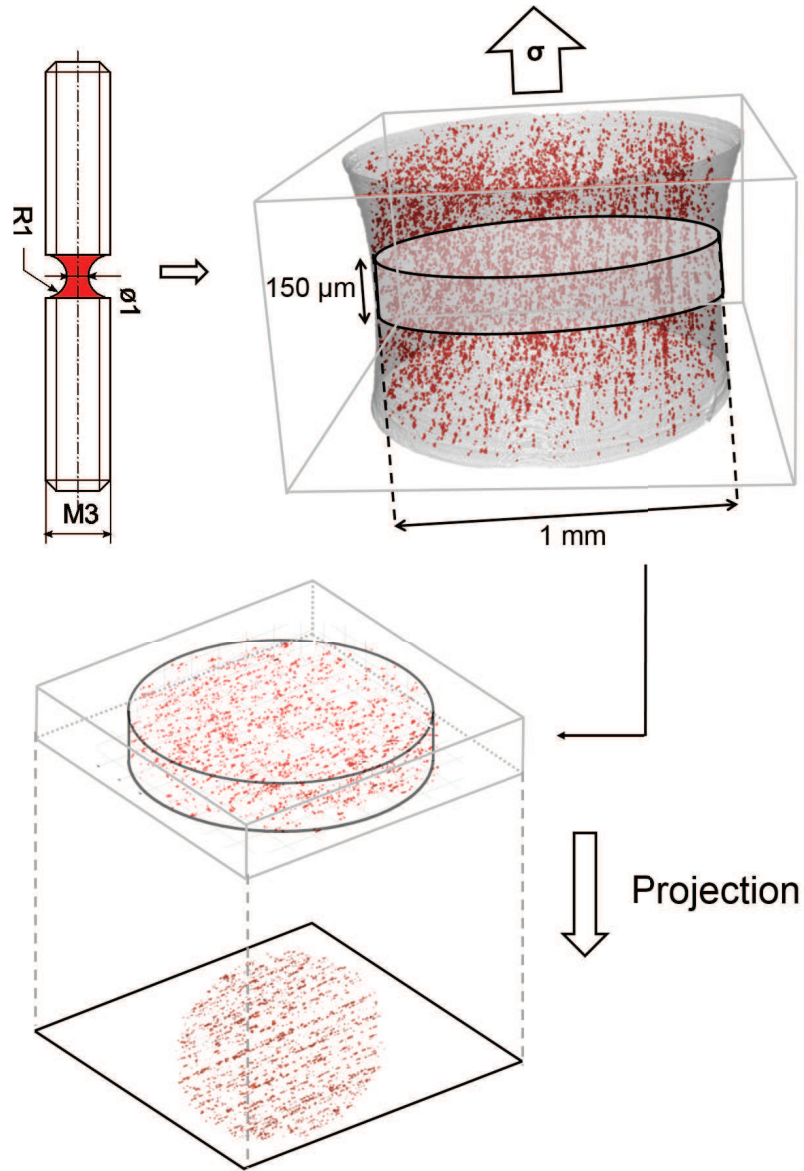


FIGURE 2.20: Vizualisation of the RVE used in the automaton cellular and the projection procedure used to compare it with experiments.

For the experimental images, all the cavities are shown. For the model images, only cavities that have already been involved in a coalescence process are shown (in order to highlight the percolation pro-

cess). Particles are not shown for clarity, but have obviously been taken into account in the model for void nucleation.

Once the coalescence criterion is satisfied for two voids, failure of the inter-void ligament is modelled by merging the two voids and replacing them by the minimum ellipsoid that envelops them both. It is thus not possible to capture perfectly the shape and orientation of the large voids that have undergone multiple coalescence process. However, it is observed in Fig. 2.21 that the comparison between the experiment and the model is very good in the overaged case. The percolation of damage process is captured by the model. The large voids that have been formed by coalescence processes are found at the same location in the experiment and in the model. In the T6 state (Fig. 2.22), the general comparison remains good but the direct mapping between the experiment and the model indicates that the first microcracks formed by the coalescence of voids are not found at the same places. This can be explained by the random nature of void nucleation.

In the overaged case, the matrix is quite soft and the void nucleation remains low. On the opposite, for the T6 case, void nucleation is quite fast attenuating the impact of the presence of initial porosities. The nucleation stress associated to each particle follows a Weibull statistics and voids nucleate first on big particles. However, for particles with roughly the same size, particles that break first are chosen at random and there is no reason for void nucleation to take place on the same particles in the model and in the experiment.

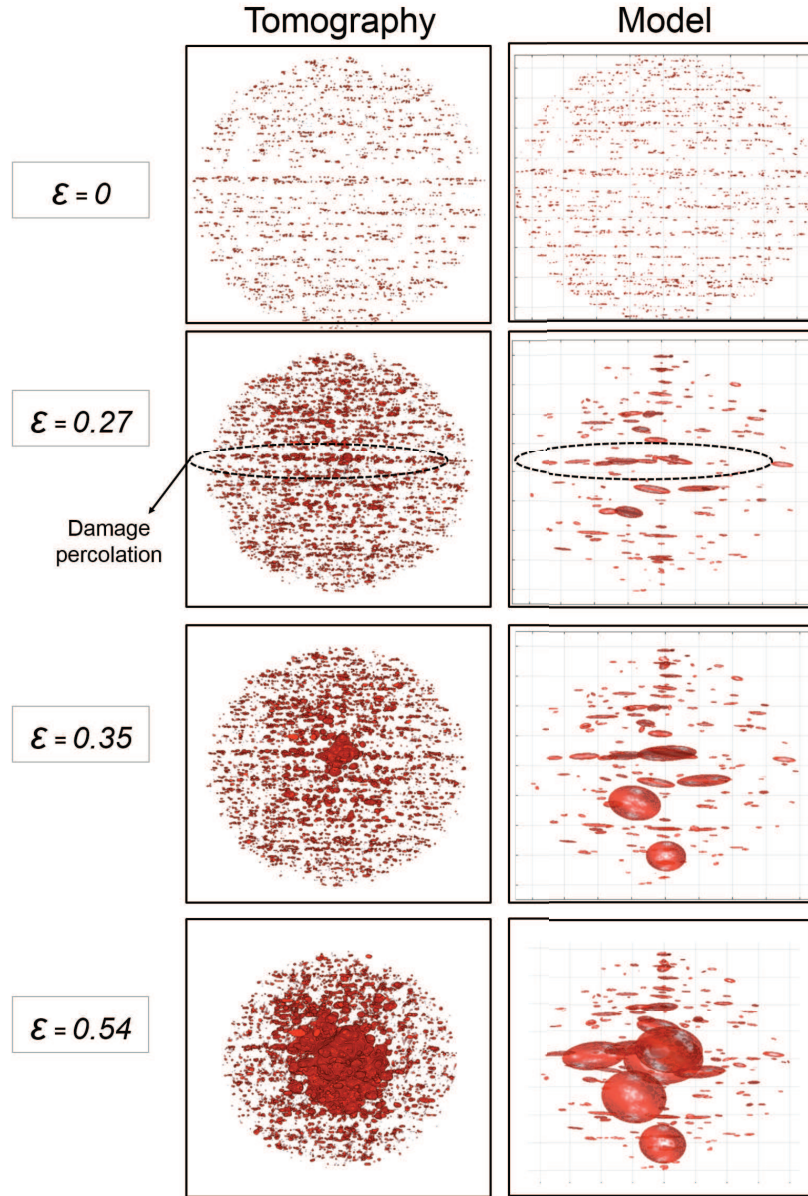


FIGURE 2.21: Comparison between the damage evolution observed by in situ tomography and the evolution predicted by the model. The matrix is in the overaged state. For the model images, only cavities that have already been involved in a coalescence process are shown (in order to highlight the percolation process).

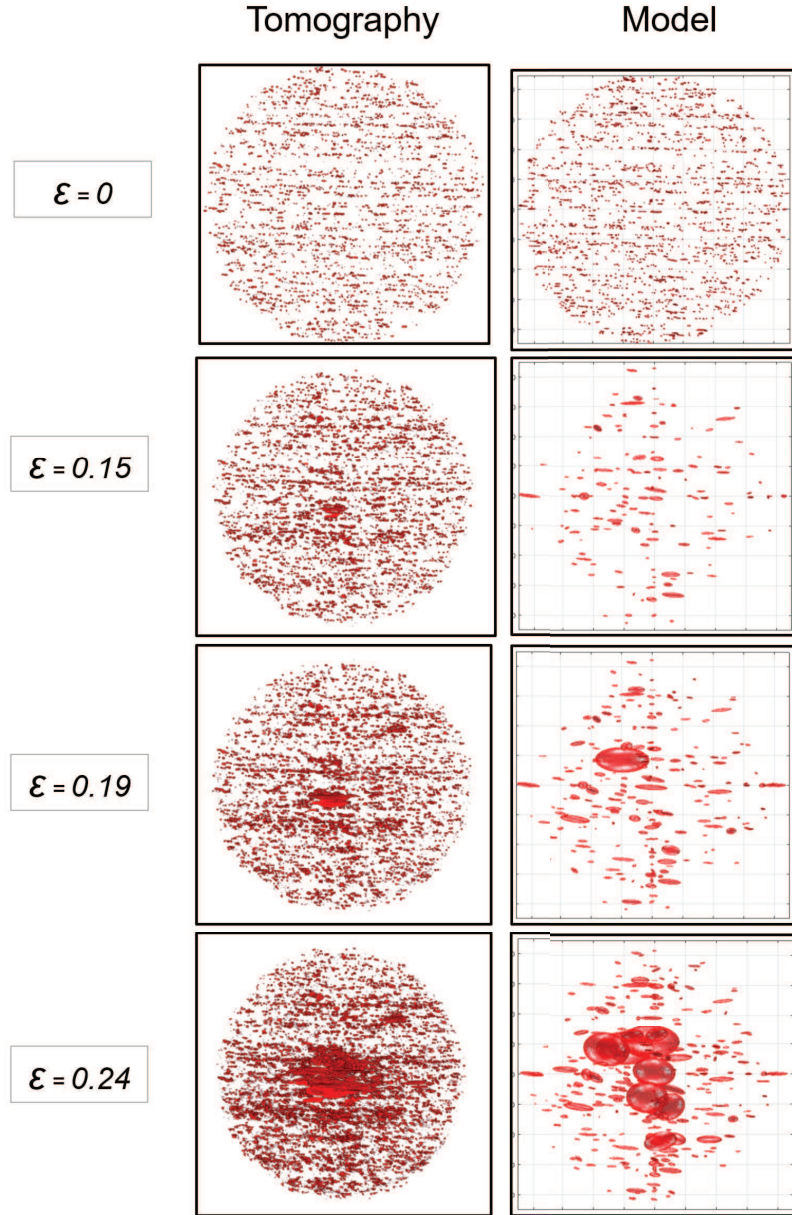


FIGURE 2.22: Comparison between the damage evolution observed by in situ tomography and the evolution predicted by the model. The matrix is in the T6 state. For the model images, only cavities that have already been involved in a coalescence process are shown (in order to highlight the percolation process).

2.9 Void tracking and effect of initial porosity

Initial porosities are observed in the Al 6056 and Al 6061 but not in the Al 6005A and this initial porosity has been shown to have a large effect on the predicted fracture strain ε_f . In order to visualize this effect, the void tracking (see section 2.2.3.3) shown in Fig. 2.23 has been modified to differentiate initial porosities from voids nucleated during straining.

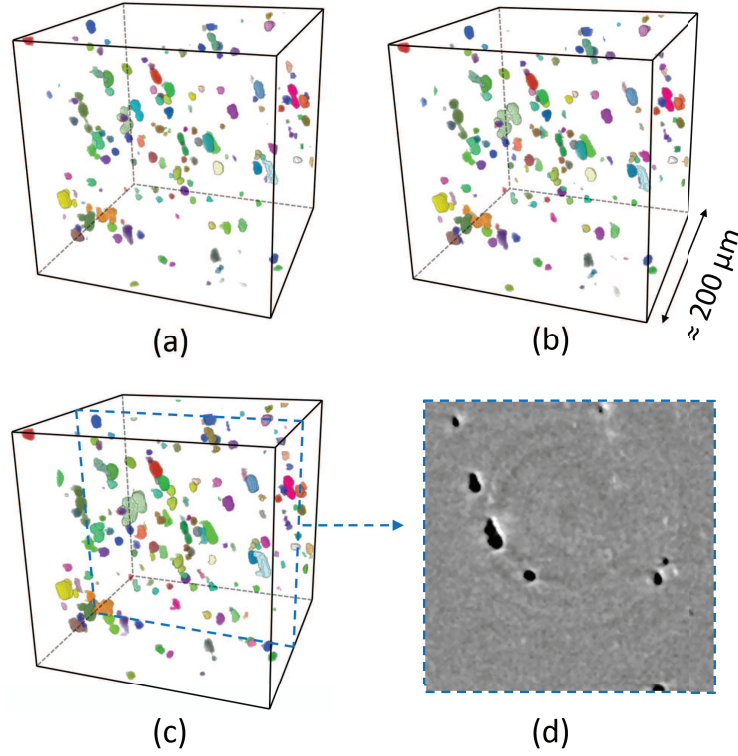


FIGURE 2.23: Example of the void tracking procedure. Each cavity which has been tracked is shown with the same color in (a),(b) and (c). A cross-section of the tomography volume is shown in (d).

In Fig.2.24, initial cavities are drawn in red and nucleated cavities are drawn in grey. Fig.2.24(b) shows the material at the beginning of plasticity and almost all cavities are in red. During straining of the specimen, void nucleation occurs and more and more grey cavities are observed in Fig.2.24(c) to (f). In case of coalescence between an initial cavity and a nucleated void, the resulting cavity has been drawn in red. This has the effect to overstate the void contribution from the initial porosity but has the advantage to highlight its contribution on the damage percolation. For example, a subregion which contains an higher initial porosity has been surrounded in Fig.2.24(b). It appears clearly that the large crack observed in Fig.2.24(e) and (f) has been formed from this subregion with the larger initial porosity. However, it should be noted that the tracking has been performed for the Al 6056

alloy in overaged condition ($\sigma_0 \approx 120$ MPa), i.e. the case for which initial porosity has the biggest impact. Indeed, when void nucleation is a dominant phase (in the T6 case for example), the impact of the initial porosity is attenuated.

2.9. Void tracking and effect of initial porosity

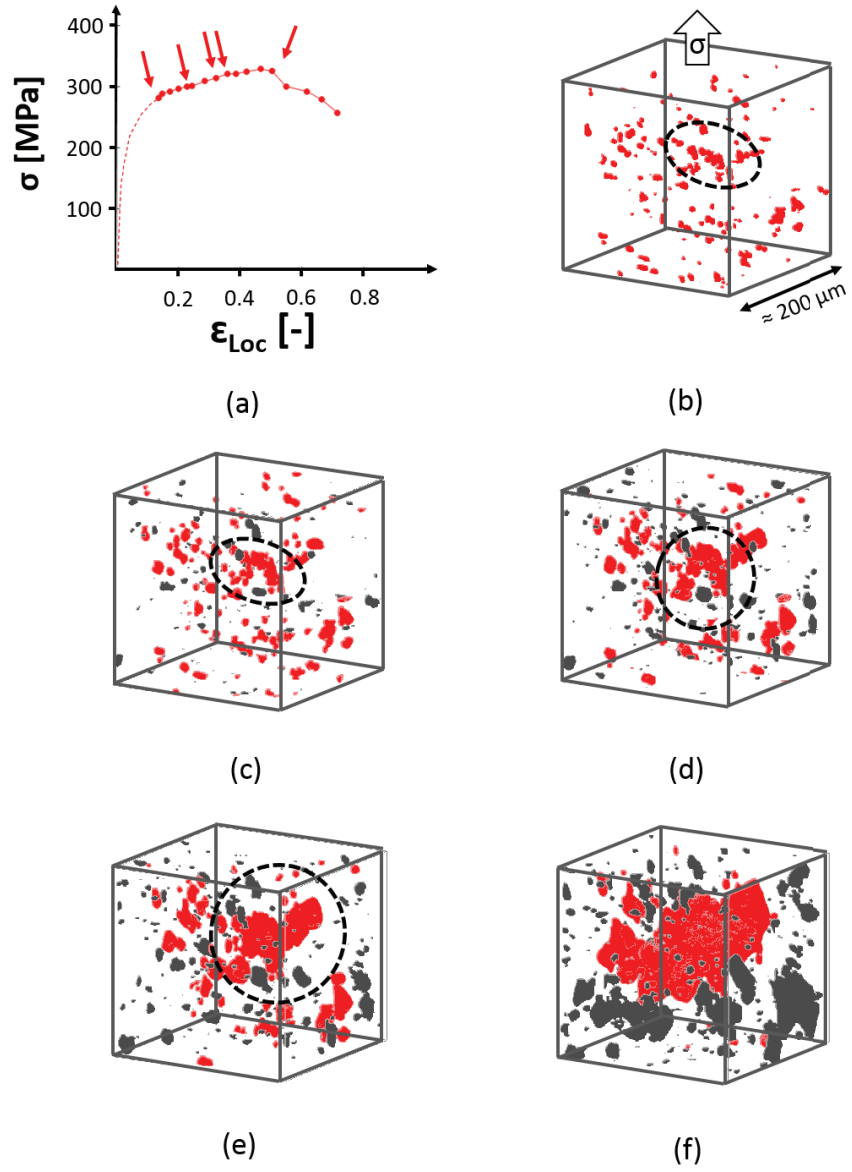


FIGURE 2.24: Vizualisation of the tracking procedure in the Al 6056. Initial voids are shown in red and nucleated voids are shown in grey.

Chapter 3

Ductilization by friction stir processing

This chapter is based on the published article *Ductilization of aluminium alloy 6056 by friction stir processing*, F. Hannard, S. Castin, E. Maire, R. Mokso, T. Pardoen, A. Simar, *Acta Materialia* 130 (2017) 121-136. The last section is not published.

3.1 Introduction

Ductile fracture is characterized by the nucleation of microvoids, followed by stable growth until coalescence of neighboring voids [161, 162, 163, 152, 164, 12]. The accumulation of void linkage leads to the formation of a macroscopic crack that finally propagates until final failure. Micron-sized iron-rich intermetallic particles constitute the main source of damage in Al alloys. These particles fracture or undergo interface decohesion [164, 12, 30, 145]. Additionally, voids may be already present from processing or from hydrogen or can also nucleate, usually at large strains, on smaller size dispersoids [152]. Factors like particle clustering, particle morphology and size have a first order impact on the fracture strain [12, 8]. It remains an important scientific and technological challenge to improve the ductility of Al alloys especially in high strength condition resulting from peak aging heat treatments.

Friction stir welding (FSW) is a solid-state welding process developed to bypass limitations associated with local melting and solidification involved in most conventional welding processes. It is particularly suitable for high strength Al alloys [124, 125]. Friction stir processing (FSP) is a process derived from FSW, involving a non-consumable tool with complex features to locally modify the microstructure through solid state mixing. The rotating tool penetrates the material until the shoulder gets into contact with the upper surface and is then displaced along the surface at a controlled velocity and rotation speed (see Fig. 3.1(a)). The heat generated by friction and deformation leads to a malleable state that promotes material flow from the front to the back of the tool and around the pin. The strain level during FSP is in the 5 to 10 range and the strain rate level is on the order of 10 to 400 s^{-1} [165]. FSP leads to a decrease of the grain size by dynamic recrystallization [166]. Kumar et al. [167] have shown a grain size reduction by a factor close to 40 in an Al-Mg-Sc alloy. These fine grains tend to be unstable during subsequent solution heat treatment, typically above 450°C in Al7010 [168]. Abnormal grain growth is likely to occur at high temperature in grains free of dislocations or free of pinning phases [168, 169]. Grains as large as a few millimeters have been reported in FSPed 7xxx series [168, 169] and cast Al alloys [170].

FSP has already been applied to cast Al alloy for the sake of im-

proving properties. Cast Al alloys present large amount of porosities due to gas involved in the molten state, or due to solidification shrinkage defects. FSP has been shown to decrease the microporosity [171, 172, 173, 174, 175, 176, 177, 178, 179] owing to the large pressure applied by the tool at high temperatures, e.g. 0.1% to 0.02% in A356 [173]. Such porosity reduction significantly improves the ductility [173, 178, 180]. The cast microstructure is also homogenized and refined by FSP due to the redistribution, erosion and breakage of large particles [173, 175, 181] improving the strength and ductility [178, 182], e.g. a total elongation improvement from 1.1% to 20% in Al-7Si-0.5Mg [178]. The fatigue performances of FSPed cast materials are also impressively improved [171, 183, 184, 185], e.g. 80% increase of fatigue stress level in a cast A356 alloy [171].

The application of FSP to improve wrought Al alloy has not yet been widely studied. In wrought alloy the amount of initial porosity is much smaller than in cast alloys, but the potential for improvement could come from the impact on the intermetallic particles. Cavaliere [186] has shown a promising increase in fatigue resistance for a 2014 Al alloy. Nevertheless, to the best of the authors knowledge, no detailed analysis is available yet in the literature about the impact of FSP on damage resistance of wrought Al alloys. In a previous study [12], we characterized and modelled the ductile failure process of three 6xxx series Al alloys (6005A, 6061 and 6056). The key element controlling the magnitude of the fracture strain in these alloys was the effect of particle size distribution and spatial distribution on the void nucleation and coalescence process. The presence of pre-existing voids has also been shown to have a first order effect on the fracture strain.

The goal of the present study is to investigate, based on microtomography and detailed image analysis, the potential of friction stir processing (FSP) to improve the ductility of Al 6056 plates through the fracture of the large second phase particles into smaller and more resistant fragments, the closing of the existing initial porosity and the homogenization of the particle spatial distribution.

3.1. Introduction

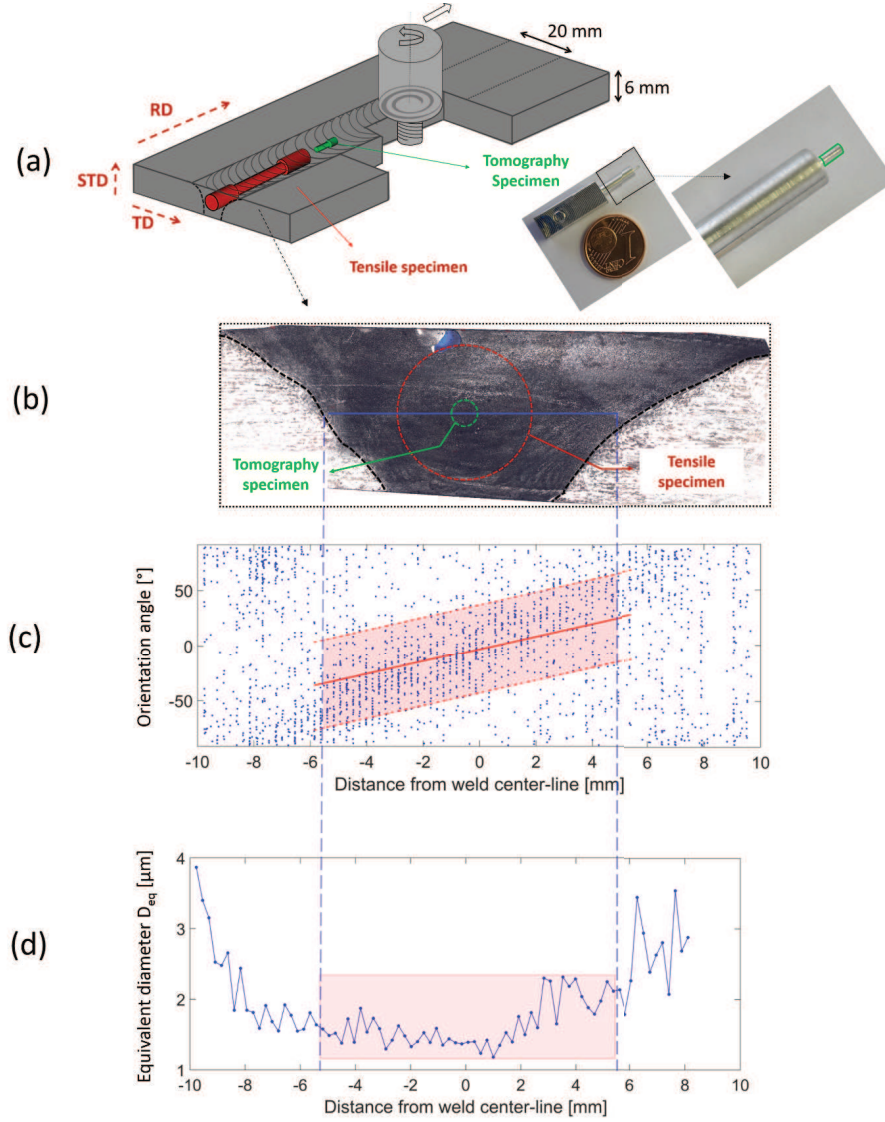


FIGURE 3.1: (a) Schematic of the FSP configuration and the geometry of extracted tensile and tomography specimens, (b) micrograph of the cross-section of the weld in a plane perpendicular to the processing direction with visualization of the tensile (red) and tomography (green) specimens location, the SEM micrographs are taken in the RD-TD plane represented by the blue line in the cross-section. (c) Angle between the major axis of the particle and the transverse direction and (d) equivalent diameter of the particles observed on the SEM micrographs.

3.2 Materials and experimental methods

3.2.1 Materials and FSP

Cold rolled 6 mm thick plates of Al 6056 were used in T4 initial condition. Up to six overlapping FSP passes (see Fig. 3.1(a)), were performed with a tool rotation rate of 500 rpm and a traverse speed of 200 mm/min along the rolling direction (RD) (except for the case of six passes performed also in the transverse direction (TD)). The tool was composed of a 20 mm diameter scrolled shoulder prolonged by a 5 mm long cylindrical M6 threaded Triflat pin, see Ref. [181]. After FSP, all samples were kept at room temperature for at least 1 month to allow natural ageing. The as-FSPed samples were then heat treated at 180°C, and some of them at 350°C as well. Heat treatments were systematically followed by water quenching.

3.2.2 SEM and X-ray microtomography

Cross-sectioning of the weld along a plane perpendicular to the FSP direction was performed for metallographic observations (Fig. 3.1). After standard polishing, the intermetallic particles were characterized based on images taken with a Field Emission Gun Scanning Electron Microscope (FEG-SEM) operated at 15 kV under electron backscattered mode and analyzed using the software ImageJ [187]. X-ray synchrotron microtomography were performed at the TOMCAT beamline of the Swiss Light Source [153]. The sample is probed by X-ray beam tuned to 20 keV using a double crystal multilayer monochromator. 2160 projections were recorded in absorption mode using 16 bit PCO.edge CMOS detector coupled with a 40X optical magnifying lens to a 5.9 micrometer thick LSO:Tb scintillator. The observed region is cuboid in shape, with dimensions $265 \mu\text{m} \times 265 \mu\text{m} \times 160 \mu\text{m}$, and isotropic voxel size of $160 \times 160 \times 160 \text{ nm}^3$. Intermetallic particles and cavities observed in the reconstructed volume were segmented by manual thresholding. Labelling and parameters measurement was then performed using a dedicated image processing plugin implemented in ImageJ [188]. The shape and the orientation of the particles are defined based on the best-fitting ellipsoid with its three semi-axes R_1, R_2, R_3 ($R_1 \geq R_2 \geq R_3$). The

3.2. Materials and experimental methods

particle aspect ratio W_p is defined as $W_p = R_1 / \sqrt{(R_2 R_3)}$ and the orientation of the particle is defined by the angle between the major semi-axes R_1 and TD (see Fig. 3.1a).

3.2.3 Uniaxial tensile tests

Uniaxial tensile tests were performed on a screw-driven universal machine under displacement control with 1 mm/min velocity. Cylindrical specimens, 4 mm in diameter, were machined along the FSP direction and along RD and TD for the base material (BM), see Fig. 3.1a. The initial gauge length was equal to 30 mm. The initial yield stress σ_0 is extracted from the stress-strain curves. The true fracture strain defined as $\varepsilon_f = \ln(A_0 / A_f)$ is determined from broken specimens, with A_0 and A_f being respectively the initial and final cross-section area.

A number of tensile tests on specimens extracted from the BM and from the six passes FSPed material were interrupted prior to necking and polished down to mid-thickness along the transverse section in order to analyze the fraction of damaged particles. If the particle is broken, its surface is reconstructed by manually merging all fragments together. Approximately 2,000 particles have been analyzed for each interrupted test.

One interrupted tensile test of the BM loaded in RD and one of the FSPed material (6 passes) have also been characterized by microtomography. These tensile tests were both interrupted at a stress level of approximately 400 MPa. The samples have been extracted in the center of the specimens, see Fig. 3.2. A method for reconstruction of broken particles observed within the 3D images is proposed in order to quantitatively study the fragmentation of each particle, see Fig. 3.2. After segmentation of the particles, a second segmentation of the cracks appearing in the broken particles was performed. These cracks are then dilated and any particles which are connected through a crack, i.e. particles which share at least one voxel with one same crack, were merged into a single particle. However it has only been possible to apply the reconstruction algorithm on the sample extracted from BM. Indeed, it was not possible to perform such segmentation for the broken particles in the FSPed material due to the very small size of the particles and hence, of their cracks.

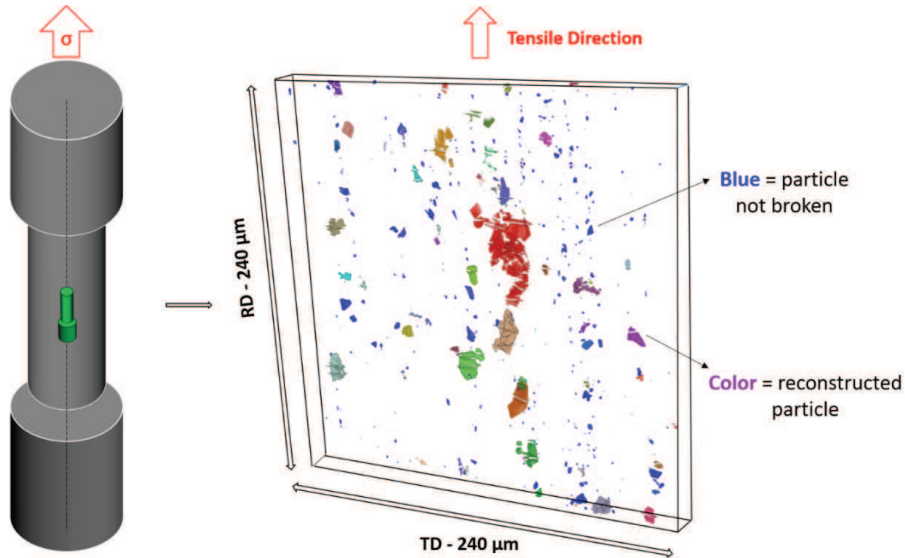


FIGURE 3.2: Schematic of a tensile sample loaded in the RD up to 420 MPa (left figure) and visualization of a sub-volume (right figure) extracted from the total volume scanned by tomography in the base material after automatized particle reconstruction. The unbroken particles are in blue and the fragments corresponding to a fragmented and further reconstructed particle are shown with the other colors (one colour per particle).

3.3 Results and discussion

3.3.1 Intermetallic particles

Fig. 3.3 shows 3D perspective of intermetallic particles as observed by microtomography. The volume fraction of particles F_p is always $\approx 0.5\%$. Indeed, FSP breaks-up the intermetallic particles but does not dissolve them. The BM contains some very large particles and the particles are preferentially aligned with RD. After 1 FSP pass, some large particles still remain intact but their spatial distribution is modified. After 3 and 6 passes of FSP, the mean particle size is clearly reduced and the spatial distribution gets more homogeneous.

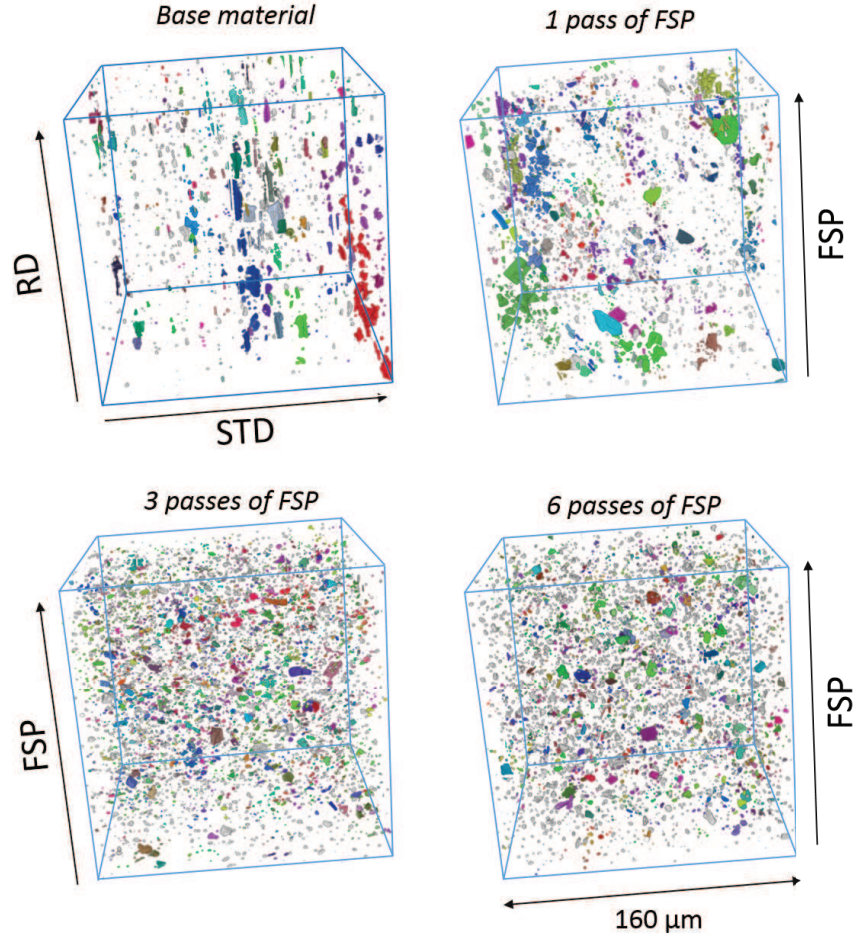


FIGURE 3.3: 3D microtomography perspectives of intermetallic particles distribution for the 6056 base material and for 1, 3 or 6 passes of FSP. White particles correspond to isolated particles, and particles with the same color belong to the same cluster according to the definition given in section 3.1. RD = rolling direction, STD = short transverse direction.

3.3.1.1 Intermetallic particles size, shape and orientation

Fig. 3.4 presents the cumulative size distribution (volume-weighted) for the intermetallic particles based on 3D X-ray microtomography images (see section 3.2.2) as a function of the number of FSP passes (N_{FSP}). The equivalent diameter D_{eq} corresponds to the diame-

ter of a sphere with the same volume as the particle. The fracture and abrasion of large brittle particles is known to depend on the nature of the pre-existing flaws in these particles and thus on their volume, since the largest defects are more likely to be found in the largest particles [12, 189]. The stress level within the particle depends on the flow stress of the matrix, on the aspect ratio of the particles W_p [34] as well as on the local particle volume fraction (due to cluster-induced stress concentration [43]).

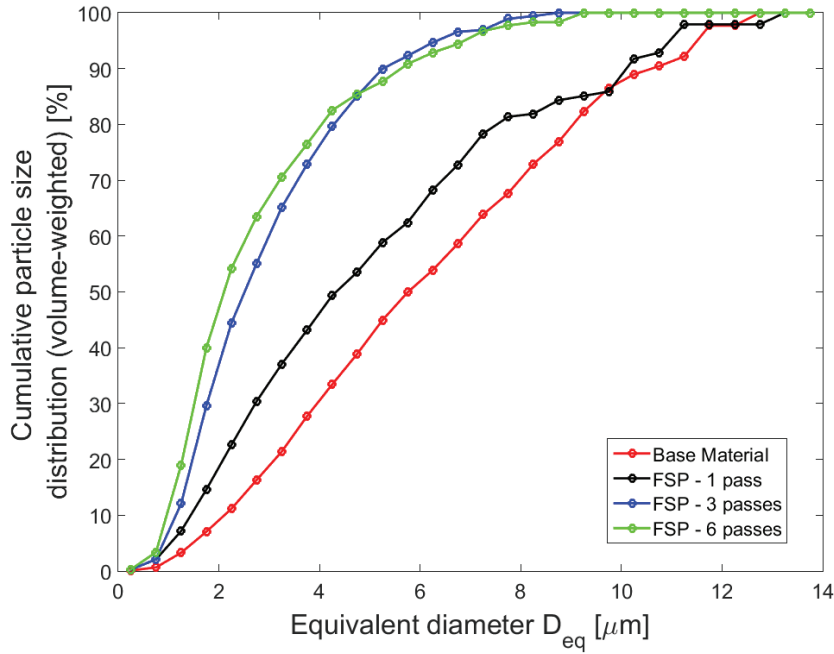


FIGURE 3.4: Cumulative particle size distribution (volume-weighted) for the intermetallic particles based on the 3D X-ray microtomography images.

Fig. 3.5(a) presents the volume-weighted aspect ratio distribution of the particles. FSP breaks the large particles into smaller and more rounded fragments; i.e. the proportion of particles with an aspect ratio close to one increases with N_{FSP} . This trend is explained by Fig. 3.5(b). In the BM, the coarse particles are more elongated, i.e. D_{eq} increases with W_p . Since the large particles are more likely to contain defects, they are more prone to break and the proportion of elongated particles decreases with increasing N_{FSP} . Fig. 3.5(c) and Fig. 3.5(d) illustrate that

3.3. Results and discussion

the large and elongated particles in the BM tend to align along RD, i.e. have an angle close to 90° with TD. Again, since these large particles are more prone to break, this preferred orientation is progressively eliminated with the increase of FSP passes. A minimum of 3 passes of FSP is required to generate significant changes.

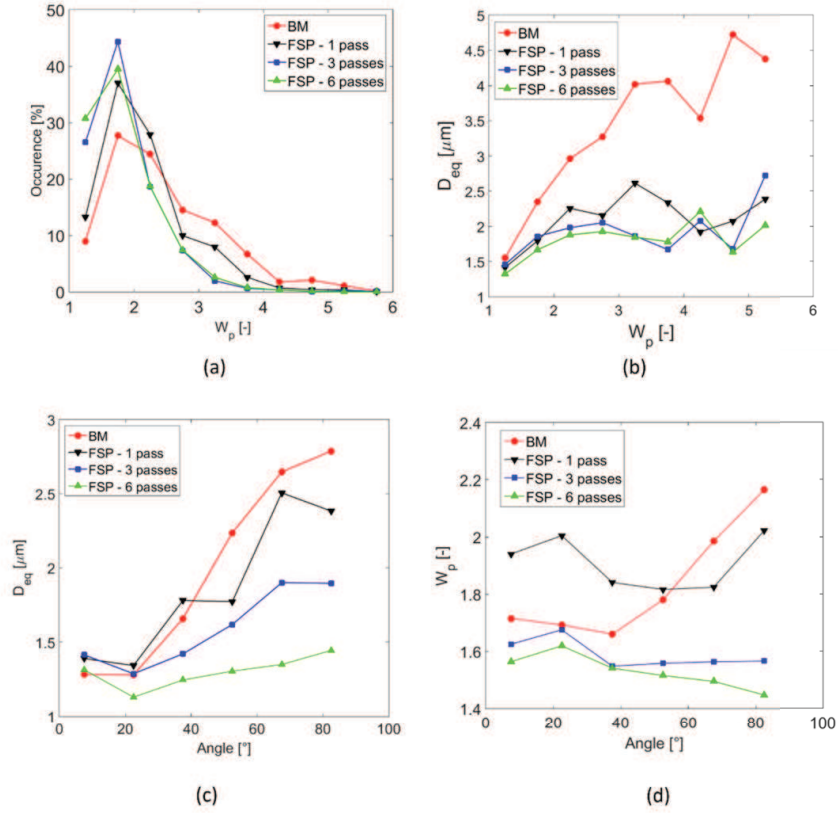


FIGURE 3.5: Morphological parameters of the particles for different number of FSP passes: (a) Distribution of the aspect ratio; (b) Equivalent diameter (D_{eq}) as a function of the aspect ratio (W_p); (c) Equivalent diameter (D_{eq}) as a function of the angle between the major axis of the particle and the transverse direction (TD); (d) Aspect ratio as a function of the angle between the major axis of the particle and the transverse direction (TD). BM = base material.

3.3.1.2 Spatial distribution of intermetallic particles

a) Short-range neighboring A 3D "finite body" tessellation [190, 191] was applied on the microtomography images in order to quantify the spatial distributions of the particles (voids are excluded). Indeed, a more classical Voronoi tessellation based only on the centroids of the particles is not well suited for particles with irregular shapes [190]. The finite body tessellation consists in the following procedure: (i) segmentation by thresholding and labelling of the particles, (ii) computation of the 3D Euclidean Distance Map (EDM), (iii) computation of the 3D watershed from the EDM. The procedure is illustrated in 2D in Fig. 3.6(a – c) for clarity and its result is shown in 3D in Fig. 3.6(d). It results in a network of cells such that every point within a cell is closer to the corresponding particle than to any other [190, 191].

Indicators of the distribution homogeneity are computed based on post-processing of the tessellated cells. The local volume fraction F_p^{local} is defined as the ratio of the particle volume to the volume of its associated cell. Neighbors are defined as the particles associated with cells that directly share one face with the cell of the given particle. The nearest-neighbor d_{nn} and near-neighbor d_n distances can then easily be calculated as respectively the minimum and mean distance between the particle and all neighbors.

The line entitled "All particles" in Table 3.1 compares the mean F_p^{local} of all cells. The standard deviation of F_p^{local} for all particles is given also as an indicator of the degree of homogeneity of the distribution [192]. As expected, the mean value of F_p^{local} is close to 0.5% in each case, i.e. similar to the global volume fraction. Indeed, FSP breaks-up the intermetallic particles but does not dissolve them and the global volume fraction remains unchanged. If compared with BM, the standard deviation of F_p^{local} is slightly higher for one pass of FSP and then reduced for 3 and 6 passes. This is a first indication that the degree of clustering is reduced by FSP, but only after a minimum of 3 passes.

Fig. 3.7 gives the distribution of d_{nn} and d_n as a function of N_{FSP} . All distances given in the following analysis correspond to true particle spacing (the size of the ligament of matrix), i.e. the minimum Euclidian distance between two voxels from each particle, and not the distance from centroid to centroid. Section 3.3.1.1 demonstrated that large in-

3.3. Results and discussion

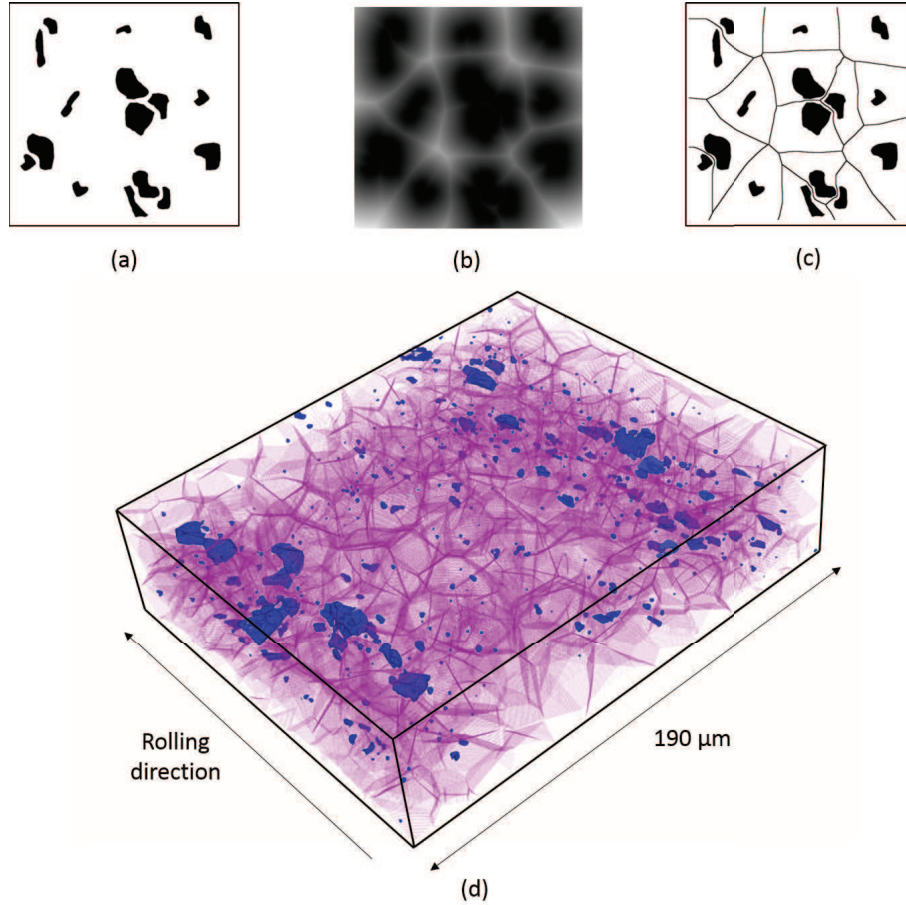


FIGURE 3.6: Example in 2D of the (a) segmented particles field, (b) distance map of the labelled particles with the distance visualized by greyscale, and (c) watershed cells and the particles superposed. (d) Visualization of the result of the procedure applied in 3D to a sub-box of the microtomography image of the base material where particles are in blue and cells are in purple.

termetallic particles are fragmented by successive FSP passes. It means that the number of particles per unit volume ρ increases and thus that the mean distance between particles decreases. In order to compare the homogeneity of the distribution on a relative basis, the measured distances have been normalized by a factor $\rho^{-1/3}$. The "occurrence" is calculated as the ratio of the population in the class divided by the total number of particles. The distribution of d_{nn} in BM shows that a lot of

Chap. 3: Ductilization by friction stir processing

		Base Metal	1 pass of FSP	3 passes of FSP	6 passes of FSP
Mean local volume fraction F_p	All particles	$0.40 \pm 0.96 \%$	$0.50 \pm 1.1 \%$	$0.46 \pm 0.64 \%$	$0.51 \pm 0.63 \%$
	Particles within clusters	0.62%	0.93%	0.55%	0.6%
	Isolated particles	0.15%	0.22%	0.3%	0.37%

TABLE 3.1: Local volume fraction resulting from the finite body tessellation ("All particles") and the clustering analysis ("Particles within clusters" and "isolated particles").

particles have at least one neighbor in very close proximity. This corresponds to particles located within a cluster. On the opposite side of the distribution, a small fraction of particles are quite distant from all their neighbors. This corresponds to isolated particles. As N_{FSP} increases, more and more particles have a normalized d_{nn} around 0.5. As detailed by Chandrasekar [193], the expected normalized d_{nn} in a perfectly random distribution of particles should be equal to 0.554. This indicates that the distribution of particles gets closer to a random distribution when N_{FSP} increases. The distribution of d_n (see Fig. 3.7(b)) shows that in the BM and after one FSP pass, a large proportion of particles have a mean normalized distance to their neighbors significantly smaller or significantly larger than one. Again, this corresponds respectively to particles outside and within clusters. After three and six FSP passes, the distribution becomes more homogeneous.

b) Clustering analysis In order to understand the influence of clusters on the damage evolution, one should be able to distinguish clusters from the isolated particles [194, 195]. An analysis proposed by Achon et al [52] was used here to further characterize the clusters within the 3D particle field observed by microtomography (see the supplementary material in section 3.6 for more details on Achon's method). A particle is assumed to belong to a cluster if all of near-neighbors identified from the tessellated particle field are within a distance $d_{Cluster}$ or less. The distance $d_{Cluster}$ should be an objective characteristic of the particles spatial distribution. If the distance $d_{Cluster}$ increases, the number of clusters increases. At some point, the number of identified clusters starts to decrease because the distance $d_{Cluster}$ becomes so large that the clusters coalesce with one another. The "clustering distance" of the distribution selected here is the value of $d_{Cluster}$ corresponding to the maximum number of clusters identified within the particle field.

This procedure distinguishes isolated particles, i.e. particles that

3.3. Results and discussion

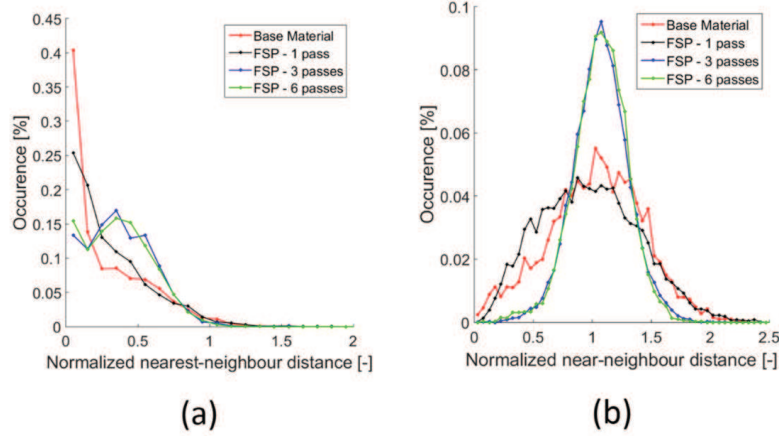


FIGURE 3.7: Distribution of (a) nearest-neighbour distances and (b) nearest-neighbour distances as a function of the number of FSP passes. Distances have been normalized by $\rho^{-1/3}$, with ρ the number of particles per unit volume. The “occurrence” is calculated as the ratio of the population in the class divided by the total number of particles.

are distant from all their neighbors by a distance larger than $d_{Cluster}$, from particles belonging to clusters. However, some clusters contain a low number of particles and no physical meaning in the sense that they could be found in a random distribution of particles. These random clusters are identified in the following manner: each particle is randomly relocated within the analyzed volume, with no overlap allowed. The analysis is then performed on this random distribution with the selected clustering distance. The largest number of particles which belong to a similar cluster within the random distribution is defined as the minimum cluster size, i.e. the mean number of particles per cluster. In other words, any particle belonging to a cluster composed of fewer particles than the minimum cluster size is considered as isolated.

Table 3.2 summarizes the main indicators resulting from the clustering analysis carried out on the microtomography images. The selected clustering distance decreases with N_{FSP} . Indeed, more and more particles are fragmented and the number of particles per unit volume increases. The mean distance between particles decreases and the clusters start coalescing with each other for a smaller distance. The mean cluster size first increases for 1 FSP pass and then decreases for 3 and

Chap. 3: Ductilization by friction stir processing

		Base Metal	1 pass of FSP	3 passes of FSP	6 passes of FSP
Selected clustering distance : $d_{cluster}$		6.4 μm	4.65 μm	4.3 μm	4.0 μm
Mean size of clusters		26 particles	35 particles	20 particles	15 particles
Percentage of particles within clusters	volume-weighted	67.2 %	61.4 %	32.2 %	15.6 %
	number-weighted	40.3 %	44.3 %	25 %	10.7 %

TABLE 3.2: Indicators resulting from the clustering analysis.

6 passes compared to the BM. The percentage of particles within clusters decreases with N_{FSP} . Note that the volume-weighted percentage of particles within clusters is higher than the number-weighted percentage in every case. This means that the large particles tend to be more clustered. These large particles are more prone to fragmentation and this will contribute to the reduction of clustering. The analysis differentiates isolated particles from particles within clusters, and the mean local particle volume fraction F_p^{local} of cells which belong to clusters compared to those of isolated particles is given in Table 1. Clusters correspond to a higher F_p^{local} than isolated particles. The mean local particle fraction increases with N_{FSP} for isolated particles. Because the particle distribution is progressively homogenized and more and more cells have a F_p^{local} similar to the global value. The mean F_p^{local} within clusters increases after 1 FSP pass, in agreement with the increased size of clusters, as large particles found in the BM break into smaller fragments. However, the redistribution remains local and is not sufficient to displace them far away from the parent particles. These fragments contribute to increase the cluster length to which the parent particle belongs and increase F_p^{local} after one FSP pass.

c) Long-range neighboring One drawback of the nearest neighbor analysis is that it places more emphasis on the nearby particles and ignores the longer range characteristics of the arrangement. A classical way to analyze medium to large particles spacing is to use the radial distribution function (RDF), i.e. the probability for a neighboring particle to be located at a distance r , in any direction [196]. However, clusters in the BM exhibit anisotropic shape with particles preferentially aligned in the RD. As a consequence the RDF analysis needs to be modified in order to take into account both distance and angular dependence of the particles spacing. The extended pair correlation function (PCF) already proposed in Refs. [196, 197] is slightly modified here and directly applied to the 3D X-ray microtomography images. The PCF function

is defined, in 3D, as the probability for a given point $[x,y,z]$ to be located inside a particle, given that another particle is located at the origin $[0,0,0]$. Fig. 3.8 shows how the PCF function can be calculated for a 3D volume extracted from the BM. The procedure is described in the supplementary material (in section 3.6).

For a perfectly homogeneous spatial distribution, the PCF would be equal to 1 for each point. A PCF value smaller or larger than 1 corresponds thus respectively to a smaller or larger probability to find a neighbor in the vicinity of a particle compared to a perfectly homogeneous distribution. The origin in the PCF plots is at the center and has a value equal to 0 because the point has to fall in a neighboring particle and not in the reference particle.

In order to interpret the PCF, Fig. 3.8(a) shows the 2D cross-section through the center of the sub-volume in the plane RD-TD, TD-STD and RD-STD for the BM. The area with the highest PCF is parallel to the RD, due to the stringer-type clustering along RD, as already qualitatively observed. On the opposite, the PCF is observed to be smaller in the TD. Fig. 3.8(b) shows the RD-TD cross-section as a function of NFSP. FSP randomizes the particles distribution and make the spatial distribution of the particles in the material more isotropic. After one FSP pass, regions of space with high PCF are still observed, but the clustering is not anymore strongly oriented along the RD compared to the BM. This indicates that clusters of particles are still found after 1 FSP pass, but these clusters have a more isotropic shape. After 3 and 6 FSP passes, the PCF approximately ranges in the interval $[0, 2]$ at each point of space, due to the strong declustering.

3.3.2 Initial porosity

Figs. 3.9(a) and 3.9(b) respectively present the evolution of the volume fraction f_0 and mean equivalent diameter of the porosity initially present, i.e. prior to testing. The initial porosity originates from intermetallic particles fracture during the rolling process [194] (see Fig. 3.9(c)) or from the gathering, during Al solidification, of the hydrogen dissolved in liquid Al [117] (see Fig. 3.9(d)). FSP significantly reduces the porosity volume fraction (see Fig. 3.9(a)) and the mean equivalent diameter of the voids (see Fig. 3.9(b)). The closure of the

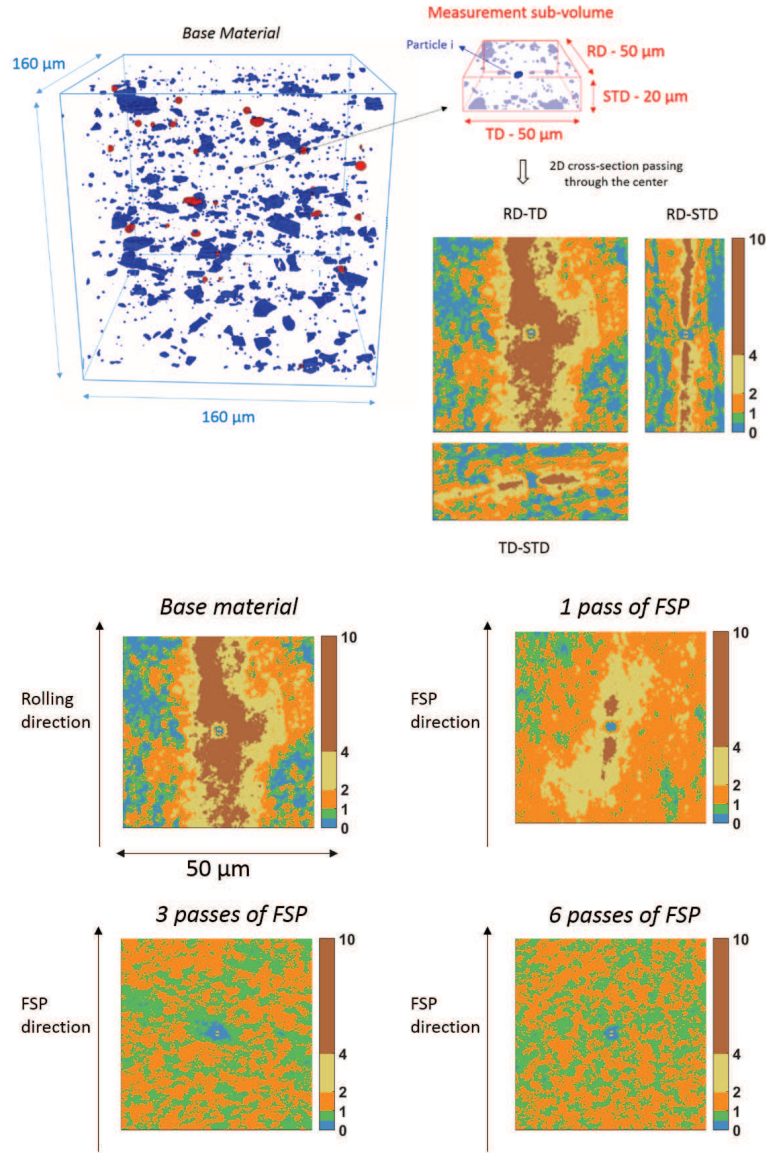


FIGURE 3.8: (a) Principle (left) and result (right) of the PCF analysis for the base material. On the left figure, intermetallic particles are presented in blue and voids in red. (b) Pair correlation functions (PCFs) from experimental images for the RD-TD plane through the center of the sub-volume for the base material and FSPed samples. PCF value smaller or larger than one corresponds respectively to a smaller or larger probability to find a neighbor in the vicinity of a particle compared to a perfectly homogenous distribution.

3.3. Results and discussion

cavities by FSP is probably due to the complex nature of material flow and the high shear strain within the deformed zone [165]. The first steps of the closure of the void due to the material flow after 1 FSP pass is qualitatively presented in Fig. 3.9(e).

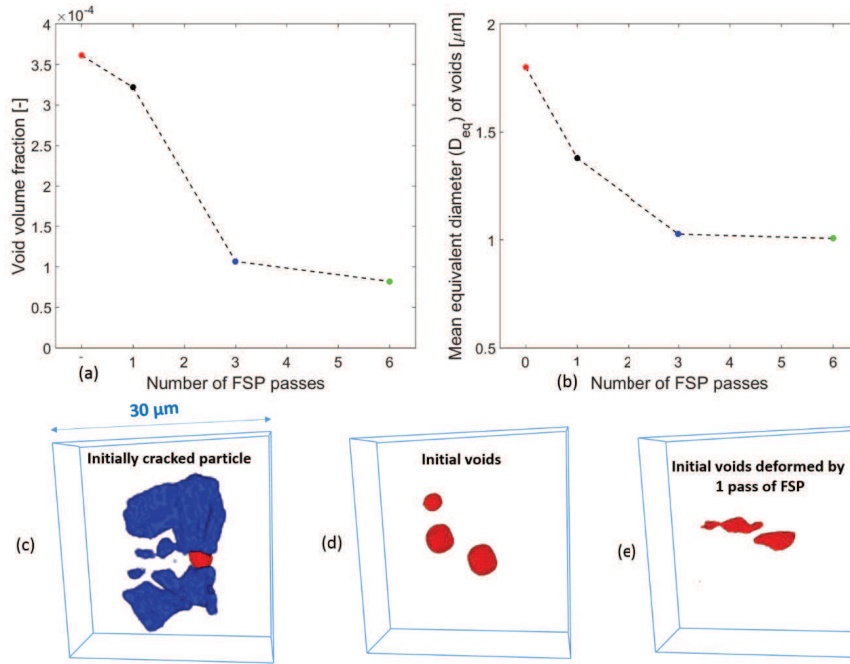


FIGURE 3.9: Evolution of (a) porosity volume fraction and (b) mean equivalent diameter of the voids as a function of the number of FSP passes. 3-D views of (c) an initially broken particle (blue) in the BM, (d) initial porosity in the BM and (e) voids observed after 1 pass of FSP. Cavities are in red.

3.3.3 Summary about microstructural evolution upon FSP

The physical scenario schematically illustrated in Fig. 3.10 emerges from the microstructural characterization presented in the sections 3.3.2 and 3.3.1:

1. In the BM, intermetallic particles are preferentially aligned along RD and this results in a stringer-type clustering. The coarse particles are the most elongated.

2. After one FSP pass, the largest (also elongated) particles are broken into smaller fragments but the redistribution of these fragments remains local. The fragments remain within the cluster to which the parent particle belongs with no reduction of the degree of clustering. However, this local redistribution is sufficient to mitigate the preferred orientation for particle clustering. The aspect ratio of the large intermetallic particles is decreased by FSP due to their breakage perpendicular to their long dimension.
3. After 3 FSP passes, the decrease of particle size progressively saturates. This saturation is due to the fact that the small broken fragments become very difficult to break. The fragments get more homogeneously distributed and the degree of clustering is reduced.
4. Performing more than 3 FSP passes (i.e. 6 FSP passes) provides little improvement in size reduction and homogenization of the distribution.
5. FSP also reduces the initial porosity owing to the extreme applied deformation.

3.3.4 Uniaxial tensile tests

3.3.4.1 Interrupted tensile tests

Fig. 3.11(a) shows the true stress-true strain response of the BM in T4 state in RD and TD, as well as of a FSPed sample after 1h at 180°C of heat treatment. This last heat treatment condition has been selected because it provides almost the same hardening behavior as the T4 BM, hence allowing comparisons of the damage evolution at similar stress levels. The BM exhibits weak plastic anisotropy. Each point on the curves corresponds to a tensile test interrupted prior to necking in order to limit the growth of nucleated voids and to facilitate reconstruction of the broken particles (see section 3.2.3). The variation of the fraction of damaged particles measured on polished cross-sections is given in Fig. 3.11(b) as a function of the applied stress. Particle fracture is the dominant nucleation mechanism in BM. For the

3.3. Results and discussion

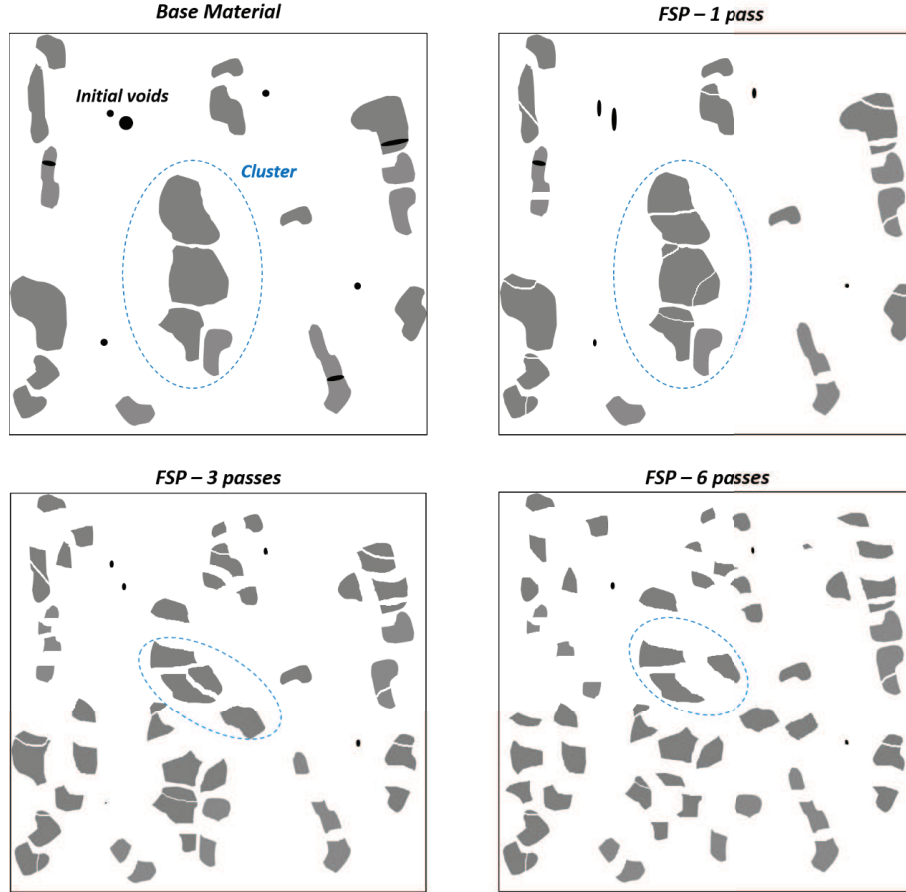


FIGURE 3.10: Schematic scenario of the microstructural evolution with the number of FSP passes.

same macroscopic stress, the fraction of broken particles in BM is significantly higher when loaded along the RD. Indeed, as discussed in section 3.3.1.1, particles are elongated and preferentially aligned along RD, which increases the load transfer arising from the deformation mismatch between the particle and matrix [44]. Furthermore, for the same stress, the fraction of damaged particles drops in the FSPed material. The large and most brittle particles in the BM have been broken into smaller and more resistant fragments in the FSPed material. Void nucleation by interface decohesion is now observed in the 6 passes FSPed material for some particles (12% to 19% of the damaged particles when

the stress increases from 353 MPa to 398 MPa). This could be due to a degraded interface between the fragments and the matrix formed during FSP. It is also likely that because the remaining fragments are the strongest parts of the particles, these are more and more difficult to break. However, particles fracture remains the main nucleation mechanism indicating that for a majority of fragments the adhesion with the matrix remains high.

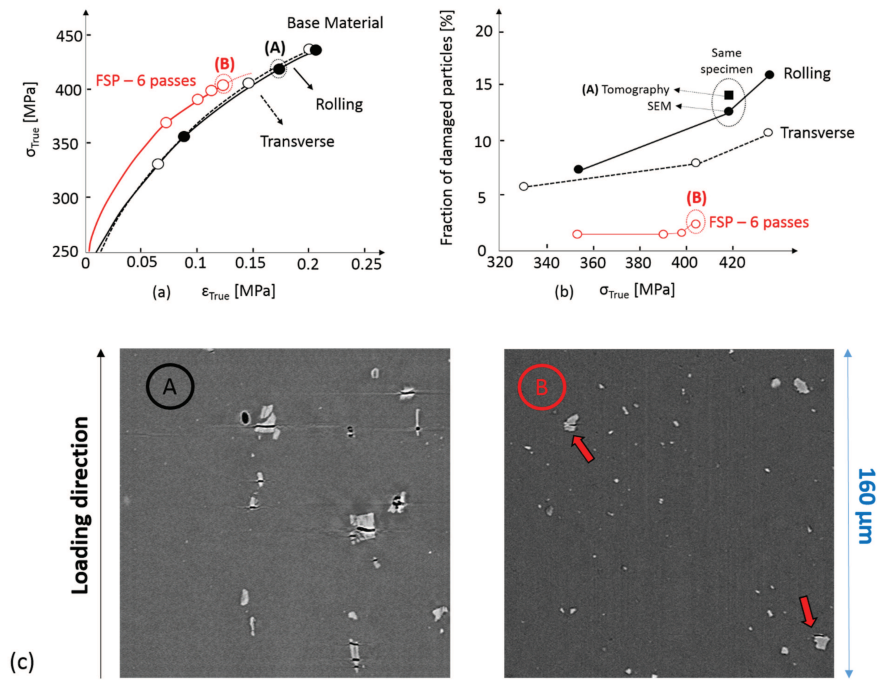


FIGURE 3.11: (a) Tensile curves of the base material loaded in the rolling and transverse direction, and after 6 FSP passes in the rolling direction. The base material is in the T4 state, and the FSPed material has been aged for 1 hour at 180°C. Each point corresponds to an interrupted tensile test and the fraction of damaged particles is given in (b). (c) X-ray microtomography reconstructed images of the two samples (A) and (B).

As explained in section 3.2.3, reconstruction of the broken particles has only been possible on the microtomography images of the BM. Micrographs of the BM and FSPed samples are given in Fig. 3.11(c) for qualitative analysis only. The loading direction corresponds to RD. Large particles preferentially aligned with RD are found within the BM

broken perpendicularly to the loading direction, at more than one location. In the FSPed material, the intermetallic particles are smaller and quite homogeneously distributed within the matrix. The fraction of broken particles is much lower compared to BM and the cracks are formed in the few larger particles not fragmented by FSP, as indicated by arrows in Fig. 3.11(c).

Fig. 3.2 shows a sub-volume extracted from the tomographic volume of BM (loaded in RD up to 420 MPa) obtained after automatized particle reconstruction, see section 3.2.3. Unbroken particles are in blue. Fig. 3.12(a) shows the fraction of broken particles as a function of the reconstructed particles size, obtained from both SEM analysis and microtomography. The differences between the two techniques are probably due to the difference of ratio between resolution and feature size and to the fact that the particles are not perfectly equiaxed. Furthermore, the particle size distribution of the BM (see Fig. 3.4) indicates that a very large fraction of particles has a size ranging between $\approx 1\mu\text{m}$ and $\approx 5\mu\text{m}$. The number of particles larger than $\approx 5\mu\text{m}$ observed in 2D by SEM is thus relatively low. In other words, the fraction of broken particles observed by SEM is less reliable for particles larger than $\approx 5\mu\text{m}$. However, the trends are the same. The microtomography analysis shows that particles with $D_{eq} < \approx 1\mu\text{m}$ are almost never damaged while particles with $D_{eq} > \approx 1\mu\text{m}$ are almost always broken. Fig. 3.12(b) also shows that while most of the particles with $D_{eq} < \approx 3\mu\text{m}$ crack only once, the number of cracks increases with particle size. This indicates that the very large particles, i.e. larger than $\approx 3-4\mu\text{m}$, are the most detrimental as they tend to break into multiple fragments. As shown in Fig. 3.4, the fraction of these large particles is highly reduced after six FSP passes and this strongly reduces the volume fraction of nucleated voids [12].

3.3.4.2 Fracture strain

Fig. 3.13(a) shows, for the BM and for the FSPed material, the variation of the fracture strains (ε_f) as a function of σ_0 corresponding to the various heat treatments. First, ε_f of BM when deformed along RD are compared with ε_f after 3 and 6 FSP passes along RD. For the same σ_0 , ε_f is significantly larger in the FSPed material. For the sake of a better

physical appraisal of the difference, Fig. 3.13(b) presents the same data in terms of engineering strain. It is remarkable that ε_f in TD is doubled after 6 passes. In the LD, ε_f is improved by 20%. This is clearly due to the fact that FSP breaks the large intermetallic particles into smaller and thus stronger fragments (see section 3.3.1.1 and Fig. 3.12), that FSP closes the existing initial porosity (see section 3.3.2 and Fig. 3.9) and that FSP reduces the degree of clustering (see section 3.3.1.2, Table 3.1, Table 3.2 and Fig. 3.9).

While relatively low plastic anisotropy was observed (see section 3.3.4.1 and Fig. 3.9(a)), the anisotropy in ε_f is significant in BM : ε_f is larger by about 0.2 in RD compared to TD. The tessellation analysis on BM revealed that the majority of cells are elongated in RD, indicating that particle clustering is stronger along RD (see section 3.3.1.2). Furthermore, the PCF analysis emphasized a stringer-type clustering along RD, providing an easy percolating crack path when loading in TD which is likely to lower ε_f . A higher fraction of broken particles was observed in BM when loaded along RD, which would be associated with a lower ε_f . This means that the effect of particle spatial distribution on coalescence is the dominant parameter setting the anisotropy in the BM. After 6 FSP passes, the anisotropy in ε_f is suppressed. This is in agreement with the PCF and tessellation analysis, from which it was concluded that FSP randomizes the distribution after a minimum of 3 FSP passes.

After 1 FSP pass along RD, ε_f decreases compared to BM when deformed along RD. Indeed, a lot of large particles (i.e. larger than about $3\ \mu\text{m}$) are still present after 1 FSP pass (see Fig. 3.4). Void nucleation is thus not expected to be significantly postponed. The fact that ε_f even decreases compared to BM can be explained based on the PCF analysis (see Fig. 3.8). Indeed, when deforming BM along RD, the coalescence process is likely to be postponed since the PCF is lower than the one along TD. However, after 1 FSP pass, the anisotropy of the PCF decreases and the coalescence process will be faster.

3.4 Damage model

An elementary 3D damage model is proposed in order to semi-quantitatively uncouple the effect of particle size and particle spatial distribution. The X-ray tomography images of the BM and FSPed material (see Fig. 3.3) are used as representative volume element (RVE), involving the positions (x_i, y_i, z_i) and the dimensions of the particles. The particles are assumed to be ellipsoidal, with main semi-axes R_1 , R_2 and R_3 ($R_1 > R_2 > R_3$) respectively aligned with RD, TD and STD, see section 3.3.1. The RVE is a cube of $160 \mu\text{m}$ side, which corresponds to a particle field ranging between approximately 4,000 particles in BM up to approximately 14,000 particles after 6 FSP passes. The model is applied for the T4 state only, exploiting the experimental data of the interrupted tensile tests (see section=3.3.4.1). The initial yield stress σ_0 , the uniform strain ε_u and the strain hardening parameters are extracted from the true stress–strain curve (in T-direction only) based on an extended Voce law, see Ref. [12]. The same hardening parameters are used in all cases, i.e. neglecting any plastic anisotropy or effect of FSP on the plastic flow behavior (see Fig. 3.11(a)). The presence of an initial porosity f_0 facilitates void linkage and accelerates ductile failure. However, this effect is attenuated for a hard matrix (T4 and T6 state) compared to a soft matrix (over-aged state). Indeed, void nucleation is more significant for particles surrounded by a hard matrix, attenuating the impact of f_0 [12]. The effect of f_0 is thus also neglected in the present analysis focused on the T4 state, see Ref. [12] for a more advanced model.

The fraction of broken particles $P(\sigma_y, V_{mean})$ is assumed to be a function of the flow stress σ_y and of the average particle size V_{mean} . The possible effects of particle size distribution (i.e. the scatter around the mean value) and particle orientation on the nucleation rate are neglected. The experimental fraction of broken particles in the interrupted tensile tests (see Fig. 3.11(b)) is then fitted using the following probability function :

$$P(\sigma_y, V_{mean}) = 1 - \exp \left(-K_1 V_{mean} \sigma_y^{K_s} \right) \quad (3.1)$$

where V_{mean} is the only parameter that depends on N_{FSP} , i.e. the same nucleation stress distribution versus particle size is used. A reason-

able fit of the broken particle fraction for the BM and 6 FSP passes (see Fig. 3.14) is obtained with $K_1 = 3.17e^{-9}$ and $K_2 = 2.7$. The anisotropy of void nucleation in BM remains limited and the same nucleation law gives an acceptable fit for both loading directions. The nucleation process is cumulative and can be seen as a nucleation front moving through the particle size distribution [198]. In other words, for a given fraction of broken particles $P(\sigma_y, V_{mean})$, voids nucleate on particles larger than a critical size V_{crit} . V_{crit} corresponds to the threshold size for which a fraction $P(\sigma_y, V_{mean})$ of particles are larger than V_{crit} . Fig. 3.14 shows that for a given applied stress, the fraction of broken particles decreases with N_{FSP} due to particle size reduction.

The nucleated void is assumed to appear in the middle of the particle and is given by the intersection of the 3D particle with a plane perpendicular to the loading direction. The equivalent radius R_{eq} of the void corresponds to the radius of a circle with the same area as the particle intersection. Void growth is modelled using the Rice and Tracey [64] isotropic void growth analysis corrected by Huang [129]:

$$\frac{dR_{eq}}{R_{eq}} = 0.427T^{1/4}\exp(1.5T)d\varepsilon \quad (3.2)$$

where ε is the effective plastic strain and T is the stress triaxiality given by $T=1/3$ (for $\varepsilon < \varepsilon_u$) and $T = 1/3 + 0.41(\varepsilon - \varepsilon_u)$ (for $\varepsilon > \varepsilon_u$), with ε_u the uniform strain [59]. The Thomason's criterion [60] is used to predict void coalescence and is evaluated for each cavity with all its near-neighbors:

$$\frac{\sigma_n}{\sigma_0} = (1 - \chi_{eff}^2) \left(0.1 \frac{1 - \chi_{eff}^2}{\chi_{eff}} + 1.2 \frac{1}{\sqrt{\chi_{eff}}} \right) \quad (3.3)$$

where σ_n is the stress component normal to the ligament between the voids and χ_{eff} is the effective void spacing. For simplification, the void aspect ratio used in eq. 3.3 is taken equal to 1 (i.e. the two voids are supposed to be spherical) since this assumption was shown in a previous study [12] to have a limited effect on the value of ε_f for this ductile Al alloy. Indeed, a significant amount of void growth occurs before the coalescence criterion is fulfilled. When this happens, the void shape is always close to equiaxed. The overall stress component normal to the ligament σ_n is approximated by $\sigma_n = \sigma_y \sin(\alpha)^2$, with α the angle between the ligament and the loading direction. The effective void spac-

3.4. Damage model

ing χ_{eff} is given by:

$$\chi_{eff} = \frac{R_{eq}^1 + R_{eq}^2}{L_{1-2}} \quad (3.4)$$

where L_{1-2} is the distance between void centers and R_{eq}^1 and R_{eq}^2 are the mean radii. The ratio of the number of cavities satisfying the coalescence criterion over the total number of particles is an indication of the number of microcracks developing within the material. The evolution of this ratio is shown in Fig. 3.15(a) as a function of the applied strain, orientation and N_{FSP} . The vertical lines indicate the experimental ε_f . A ratio of 10% of microcracks compared to the number of particles corresponds reasonably well with the experimental ε_f (see Fig. 3.15(a)). If this critical ratio is chosen as an empirical fracture criterion, the model captures the anisotropy on ε_f for BM. Furthermore, the model predicts isotropic ε_f after 6 FSP passes, owing to the particle distribution randomization (see section 3.3.1.2 and Fig. 3.8). The results of the model for 1 FSP pass indicates a limited improvement of ε_f compared to the BM, in good agreement with the experiments. In the case of 3 FSP passes, ε_f is significantly larger due to highly reduced particle size (see section 3.3.1.1). After 6 FSP passes, ε_f is not much improved because of particle size saturation (see Fig. 3.4) and the similar spatial distribution after 3 and 6 FSP passes (see section 3.3.1.2).

This model allows decoupling the effect of the void size on nucleation and of the spatial distribution on coalescence. Indeed, if the mean size V_{mean} in eq. 3.1 is kept constant and equal to the value of BM (i.e. if all samples have the nucleation law given by the red curve in Fig. 3.14), the effect of the particle size on void nucleation is not taken into account. Any change in ε_f is then related to the particle spatial distribution only. Results of the model without the size effect on void nucleation are provided in Fig. 3.15(b). Without the effect of particle size on the void nucleation, ε_f of the FSPed samples remains between ε_f of BM along RD and TD. Without FSP, the clustering alignment along RD explains the low value of ε_f in TD. This effect disappears after FSP. This analysis shows that the improvement of ductility with N_{FSP} is mainly due to the delay on void nucleation owing to the reduction of particle size.

3.5 Conclusions

Friction stir processing (FSP) has been performed on a 6056 rolled Al alloy. Detailed analysis of 3D high resolution X-ray synchrotron tomography data of the BM and FSP after 1, 3 and 6 FSP passes have provided the following information:

- The intermetallic particle size decreases with the number of FSP passes.
- The large intermetallic particles are also initially more elongated and are rounded up by FSP due to their breakage by the large deformations involved in the process.
- FSP homogenizes the particle distribution, as quantitatively demonstrated by the tessellation, clustering and pair correlation functions analyses.
- The initial porosity present in the BM is first reduced in size and then suppressed by an increasing number of FSP passes.
- A simple FSP pass is not sufficient to ensure the full potential of the process to break the intermetallic particles and to homogenize their distribution.

Tensile testing and interrupted tensile tests of BM and FSPed samples have provided the following key results:

- The fracture strain of FSP samples is improved by almost 100% in the best cases compared to the BM tensile samples at identical yield strength.
- The nucleation stress for the fracture of intermetallic particles (i.e. the source of damage in these alloys) increases in the FSP samples.
- Friction stir processing leads to an isotropic fracture behavior.

A simple damage model has been used to uncouple the effect of particle size and spatial distribution. The homogenization of the particle distribution is the main reason for the isotropy of the FSPed material. The reduction in size of the intermetallic particles, retarding void

3.5. Conclusions

nucleation, explains the improvement of the damage resistance of the FSPed samples. Void nucleation retardation has been quantitatively characterized based on ex situ tensile test in X-ray microtomography. Earlier works have shown that miniature tensile specimens can be used to determine the mechanical and fracture properties of the weld [61,62]. Future work will consider in situ tensile testing of such miniature specimens in order to quantitatively characterize the evolution of the damage process. It will allow validating the effect of FSP on void nucleation and gain insight on its impact on the void growth and the coalescence process.

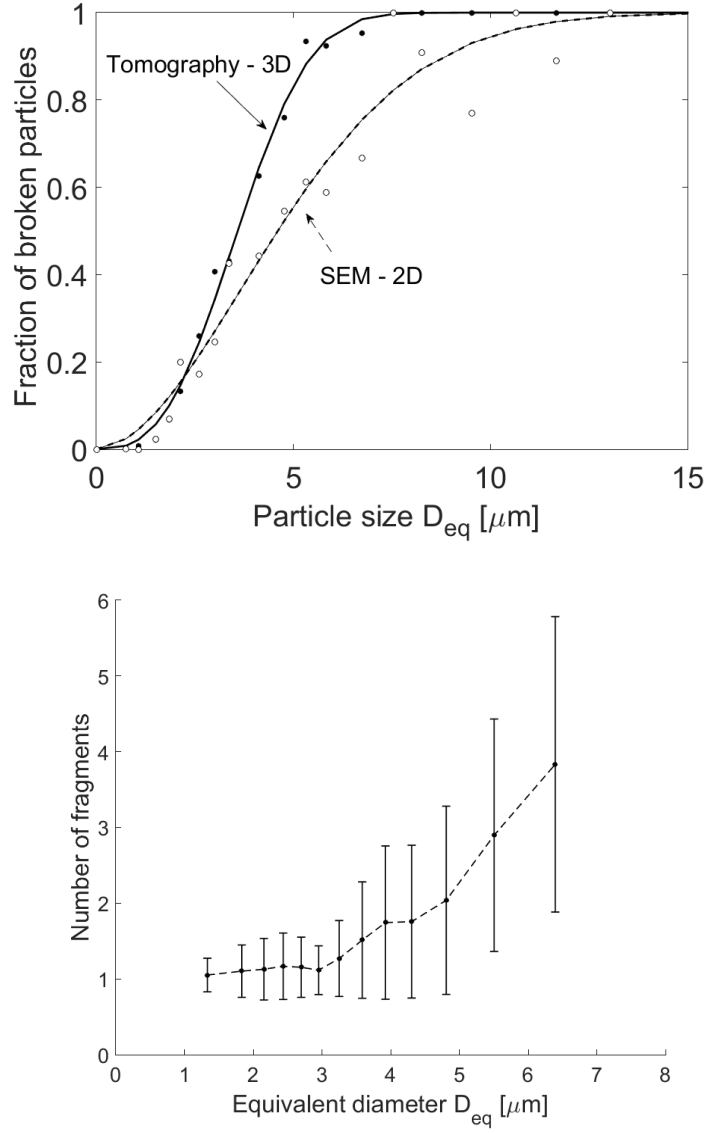


FIGURE 3.12: Results obtained on the interrupted tensile test on BM, in the T4 state, loaded in RD up to 420 MPa and characterized by microtomography: (a) Fraction of broken particles as a function of the equivalent particle diameter (i.e. size) obtained from SEM analysis (over 2,000 particles analyzed) and microtomography (over 10,000 particles analyzed). (b) Number of observed fragments as a function of the size of the broken particles.

3.5. Conclusions

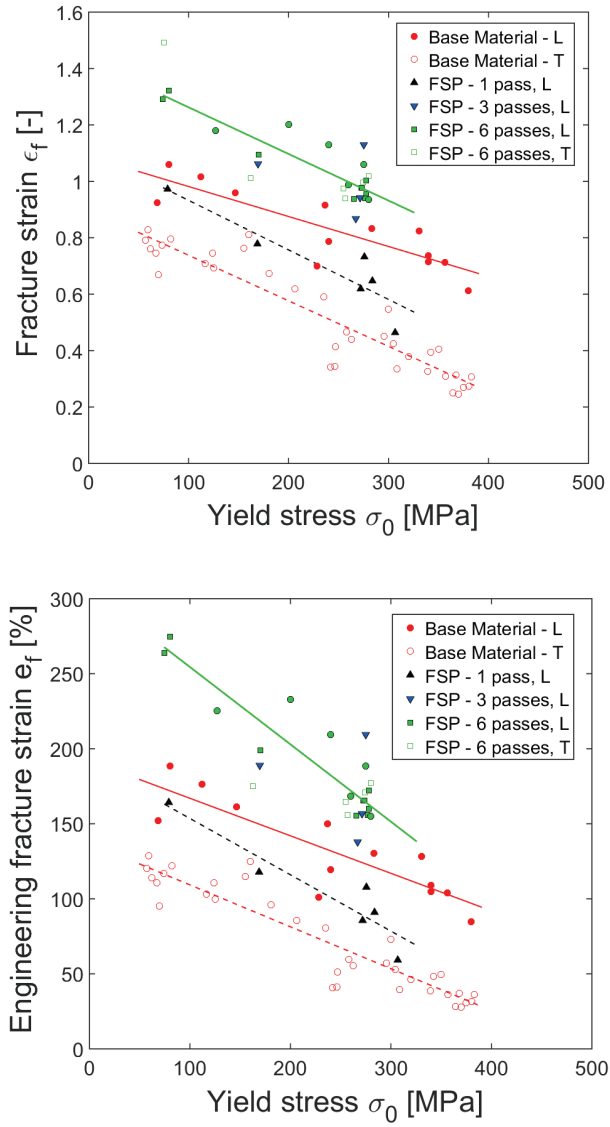


FIGURE 3.13: Variation of the (a) true fracture strains and (b) engineering fracture strain extracted from the uniaxial tensile tests as a function of the yield stress corresponding to the different heat treatments.

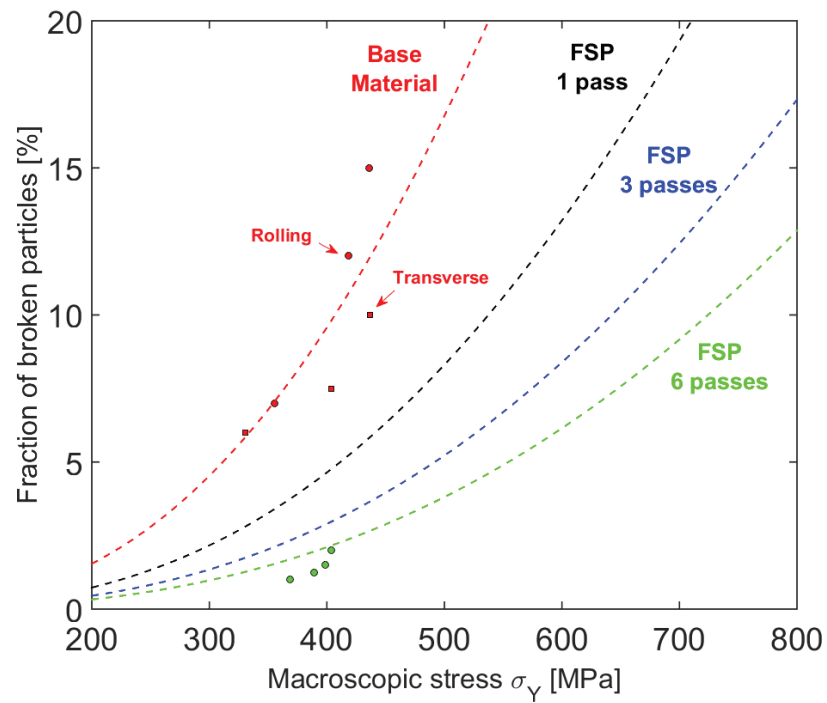


FIGURE 3.14: Fraction of broken particles as a function of the macroscopic flow stress. Comparison of experimental results (points) with the nucleation model (lines).

3.5. Conclusions

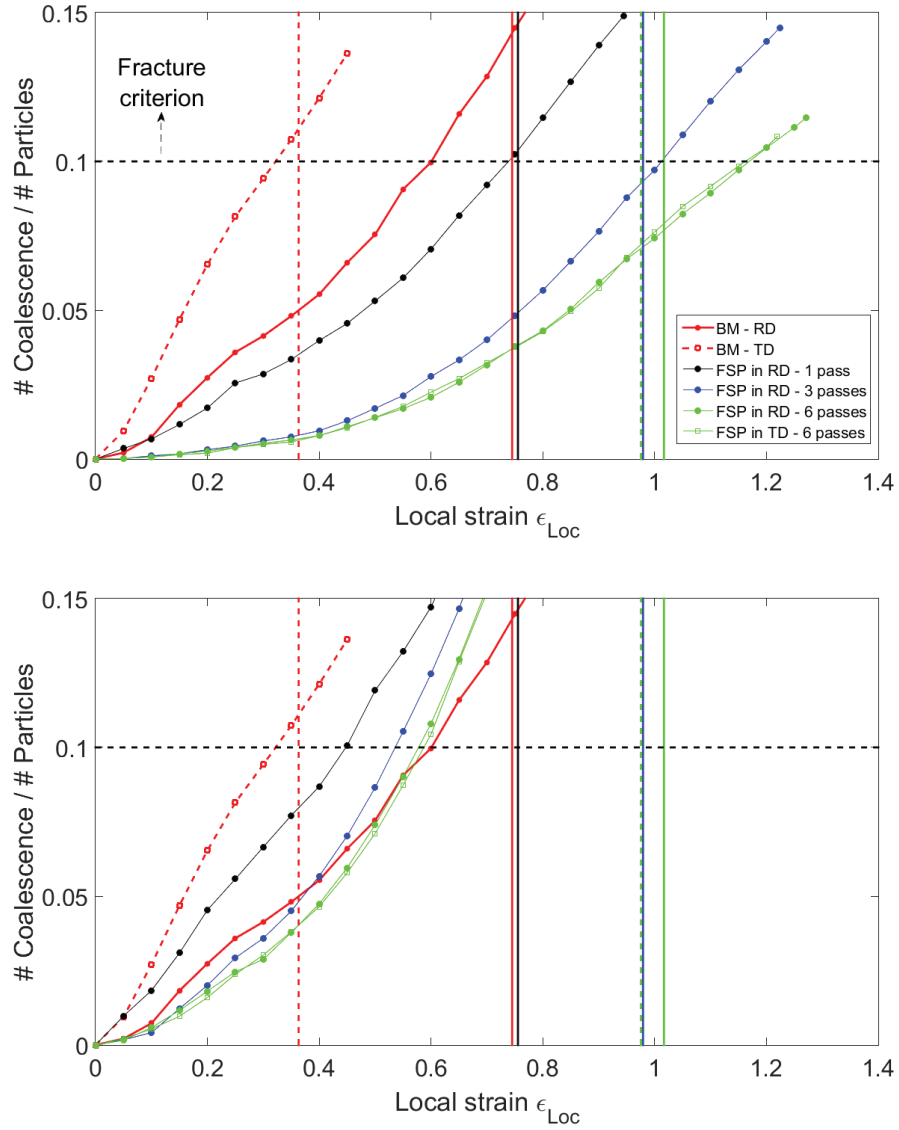


FIGURE 3.15: Results of the model applied on the T4 state. Ratio between the number of cavities couples satisfying the coalescence criterion and the number of particles, for the model (a) with and (b) without the effect of particle size on void nucleation. The vertical lines indicate the experimental fracture strain (ϵ_f).

3.6 Supplementary material to Paper FSP

3.6.1 Pair correlation function

A classical way to analyze medium to large particles spacing is to use the radial distribution function (RDF), i.e. the probability for a neighboring particle to be located at a distance r , in any direction [1]. However, the RDF analysis is not suitable if particles are preferentially aligned in a specific direction and the RDF analysis needs to be extended in order to take into account both distance and angular dependence of the particles spacing. This extended pair correlation function (PCF) proposed in Refs. [1, 2] is slightly modified here and has been directly applied to our 3D X-ray microtomography images.

In the following section, the PCF analysis is compared with the RDF analysis for three particular distributions (random, periodic and clustered). In order to simplify the understanding, both analysis are performed on 2D images but the discussion can be extended to 3D images (as done for 3D X-ray microtomography images in the main paper). The RDF and PCF analyses are first detailed for one distribution of particles and then discussed for three particular distributions (random, periodic and clustered).

RDF/PCF construction process

The RDF is constructed by drawing concentric circles at regular intervals around the centroid of a particle. The number of particle centroids falling in an annulus of radius r , $N(r)$, is then divided by the area of the annulus. This procedure is then repeated for each particle in order to determine $N(r)$ as an average over all the particles, $\overline{N(r)}$. The RDF is then given by $g(r) = \frac{\overline{N(r)}}{\rho}$, with ρ the average density of particles in the image. Fig. 3.16 shows a schematic of the principle of the method and the result of the RDF analysis calculated for a random distribution of non-overlapping particles.

The PCF function is defined, in 2D, as the probability for a given point $[x,y,z]$ to be located inside a particle, given that another particle

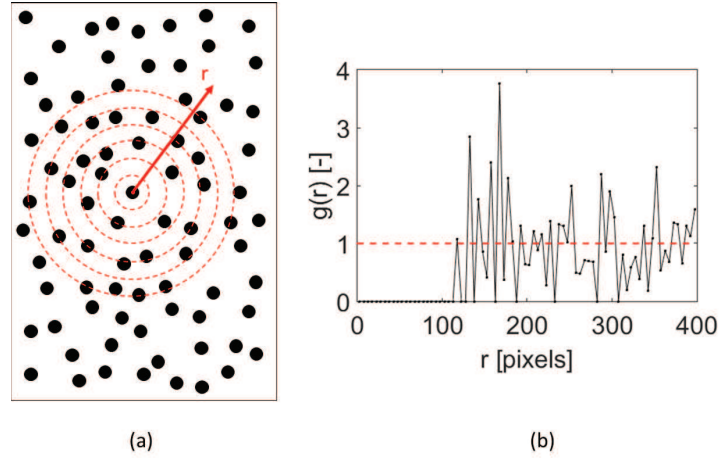


FIGURE 3.16: (a) Principle of the RDF analysis for a random distribution. (b) Radial distribution function $g(r)$.

is located at the origin $[0,0,0]$. Fig. 3.17 to Fig. 3.20 show how the PCF function can be calculated for this same random distribution:

- *Define a measurement frame* (see Fig. 3.17). This size of frame corresponds to the maximum distance r at which the RDF is computed in Fig. 3.16.
- *Counting procedure.* The center of the measurement frame is moved to the center of a first particle (see Fig. 3.17). Each pixel of the measurement frame occupied by neighboring particles is equal to one and all the other pixels occupied by the matrix are equal to zero. The measurement frame is then moved to the location of a second particle, a similar counting is performed (see Fig. 3.18) and the counts are then added up and so on for all particles (see Fig. 3.19). In order to avoid any boundary effect, the particles for which the measurement frame is not completely contained within the image are not taken into account for the analysis.
- At the end of the counting procedure, each pixel of the measurement frame has a value which corresponds to the number of times its specific location corresponds to a neighboring particle (see Fig. 3.20.(a)). Each pixel value is then divided by the total number of

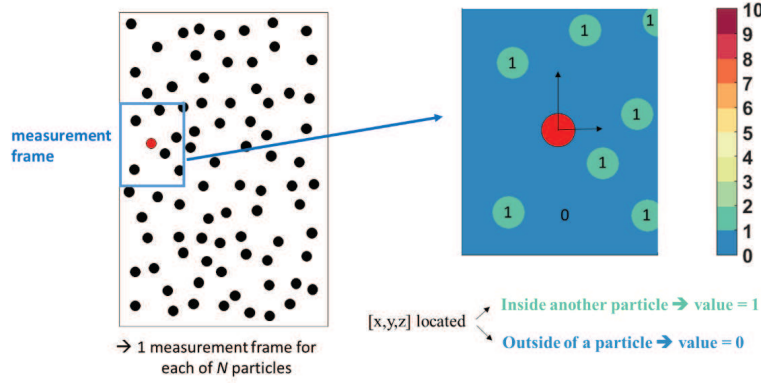


FIGURE 3.17: Principle of the PCF analysis (here in 2D) for a random distribution. The particles and the measurement frame are shown on the left figure.

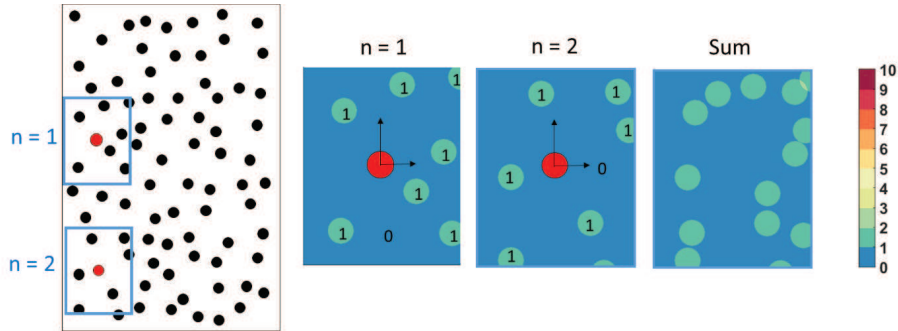


FIGURE 3.18: Principle of the PCF analysis (here in 2D) for a random distribution. Counting procedure after 2 particles.

particles (that entered the counting procedure, see Fig. 3.20.(b)). This value, between 0 and 1, corresponds to the local volume fraction associated to a particle and its near environment.

- This local volume fraction is additionally normalized by the global particle volume fraction over the entire image (i.e. 10.5%, see Fig. 3.20.(c)). This normalized value is called the PCF.

For a perfectly homogeneous spatial distribution, the PCF would be equal to 1 for each point. A PCF value smaller or larger than 1 corresponds thus respectively to a smaller or larger probability to find a

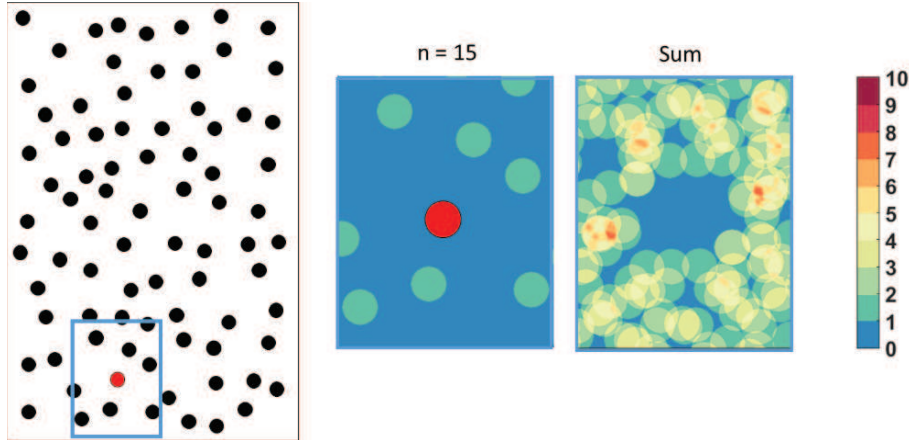


FIGURE 3.19: Principle of the PCF analysis (here in 2D) for a random distribution. Counting procedure after 15 particles.

neighbor in the vicinity of a particle compared to a perfectly homogeneous distribution.

RDF/PCF for three particular distributions, see Fig. 3.21

Random distribution At short distances (small r), the RDF is equal to zero and the first peak corresponds to the average location of the nearest-neighbour (in good agreement with the expected nearest-neighbour distance for a perfectly random distribution of particles, equal to $\frac{0.554}{\sqrt{\rho^2}}$ [3] or 98 pixels in this case). Similarly, the PCF is equal to zero around the center of the measurement frame over approximately the same distance. As the distance r increases, the RDF oscillates corresponding to an alternation between enhanced ($g(r)>1$) and reduced ($g(r)<1$) probability of finding a particle compare to a perfectly random distribution of particles ($g(r)=1$). However, because the distribution is randomly distributed, peak values remain close to 1 (corresponding to the average density of particles). Again, the PCF exhibits the same trends.

Periodic distribution The RDF is equal to 0 except for a limited number of peaks which correspond to the locations of the neighbors in the periodic structure. Similarly, the PCF is equal to 0 except for the locations of the neighbors. Peak values are high (both in the PCF and

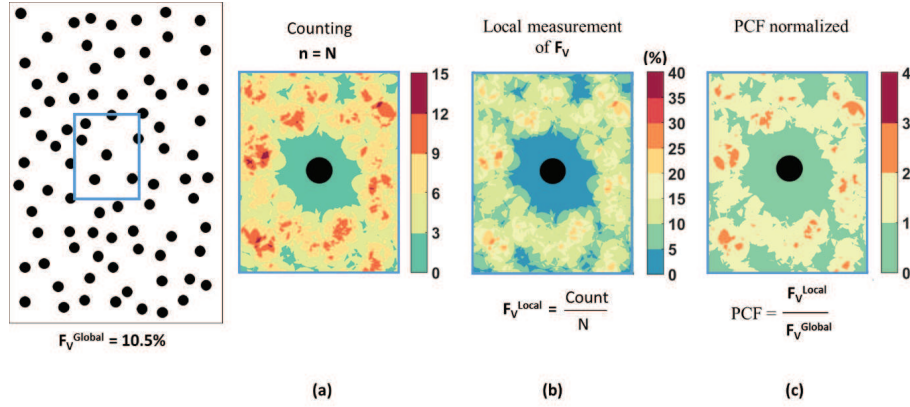


FIGURE 3.20: Principle of the PCF analysis (here in 2D) for a random distribution. Results of (a) the counting procedure, (b) local volume fraction and (c) PCF.

RDF analysis) because the neighbors are located at the same place for each particle within a periodic structure.

Clustered distribution The clustered distribution is composed of the same number of particles (and same density) which have been re-distributed in the form of clusters. These clusters exhibit anisotropic shape on purpose, with random orientation in the first case or with particles preferentially aligned in the vertical direction in the second case. In both cases, there is one significant local maximum on the RDF curve occurring at a smaller distance compared to the random distribution. This is due to the fact that the nearest-neighbor within a cluster is located much closer compared to the random case. However, if this significant peak in the RDF is a good indicator of clustering, it does not give any indication about the structure of the clusters. On the opposite, it is very clear from the PCF analysis that the clustering is either random or oriented along the vertical direction. These conclusions justify the use of the PCF analysis rather than the RDF analysis for our base material and FSP samples.

3.6. Supplementary material to Paper FSP

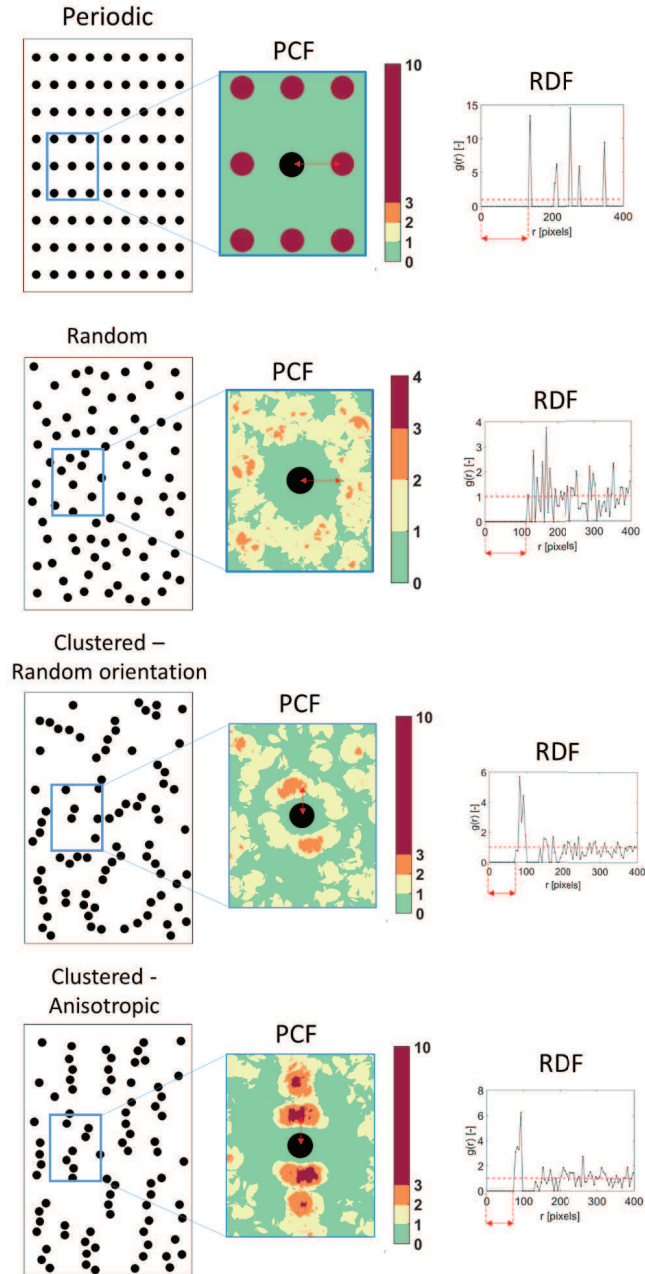


FIGURE 3.21: Comparison between the PCF and the RDF analysis applied on a periodic, a random and a clustered distribution.

3.6.2 Clustering analysis

An analysis proposed by Achon et al [4] was used to characterize the clusters within the particle field. A particle belongs to a cluster if all its near-neighbors identified from the tessellated particle field are within a distance $d_{Cluster}$ or less. This procedure is illustrated here, in 2D, for an example image (see Fig. Fig. 3.23). If the distance $d_{Cluster}$ increases, the number of clusters increases (see Fig. Fig. 3.22 and Fig. 3.23, point A). At some point, the number of clusters identified will start to decrease because the distance $d_{Cluster}$ becomes so large that the clusters coalesce with each other (see Fig. 3.22 and Fig. 3.23, point C). The “selected clustering distance” of the particles distribution is the value of $d_{Cluster}$ corresponding to the maximum number of clusters identified within the particle field (see Fig. 3.22 and Fig. 3.23, point B). This procedure distinguishes between isolated particles, i.e. particles that are distant from all their neighbors by a distance larger than $d_{Cluster}$, from particles belonging to clusters (see Fig. 3.23).

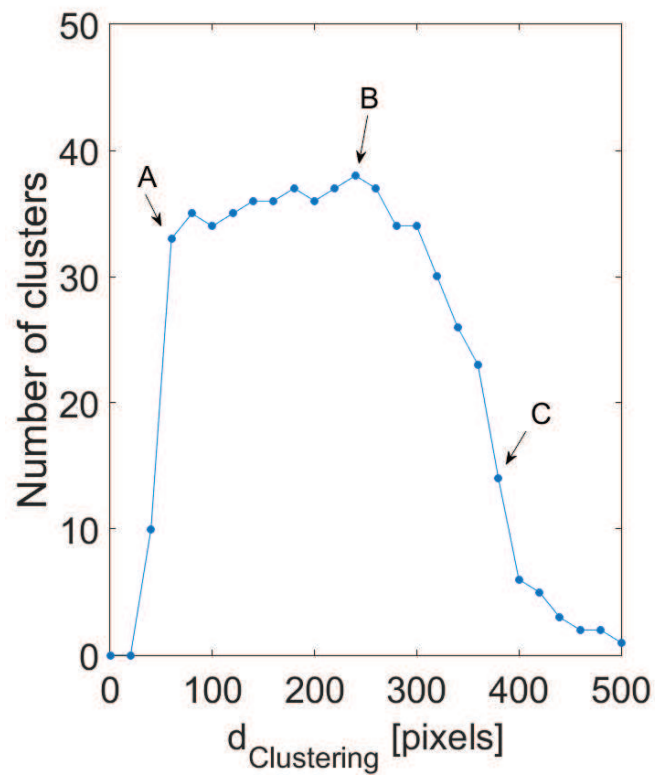


FIGURE 3.22: Results of the clustering analysis performed on the example image shown in Fig. 3.23. Points A,B,C correspond with the images A,B and C in Fig. 3.23.

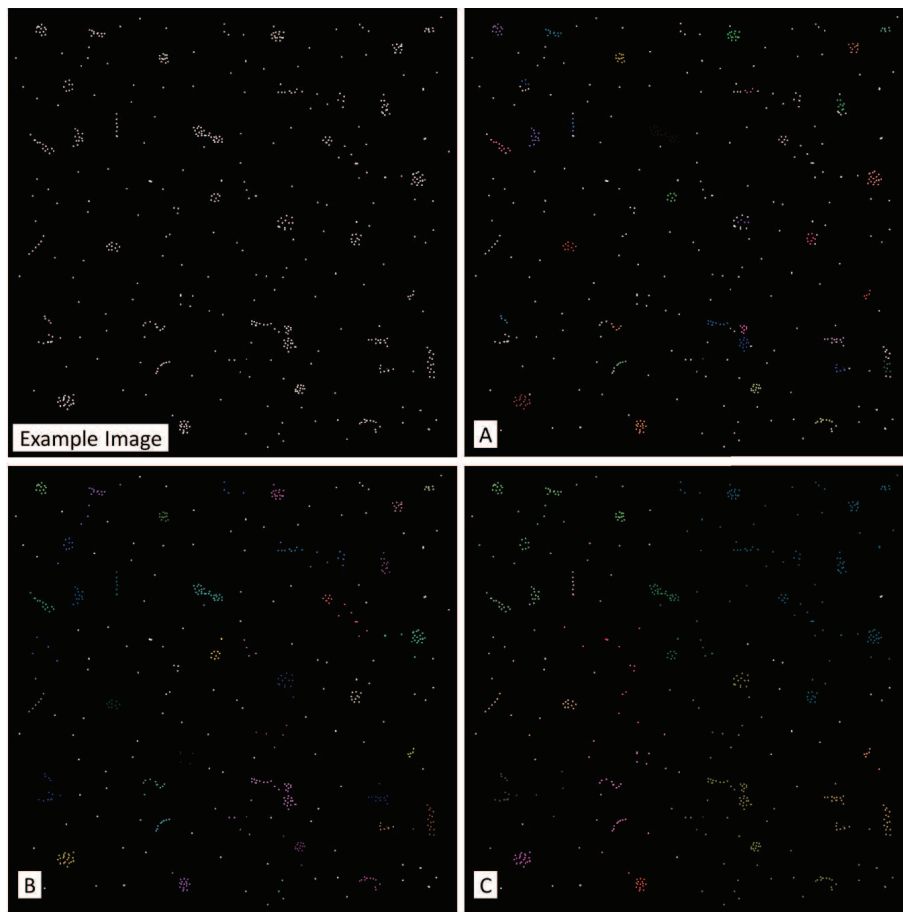


FIGURE 3.23: Example of a “test image” and results corresponding to points A, B and C of Fig. 3.22. White particles correspond to isolated particles, and particles with the same color belong to the same cluster.

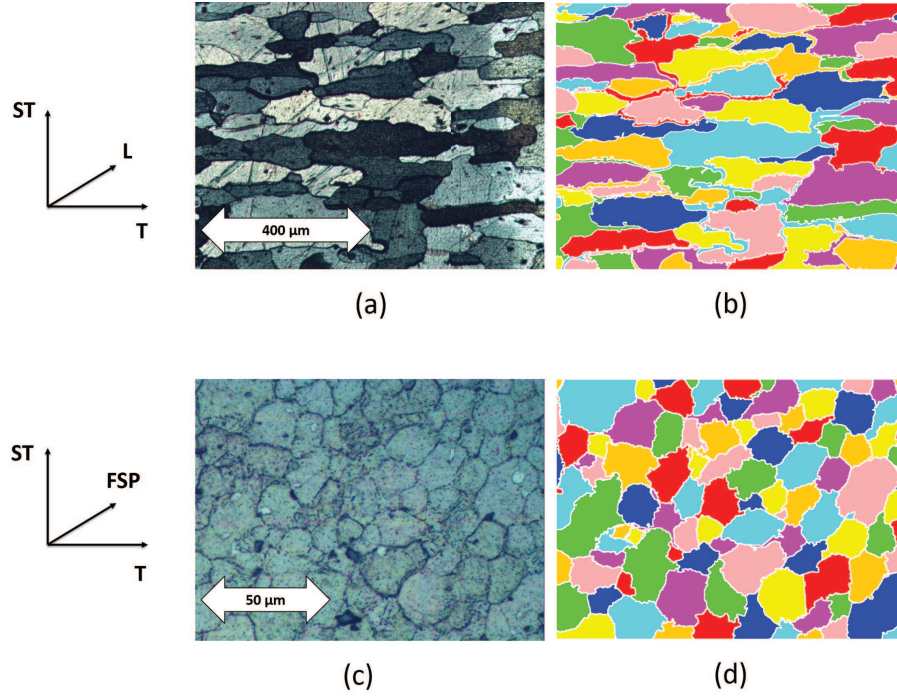


FIGURE 3.24: Micrography of the grain structure in (a) the base material and (c) the FSPed material. Measurements for grain size have been performed as shown in Fig.(b) and (d).

3.6.3 Effect of FSP on grains

The average equivalent diameter D_{eq} in the base material is equal to 137 μm in the plane LT and 87 μm in the plan ST-T (see Fig. 3.24(a)), and is reduced to 10 μm after FSP (see Fig. 3.24(c)).

Fig. 3.25 shows a micrography of the cross-section of the weld in a plane perpendicular to the FSP direction. Prior to optical microscopy, the section was chemically etched in a Keller solution [122].

3.6.4 Abnormal grain growth

In this chapter, the as-FSPed samples were then heat treated at 180°C and some of them at 350°C as well. This has allowed cross-

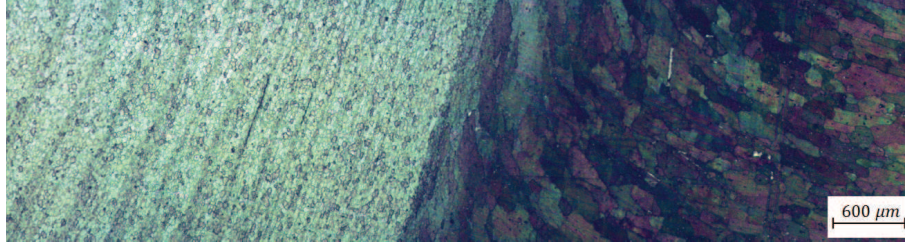


FIGURE 3.25: Micrography of the cross-section of the weld in a plane perpendicular to the processing direction

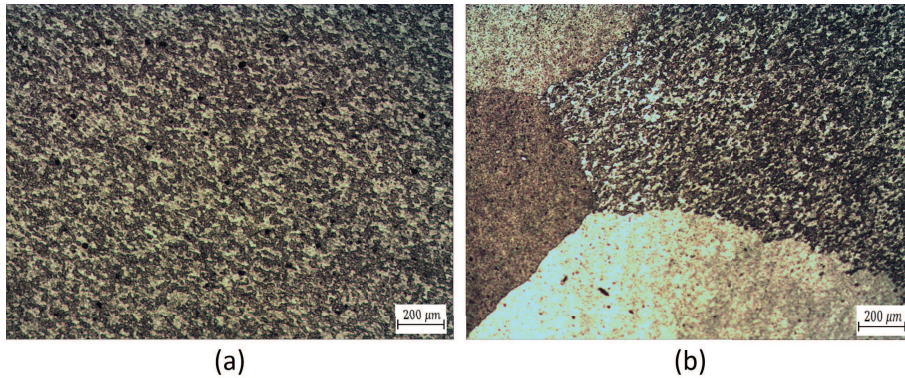


FIGURE 3.26: Micrography of the grain structure in (a) the as-FSPed material and (b) after 20min at 475°C. The initiation of abnormal grain growth is observed in (b).

comparisons of the fracture strain ε_f at a wide range of yield strength σ_0 (see Fig. 3.13). However, the largest values of yield strength ($\sigma_0 > 300\text{MPa}$) could not be obtained with these heat treatments for the FSPed samples.

A post-weld annealing heat treatment (475°C) was thus tried. Unfortunately, this annealing treatment induces abnormal grain growth (AGG) (see Fig. 3.26). The abnormal grain growth aspect is different from conventionally processed aluminum alloys and results from unique as-processed friction stir microstructure that contains strain gradients and microstructural gradients that can lead to microstructural instability [199].

AGG is not a problem *per se* since the contribution of the grain size

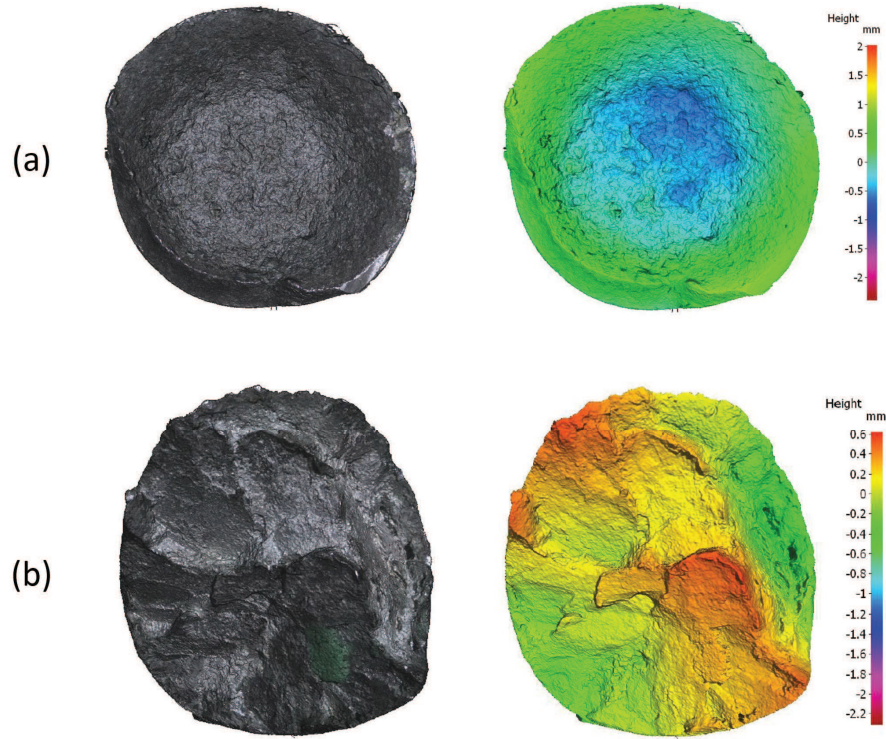


FIGURE 3.27: Fracture surface of a FSP tensile specimen (a) without and (b) with post-weld annealing heat treatment. The fracture mode changes from (a) ductile transgranular to (b) intergranular fracture. The color map indicates the height.

to the strengthening is negligible compared with the effect of precipitates in 6xxx series aluminum alloys[122, 123]. The problem of AGG is that it results in a change of fracture mode from ductile transgranular (see Fig. 3.26(a)) to intergranular (see Fig. 3.26(b)) fracture decreasing significantly the fracture strain of the material.

3.7 Selective formability

3.7.1 Concept of FSP in order to improve formability

Sheet metal forming, such as sheet bending, is a common industrial technique for shaping of metallic components. Sheet-metal formability is generally defined as the ability of the sheet metal to undergo the desired shape change without failure [200]. The term formability is somewhat ambiguous as it may refer to two different conditions, depending on the nature of the manufacturing process. In sheet stretch forming operations, the limiting condition is the strain at which localization is initiated (as typically identified with a Forming Limit Diagram). In this case, the uniform strain identified with a tensile test is an indicator of the formability of a material. The second limiting condition for formability is fracture and is the limiting condition in bending and under crash conditions [123]. In this case, an appropriate indicator of the formability is the fracture strain measured in a tensile test. Furthermore, anisotropy of the sheet is another important factor affecting the bendability. Indeed, the formability of Al alloys is better when the bend axis is perpendicular to the rolling direction (therefore producing the main tensile stresses along RD). This anisotropic bendability is due to the preferred orientation of the particles stringers along the rolling direction [200], similarly with what was observed in the Al 6056.

Bending of thick aluminium plates is usually restricted to low bend angles due to cracks forming in the surface undergoing tensile stresses and propagating through the bulk material. Consequently, thick-section aluminium structures, such as C-channels, are typically fabricated by fusion welding together aluminium plates [201]. Recently, Mahoney *et al.* [201] proposed to locally improve material properties of the plate with FSP in order to enhance the ductility and to enable bending of the plate. Although results are limited, the method has been applied on different aluminium alloys, different material thickness and studies can be classified in three categories depending on the potential applications [202]:

- FSP of sheet to enable superplastic forming [203, 204], i.e. high temperature forming. The mechanisms involved in superplastic forming are quite different compared to the room-temperature

3.7. Selective formability

formability and will not be discussed here. However, it is interesting to comment a bit on the "Selective Superplastic Forming" concept introduced by Wang *et al.* [205]. Indeed, only selected regions require enhanced formability in many components. This means that only the region undergoing large deformation needs the refined microstructure. These local microstructural modifications, i.e. on a "selective" basis, cannot be achieved by conventional processing and leads to very economical manufacturing.

- FSP of thin plate (between 6 and 13mm), followed by room temperature bending. The only study found in this category was performed by Smith and Mishra [202] on Al AA 5083-H116, 8mm-thickness plates. The FSP-bending approach was compared with the more classical gas metal arc welding (GMAW) for the fabrication of an aluminium angle. A multitude of material properties were investigated and it was observed that FSP-bending method gives better results in terms of hardness, corrosion and fatigue properties.
- FSP of thick plate (larger than 13mm), followed by room temperature bending. A few researches in this category have been performed by Mahoney and co-workers : (i) on Al 2519-T87, 25mm thickness [126], (ii) Al 7050-T7451, 50mm thickness [206] and (iii) on Al 6061-T6, 25mm and 150mm thickness [206].

It has been shown in this chapter that FSP is an efficient way to improve the fracture strain of the Al 6056. Furthermore, FSP homogenizes the particle distribution and results in isotropic fracture behaviour. The goal of the following section is thus to use FSP in order to improve the bendability of the alloy and reduce the bend limits associated with the anisotropy of the sheet. This method combining FSP and room temperature bending will be called "selective formability" by analogy with the "Selective Superplastic Forming" concept introduced by Wang *et al.* [205]. The concept is schematically represented in Fig.3.28. The method will be applied on thin plates (2.5mm), i.e. the second category developed above. To the best of the authors knowledge, no analysis is available yet in the literature about the impact of FSP on the formability of such thin plates.

In order to clarify the origin of the improved formability associ-

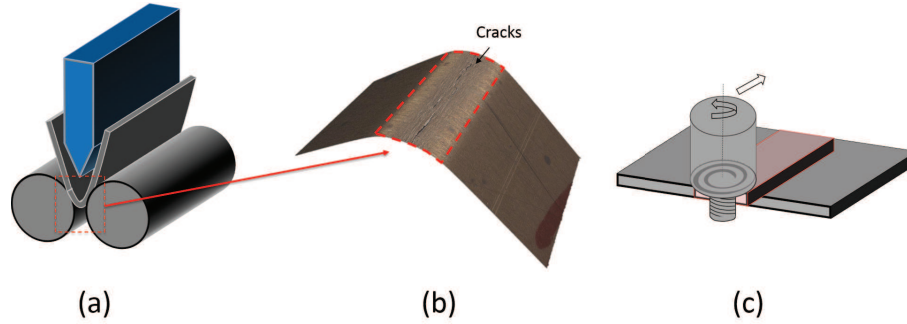


FIGURE 3.28: (a) Bending test set-up, (b) cracking initiation in the bent sheet and (c) concept of "Selective Formability".

ated with FSP, a few more details will be given on the study carried out on the Al 6061 [206], because this alloy is quite similar with the Al 6056 used in the present study. The 25-mm-thick 6061-T6 plates used in [206] were processed using a linear raster pattern. The FSP tool was moved by a small displacement compared to the diameter of the tool and the material is thus processed several times. According to Mahoney *et al.* [206], this increase of ductility can be attributed to both the refined and more homogeneous microstructure and to the annealed condition of the surface material, but the two separate contributions were not quantified. However, it should be pointed out that the processing parameters included a tool rotation rate of 600 RPM and a travel speed of 100 mm/min and that no attempt was made during FSP to minimize the heat input [126]. In comparison, the FSP parameters used to process the Al 6056 in the present study (500 RPM and 200 mm/min) represent a lower heat input. This means that an higher heat of processing will be available in the Al 6061 case to dissolve strengthening precipitates in the matrix. Furthermore, FSP is performed on Al 6061 plates in the T6 state, while in the present study it is performed in the T4 state. Heinz and Skrotzki [207] observed in a 6xxx series Al alloy that strengthening precipitates are much more strongly dissolved during welding in the T6 state compared to the T4 state, and the microhardness in the weld was decreased by $\approx 15\%$ and $\approx 30\%$ when the alloy was processed in the T4 and T6 temper, respectively. In other words, the FSP method used by Mahoney *et al.* [206] on the Al 6061 causes a markedly stronger dissolution of strengthening precipitates and a large hardness decrease ($>40\%$) was observed in the FSP zone. On the opposite, only a limited hardness

decrease ($\approx 15\%$) was observed in the FSP zone for the Al 6056 used in this study. .

3.7.2 Validation of the selective formability concept

Bending was performed on a Zwick Z-250kN servo-hydraulic testing system following the "Verband Der Auto-mobilindustrie" (VDA) 238-100 test specification for metallic materials, see Fig. 3.28(a). Bending tests were performed on 70 mm x 25 mm x 2.5 mm samples, with the bending line perpendicular (BM-L specimen) and parallel (BM-T specimen) to rolling direction, therefore producing the main tensile stresses along rolling and transverse direction, respectively. The bending test is stopped when the force-displacement curve shows a significant drop, i.e. at the initiation of cracking, and the bending radius are measure manually afterwards.

The force-punch displacement curves are shown in Fig. 3.29(a). For the base material, the curves are similar for both direction. This is in good agreement with the limited plastic anisotropy which has been observed previously for the base material. However, the maximum force, i.e. initiation of cracking, occurs at a lower punch displacement for the BM-T specimen. This is in good agreement with the tensile test results performed on the BM, which showed a significant higher ductility (fracture strain) when loading along the rolling direction compared to the transverse direction. For the FSPed material, it is observed that variability of the test seems larger than for the BM. This might be due to the fact that the surface of the FSP samples have been slightly polished in order to removed the roughness due to the processing. It is clear that the yielding of the specimen occurs at a slightly lower force compared to the base material due to partial dissolution of the hardening precipitates. However, the general trend of the FSP curves remain similar with the BM curves. It is clear that despite the larger variability of the results, crack initiation occurs systematically at larger punch displacement for the FSP samples, but at similar maximum bending force. The bending angle which has been reached at crack initiation for a BM-L sample is compared with an FSP sample, Fig. 3.29(b). It is clear that the maximum bending angle which can be reached is significantly larger

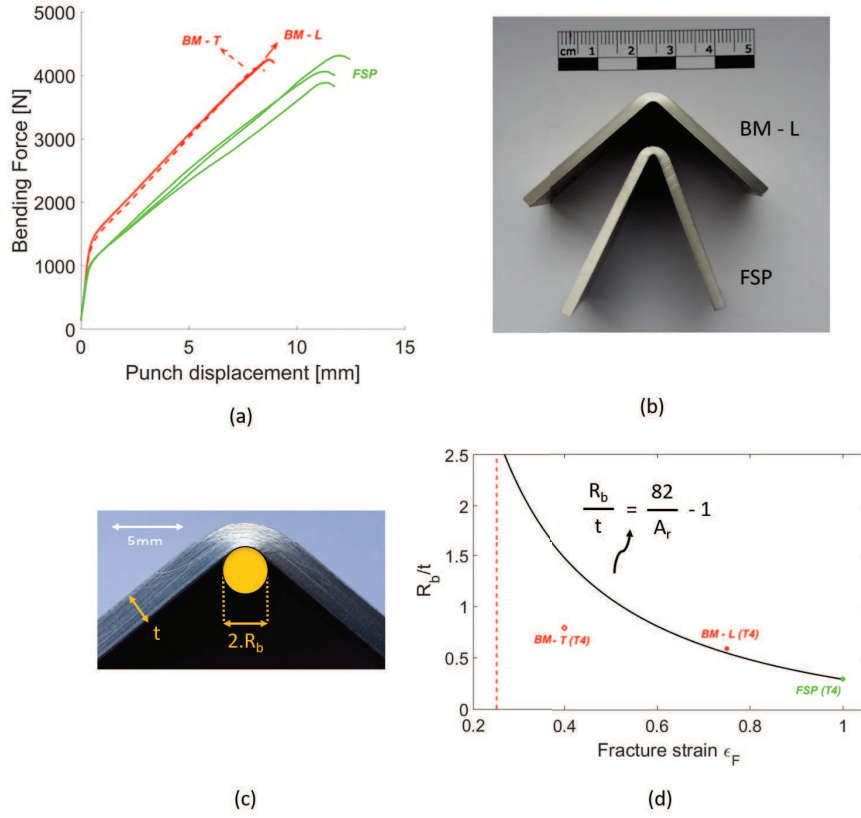


FIGURE 3.29: (a) Force-punch displacement curves from bending tests. (b) Specimens BM-L and FSP at the initiation of cracking. (c) Measure of the bending radius R_b . (d) Comparison between experimental results and eq. (3.5)

for the FSP sample. This is a first hint that FSP improve the formability of the sheet.

The particular radius at which cracks appear on the tensile surface of the bent sheet is called the minimum bend radius R_b . The formability of the sheet increases if R_b decreases, and a minimum bend radius of zero means that the sheet can be bent on itself. R_b is usually given in terms of the sheet thickness t . Indeed, this ratio is related to the the fracture strain of a material during uniaxial tensile test. The following

3.7. Selective formability

empirical relationship has been found by Datsko and Yang [208] :

$$\frac{R_b}{t} = \frac{60}{A_r} - 1 \quad (3.5)$$

where A_r is the percentage reduction in area of a tensile test specimen ($\epsilon_f = \ln(100/(100 - A_r))$). R_b has been measured as shown in Fig. 3.29(c). Fig. 3.29(d) shows that increase of formability (i.e. R_b/t decreases) for the FSP-ed material is quite well predicted with eq. (3.5) when compared with the BM-L specimens. However, the observed formability of the BM-T specimens is larger compared to eq. (3.5).

Chapter 4

Quantitative assessment of the impact of second phase particle arrangement on damage and fracture anisotropy

This chapter is based on the submitted article *Quantitative assessment of the impact of second phase particle arrangement on damage and fracture anisotropy*, F. Hannard, A. Simar, E. Maire, T. Pardoen (2017).

4.1 Introduction

Ductile fracture results from an overlapping sequence of nucleation, growth and coalescence of small internal cavities leading to macroscopic failure. The current state of micromechanical understanding of the damage stages is relatively advanced and has allowed the development of predictive models for sufficiently homogeneous materials [24]. Indeed, ductile damage has traditionally been treated as a global process, see for instance the enhanced version of the Gurson model [3, 4, 5, 135], the material being often treated with "average" microstructural properties assuming a strict progression from nucleation to coalescence. However, there are considerable experimental evidences which indicate that the ductile damage process is strongly affected by microstructure heterogeneities.

The different types of microstructure heterogeneities which can influence the damage process have been thoroughly reviewed in Ref. [27]. Heterogeneities can impact the damage process in two ways. First, coalescence might occur in some clustered regions or regions in which void nucleation was fast and void close to one another while other regions of the material do not exhibit any damage yet. Second, the anisotropy of the microstructure impacts the fracture behaviour and can induce significant anisotropy in the mechanical properties, which is at the core of the present work.

Three types of sources of anisotropy can impact the fracture behaviour :

1. Plastic anisotropy is related to the crystallographic texture as caused for instance by the preferred orientation of the grains along the rolling direction. The plastic flow anisotropy has indeed a significant influence on the ductile failure as void growth is driven by plastic deformation of the surrounding material [209, 25].
2. A morphological anisotropy can originate both from the shape of the voids or from the shape of the particles at which voids nucleate with a preferential orientation of their main axis. For instance, void nucleation by particle cracking is dependent on the loading

direction because particles preferentially aligned along the processing direction [210, 13] undergo larger load transfer [34]. Benzerga and Keralavarma used cell model analyses to show that the void aspect ratio significantly affects the overall ductility [211].

3. A topological anisotropy can result from the spatial distribution of particles. It particularly affects the void coalescence step. During the forming process, brittle intermetallic particles are broken and a stringer-type particle clustering develop along the rolling direction. This source of anisotropy is further addressed in this study and a deeper coverage of the literature is provided in the next paragraphs.

The topological anisotropy of fracture properties has been increasingly characterized in recent years. One of the first analysis of the void spacing effect on the coalescence stage is due to Koplik[212] and then further commented in Ref. [5]. Lassance *et al.* [76] have used finite elements (FE) unit cell calculations to study the effect of void distribution on ductility. It was concluded that for a metal with a volume fraction of particles of 1%, the ductility might change by more than a factor of 2 if the void spacing varies by a factor 2 when comparing two orthogonal directions. Thomson *et al.* have shown that the orientation of a particle cluster with respect to the direction of loading significantly influences the failure strain [130]. This effect has been studied experimentally in a model (Al-Si) material specifically fabricated to accentuate second-phase particle clustering [213]. The formability was indeed found to be reduced when the loading direction was perpendicular to the major axis of the particle stringers. The clustering of particles into bands has also been linked to the anisotropic ductility in Al-2024 rolled plates as it creates an easy percolating crack path perpendicular to the rolling direction [214]. The effect of the loading direction on the ductility of two commercial aluminium alloys (AA5182 and AA5754) was investigated in Ref.[21]. The lack of orientation effect on the ductility of the AA5754 compared to AA5182 was attributed to a lower propensity for large and oriented stringer-like clusters. Bouchard *et al* [215] observed a strong anisotropic fracture behaviour in a classical manganese steel grade, while the hardening behaviour did not depend much on the direction of loading. In situ X-ray tomography tensile tests revealed that the strong anisotropy was mainly related to the direct influence of

4.1. Introduction

the particular inclusion spatial distribution with very elongated and aligned MnS inclusions. An anisotropic void distribution also has a significant effect on the resistance to crack initiation [216].

In what is probably the most advanced study to date of the anisotropy effects on ductile fracture by Benzerga *et al.* [217], all three sources of anisotropic damage, i.e. plastic, morphological and topological anisotropy, were characterized and it was concluded that the effect of void spacing on the void coalescence is of primary importance to explain the orientation dependence of ductility. However, more experimental work is required to develop simple and quantitative microscopic indicators of particle clustering and of its degree of anisotropy. The limited attention that has been given to this major effect is probably related to the difficulty to quantitatively distinguish between the various sources of damage.

In our previous study [12], the ductility of three industrial Al alloys has been investigated under a wide range of heat treatment conditions. These three alloys involve relatively similar composition and volume fraction of iron-rich intermetallic particles (around 1%). However, the size of the particles is very different with an average equivalent diameter equal to $1.3\mu m$, $2.1\mu m$, and $3.1\mu m$ for the Al 6005A, Al 6061 and Al 6056, respectively. Major differences among the fracture strains in the different alloys were observed which were shown to be primarily dictated by the effect of particle size distribution with the alloy exhibiting the smallest particles nucleating later and thus being the most ductile. In the present investigation, the anisotropy of ductility between the main forming direction and the transverse direction has been characterized for these three alloys. The purpose of the paper is to propose a quantitative approach to relate the propensity to fracture anisotropy with a simple microscopic parameter characterizing the degree of anisotropy of the spatial distribution of second phase particles.

4.2 Experimental methods

4.2.1 Materials

The Al alloys were provided in the form of extruded plates (6005A alloy) or of cold rolled plates (6061 and 6056 alloys). The thickness of the plates is equal to 6 mm for the 6005A and 6056 alloys and 4.7 mm for the 6061 alloy. These three alloys all present Mg and Si as main alloying element. Their composition are given in Ref. [12]. Heat treatments at 350°C for various durations ranging from 10 s to 20 days have been performed on each material initially in the T6 state. Cross-comparisons of the three alloys can thus be made at similar initial yield stress.

4.2.2 Macroscopic tensile tests

Uniaxial tensile tests are performed under displacement control at constant velocity (1 mm/min). The tensile specimens are cylindrical, 4 mm in diameter, and the initial gauge length is equal to 30 mm. Tensile specimens were extracted perpendicular and parallel to the processing direction. The initial yield stress and the true strain at necking are noted σ_0 and ϵ_u , respectively. The true fracture strain has been measured post-mortem on the broken specimens and is quantified by $\epsilon_f = \ln\left(\frac{A_0}{A_f}\right)$, where A_0 and A_f are the initial and final cross-sectional area, respectively.

4.2.3 Microstructure characterization

The characterization of the intermetallic particle fields has been carried out based on scanning electron microscopy (SEM) and synchrotron-based X-ray microtomography. After standard polishing, the intermetallic particles are characterized by SEM operated at 15 kV

4.3. Results

under electron backscattered mode. SEM images, taken at a magnification of 600x in the processing-transverse plane, are combined in order to determine the spatial distribution of particles over a statistically representative area ($\approx 1.25\text{mm}^2$).

X-ray microtomography experiments are performed at the TOMCAT beamline of the Swiss Light Source [153]. The observed region is a cube of $260\text{ }\mu\text{m}$ side length and isotropic voxel size of $325 \times 325 \times 325\text{ nm}^3$, see [12] for more details.

4.2.4 In situ tensile testing coupled to X-ray microtomography

In situ tensile tests coupled to X-ray microtomography (voxel size of $0.6 \times 0.6 \times 0.6\text{ }\mu\text{m}^3$) have been performed for the Al 6056 in T4 temper ($\sigma_0 \approx 250\text{MPa}$) in order to further investigate the effect of the loading direction on the evolution of the damage process and to understand the role of microstructure on fracture anisotropy. These tests are also performed at the TOMCAT beamline of the Swiss Light Source [153]. A dedicated in-situ tensile testing machine described in Ref. [154] has been mounted on the rotation stage of the 3D tomography setup [155]. The mini smooth tensile specimens, with diameter of 1 mm, are deformed up to fracture while the tensile force F is recorded. The data acquisition is made at regular discrete deformation steps.

4.3 Results

4.3.1 Mechanical properties

Fig. 4.1 summarizes the results extracted from the tensile tests for the three alloys, loaded along the rolling and transverse directions, as a function of the initial yield stress σ_0 (corresponding to the various heat treatments). For the same value of σ_0 , the tensile strength σ_{max}

is very similar in every case (Fig. 4.1(a)). Strain rate sensitivity is weak in the 6xxx serie aluminium alloys [218] and, in agreement with the Considère condition, the mean strain hardening exponent n is equal to the strain at necking ϵ_u . The necking strains follow the same “banana” shape trend for the three alloys when plotted as a function of yield stress, independently of the loading direction (Fig. 4.1(b)). It can thus be concluded from these observations that the plastic yielding behaviour is quite isotropic for the three alloys, i.e. the hardening behaviour does not depend much on the loading direction.

However, the fracture strains are very different when comparing the three alloys (Fig. 4.1(c)) and this has already been shown, see Ref.[12], to be primarily dictated by the effect of particle size distribution with the alloy exhibiting the smallest particles being the most ductile. Fig. 4.1(c) also shows that while the fracture strain is very similar for both loading directions in the 6061 and 6005A Al alloys, the anisotropy is significant for the Al 6056. Indeed, in this alloy, the fracture strain in the rolling direction is larger compared to the transverse direction at identical yield strength, especially for heat treatments leading to high strength.

4.3.2 Microstructure characteristics

Fig. 4.2 shows part of the particle fields analysed using SEM images and Fig. 4.3 (left) shows a sub-volume extracted from the full tomographic volume for the three alloys. As expected, the particles are preferentially aligned along the processing direction, leading to a stringer-type clustering. It seems that the clustering is more pronounced in the case of the Al 6056 but the quantification of the degree of clustering by visualization can be misleading as the size of the particles differ among the alloys. A rigorous method to quantify the degree of anisotropy of particle clustering is proposed in the following.

4.3. Results

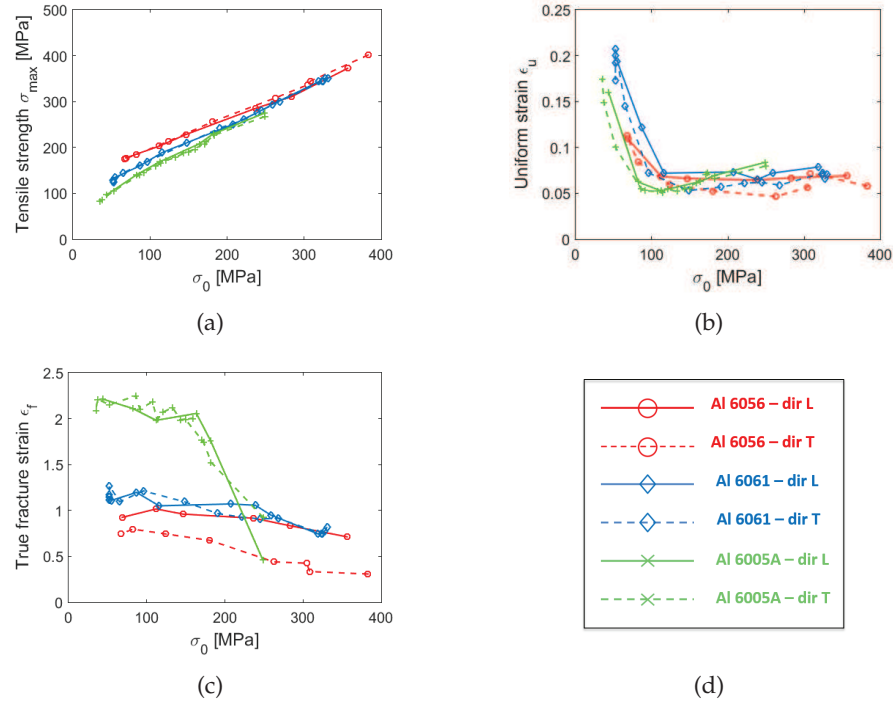


FIGURE 4.1: Variation of the different parameters extracted from the uniaxial tensile tests on the three alloys as a function of the yield stress corresponding to the different heat treatments: (a) tensile strength of three Al alloys; (b) true fracture strain; (c) true strain at the onset of necking.

4.3.2.1 Quantification of the degree of microstructural anisotropy

The main question addressed here is whether the degree of anisotropy of particles clustering along the processing direction is large enough to affect the macroscopic ductility. In other words, the goal is to find a microstructural parameter which determines if the configuration of stringers can potentially lead to a percolating damage path perpendicular to the processing direction. The problem is physically related to the percolation of damage/microcracks over a length scale large enough to trigger the catastrophic failure of the specimen, hence it is necessary to quantify the long range characteristics of the particles spatial distribution. This means that more classical indicators

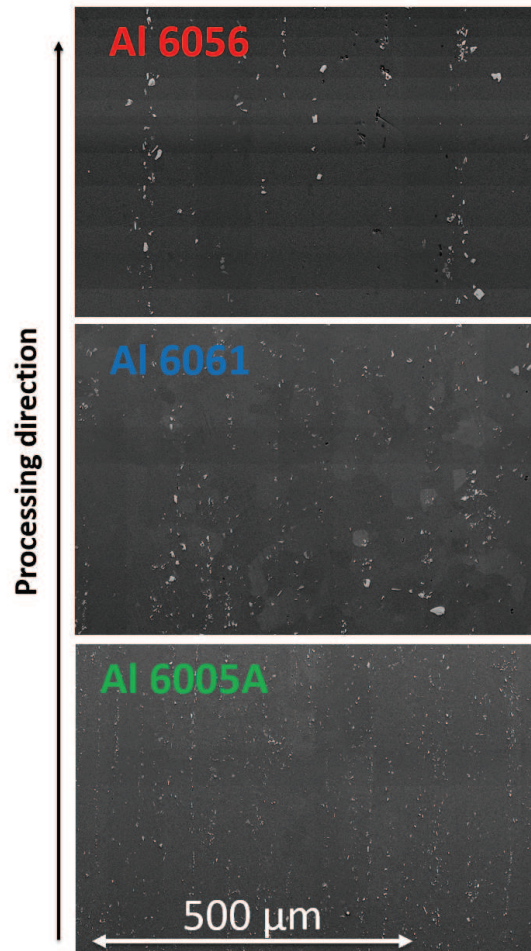


FIGURE 4.2: SEM micrographs taken in the processing-transverse plane showing the distribution of intermetallic particles (in white) in the Al 6056, Al 6061 and Al 6005A.

based on the nearest neighbor analysis or Voronoi tessellation are not rich enough, as they focus on the short range characteristics of the particles neighbourhood. As a consequence, an extension in 2D and 3D of the radial distribution function (RDF) analysis is applied. In 1D, the RDF corresponds to the probability for a neighboring particle to be located at a distance r , in any direction [196]. The extended pair correlation function (PCF), in 2D and 3D, takes into account both

4.3. Results

the distance and angular dependence of the particles (preferentially aligned in one direction). **The PCF function is defined, in 3D, as the probability for a given point at position $\vec{r}$ to be located inside a particle, given that another particle is located at the origin of the reference system.** A PCF value smaller or larger than 1 corresponds, respectively, to a smaller or larger probability to find a neighbor in the vicinity of a particle compared to a perfectly homogeneous distribution. The two-point correlation function, which is closely related to the PCF described here, has been largely discussed in [219, 10, 220] and already been used to characterize anisotropic microstructures [221]. The PCF has already been successfully used to quantify for instance the effect of 3D particles distributions on particle stimulated nucleation during recrystallization [197] or to quantify microstructure homogeneization by friction stir processing [13].

The PCF analysis is directly applied in 2D to the SEM images and in 3D to the microtomography images. In the 3D case, 2D cuts of the PCF in the processing-transverse plane are plotted in order to allow direct comparison with the analysis performed in 2D on SEM images. The PCF of the Al 6056, Al 6061 and Al 6005A are shown in Fig. 4.3. Globally, Fig. 4.3 shows that the SEM and microtomography based PCF are similar for all three alloys. Clearly, the PCF is anisotropic in each case with the highest PCF value parallel to the processing direction. As already explained, this is due to the stringer-type clustering along the processing direction. The anisotropy of the PCF of the Al 6056 (Fig. 4.3(a)) is very pronounced, with high PCF values (≈ 4) along the rolling direction, extending over the entire frame (more than $50\ \mu\text{m}$). On the opposite, the anisotropy observed in the PCF corresponding to the alloys Al 6061 and Al 6005A is less marked. Indeed, the area with high PCF values is more confined in both cases compared to the Al 6056. If a PCF value larger than 3 is chosen as an indicator of particle clustering, the analysis indicates that particles are clustered into long bands in the Al 6056. On the opposite, particles are arranged into stringer-type clusters extending over a length of $\approx 25\ \mu\text{m}$ for the Al 6061 and $\approx 10\ \mu\text{m}$ for the Al 6005A.

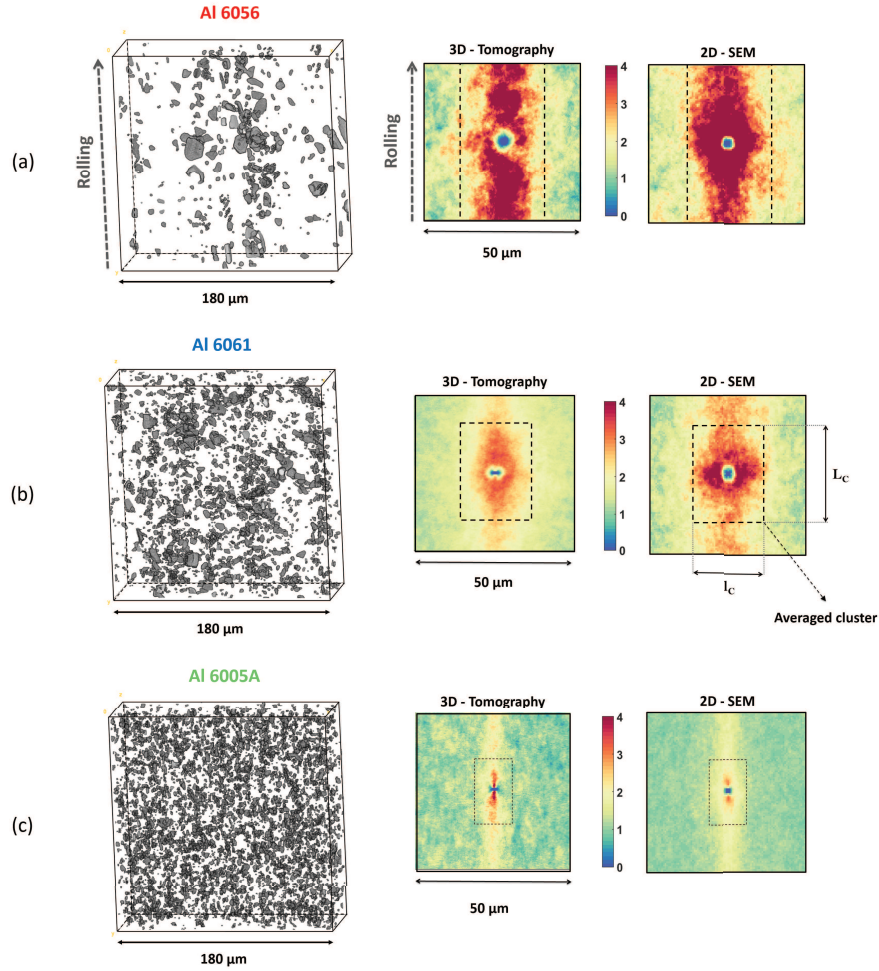


FIGURE 4.3: (*Left*) 3D microtomography perspective of particles distribution in (a) 6056, (b) 6061 and (c) 6005A aluminium alloys and (*Right*) corresponding pair correlation functions (PCFs) for the RD-TD plane through the center of the sub-volume. A PCF value smaller or larger than one corresponds respectively to a smaller or larger probability to find a neighbor in the vicinity of a particle compared to a perfectly homogenous distribution.

4.3.2.2 Quantification of particle clustering

In the previous section, it was shown with the PCF analysis that the anisotropic ductility of the Al 6056 is caused by a very pronounced

4.3. Results

anisotropy of the particle distribution, while it is less marked in the alloys Al 6061 and Al 6005A. However, this conclusion is based on cross-comparison only and an inherent problem of the PCF analysis is that it does not provide an absolute criterion that would indicate if the degree of anisotropy of the clustering is large enough to affect the macroscopic ductility. This is due to the fact that the PCF analysis does not distinguish between the contribution from particles belonging to the same cluster and particles belonging to different clusters, i.e. the PCF does not provide information about the percolation of clusters [219].

An analysis proposed by Achon et al. [131] was used to further characterize the clusters within the 2D particle field observed by SEM. This procedure distinguishes isolated particles, i.e. particles that are distant from all their neighbours by a distance (from centroid to centroid) larger than a distance defined thus as $d_{Cluster}$, from particles belonging to clusters. The distance $d_{Cluster}$ is an objective (i.e. user independent) characteristic of the particles spatial distribution. When the distance $d_{Cluster}$ increases, the number of clusters increases. At some point, the number of identified clusters starts to decrease because the distance $d_{Cluster}$ becomes so large that the clusters coalesce with one another. The “clustering distance” of the distribution selected here is the value of $d_{Cluster}$ corresponding to the maximum number of clusters identified within the particle field, see [12] for more details on Achon’s method. A visualisation of a few clusters is shown in Fig. 4.4 for the three alloys. White particles correspond to isolated particles and particles with the same color belong to the same cluster.

The main indicators resulting from the clustering analysis are shown in Table 4.1. The characteristic clustering distance $d_{Cluster}$ decreases with the size of the particles (equal to $1.3\mu m$, $2.1\mu m$, and $3.1\mu m$ for the Al 6005A, Al 6061 and Al 6056, respectively). Indeed, since the volume fraction of particles is approximately the same in the three alloys, the number of particles per unit volume increases (the average distance between particles decreases) when the particles size decreases. However, the average number of particles forming a cluster is always about 9 and the percentage of particles which are located inside clusters is always approximately 60%. The size and the shape of

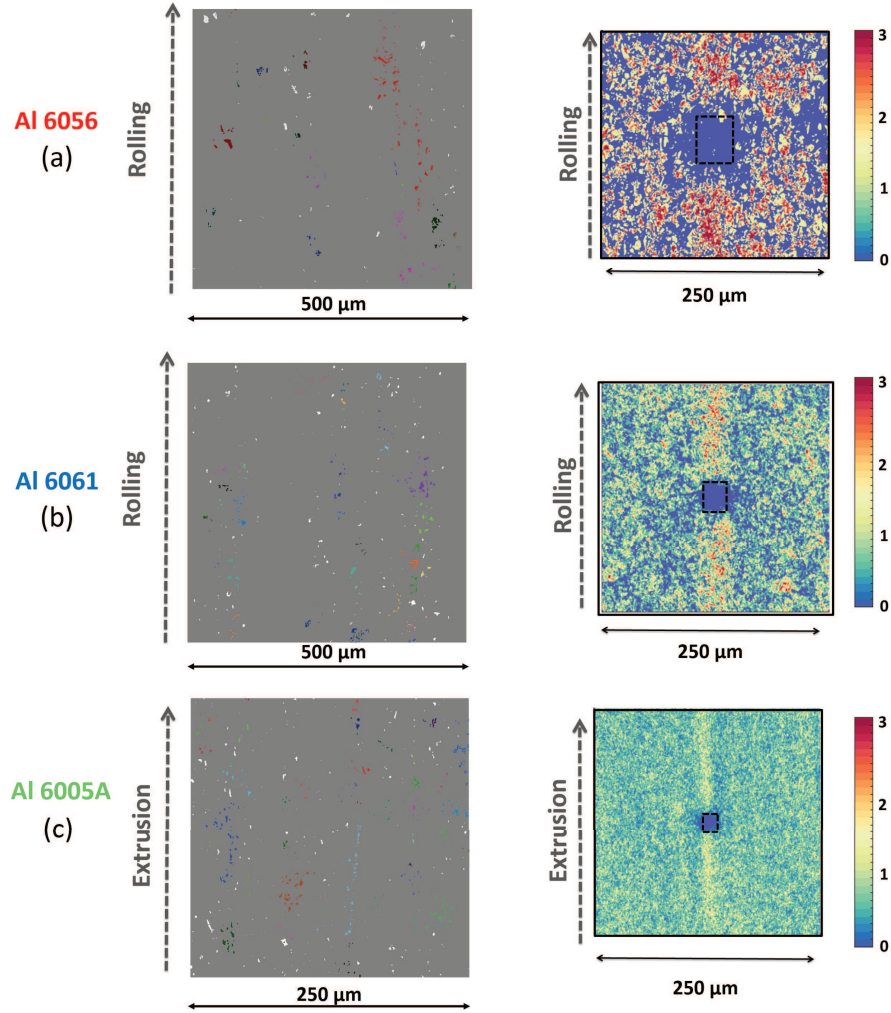


FIGURE 4.4: (Left) Visualisation of a few clusters from particle distribution shown in the SEM images of Fig. 4.2. (Right) PCF analysis performed on clusters from SEM images for the (a) Al 6056 and (b) Al 6005A.

the clusters have been characterized with the minimum bounding box enclosing all the particles belonging to the same cluster. The average side length of the box along the rolling/extrusion direction is given by L_C and the side length along the transverse direction is given by l_C . The averaged bounding box of the clusters in Table 4.1 has been reported for each alloy in Fig. 4.3. The clustering analysis is coherent

4.3. Results

with the PCF analysis in terms of cluster size and shape. Indeed, in each case, the average cluster approximately corresponds to the zone which contains all PCF values larger than ≈ 3 .

Parameter	Al 6056	Al 6061	Al 6005A
Clustering distance d_{Cluster}	22.5 μm	13.5 μm	7.5 μm
Average number of particles forming a cluster	9.3 particles	9.6 particles	8.5 particles
% of particles within clusters (volume-weighted)	62.3 %	63.8 %	63.4 %
Cluster size (L_c/l_c)	57/27 μm	31/23 μm	21/12 μm

TABLE 4.1: Indicators resulting from the clustering analysis.

4.3.2.3 Quantification of the clustering anisotropy

After the identification of the clusters, the cluster distribution itself can be characterized by the PCF analysis as well. Thus, each cluster is treated as a group and the PCF analysis is performed in the same way as previously but this time on clusters. The large particle fields characterized by SEM contain several hundreds of clusters allowing a statistical analysis of their arrangement. The results are shown in Fig. 4.4. The central box represents the average cluster size characterized with the minimum bounding box enclosing all the particles belonging to the same cluster, as already explained in section. 4.3.2.2. This average cluster size is larger for Al 6056 compared to Al 6061 and Al 6005A. Large scale clustering approximately corresponds to the zone which contains all PCF values higher than about 3. In the Al 6056 (Fig. 4.4(a)), large-scale clustering appears along the rolling direction. In other words, there is a large-scale clustering (i.e. clusters of clusters) along the rolling direction. On the opposite, in the Al 6061 and Al 6005A (Fig. 4.4(b,c)), the PCF analysis is much more homogeneous. The anisotropy of the PCF remains very limited and the PCF is close to 1 everywhere, with no large-scale clustering.

The analysis indicates that the anisotropy of the ductility in the Al 6056 is linked to the length scale over which the particle distribution exhibits anisotropy, and this will be further clarified in the following section.

4.3.3 Damage evolution: experimental results

Fig. 4.5 shows a 2D cut through the middle of two tensile specimens, just before fracture, loaded either along the transverse (Fig. 4.5(a)) or the rolling direction (Fig. 4.5(b)). The evolution of the minimum cross-sectional area in the necking region (A_{loc}) provides an estimate of the local true tensile strain $\varepsilon_{loc} = \ln\left(\frac{A_0}{A_{loc}}\right)$ and of the true macroscopic axial stress $\sigma_{zz} = F / A_{loc}$. The tensile stress-strain curves extracted from the in situ data is given in Fig. 4.5(c) for both loading directions up to large deformations. Fig. 4.5(c) shows that plastic anisotropy is small as already concluded from the macroscopic tensile tests. The influence of the spatial distribution of intermetallic particles can be quantitatively observed in the insets of Fig. 4.5(a) and Fig. 4.5(b). When loading in the transverse direction, coalescence events are already detected at the early stages of necking. Looking at the inset of Fig. 4.5(a), coalescence events are generalized inside clusters (i.e. intracluster coalescence) at deformation levels similar to the uniform elongation (i.e. well before fully developed necking). On the contrary, when the loading is in the rolling direction (see the inset of Fig. 4.5(b)), a stable void growth process is pursued even after significant amount of necking, with only a very small number of coalescence events.

There exist thus two coalescence stages of ductile damage within heterogeneous materials that include intra- and inter-cluster coalescence [130, 222]. The evolution of damage is thus strongly influenced by the degree of anisotropy of the particle clusters. The sequence is analyzed in more details in the following paragraphs.

a) Intra-cluster coalescence. Early intra-cluster coalescence stage is observed in the in situ testing of the Al 6056 along the rolling direction, see Fig. 4.6(a) and (b) in which a few clusters involving some coalescence events have been encircled. Intra-cluster coalescence consists in the linking of a few voids over a distance of approximately $50 \mu m$, in good agreement with the cluster size (see Fig. 4.3(a) and Table 4.1).

b) Inter-cluster coalescence. The final fracture is triggered by a second stage of inter-cluster coalescence which leads to the collapse of

4.3. Results

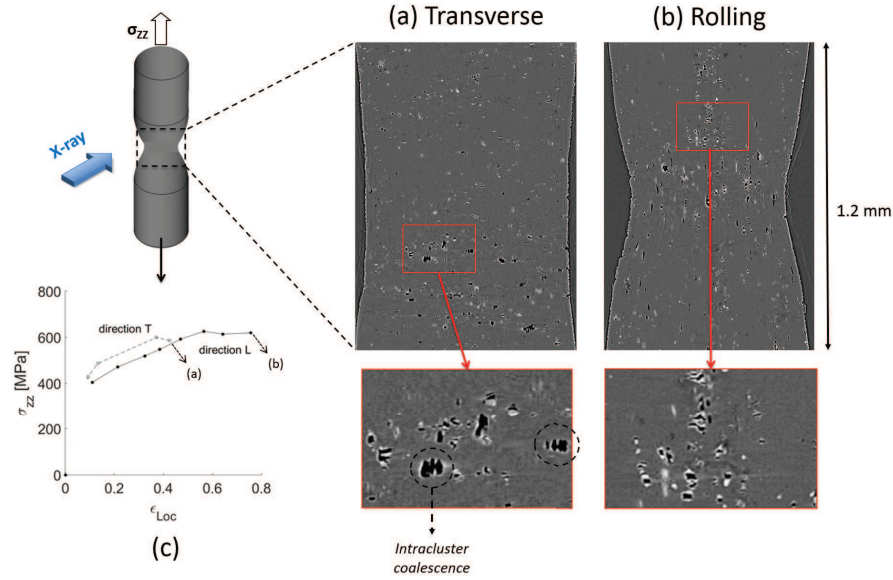


FIGURE 4.5: X-ray microtomography reconstructed images just before fracture, of the tensile test performed on Al 6056-T4 loaded in (a) the transverse direction and (b) the rolling direction. Voids appear in black in the grey matrix and IM particles appear in white. The tensile curves in (c) correspond to the in situ tensile tests shown in (a) and (b).

the matrix ligament between microcracks formed by the intra-cluster coalescence stage. Again, this is in good agreement with what is observed in Fig. 4.6(c) which corresponds to the material state just before fracture. This shear localization between clusters should not be confused with a macroscopic shear localization extending over many voids at a much larger scale [223]. Indeed, the cluster localization is occurring at a mesoscale, over a distance of approximately $150\mu m$.

c) Coalescence mechanisms at the different length scales.

Fig. 4.6(d) shows a zoom of two microcracks formed by intra-cluster coalescence, which confirmed the internal necking process. The formation of a second population of cavities is also observed in the ligament between these two microcracks. Fig. 4.6(e) shows a zoom of the two same microcracks at a slightly larger deformation. The ligament between these microcracks is collapsing by void sheeting initiated on this

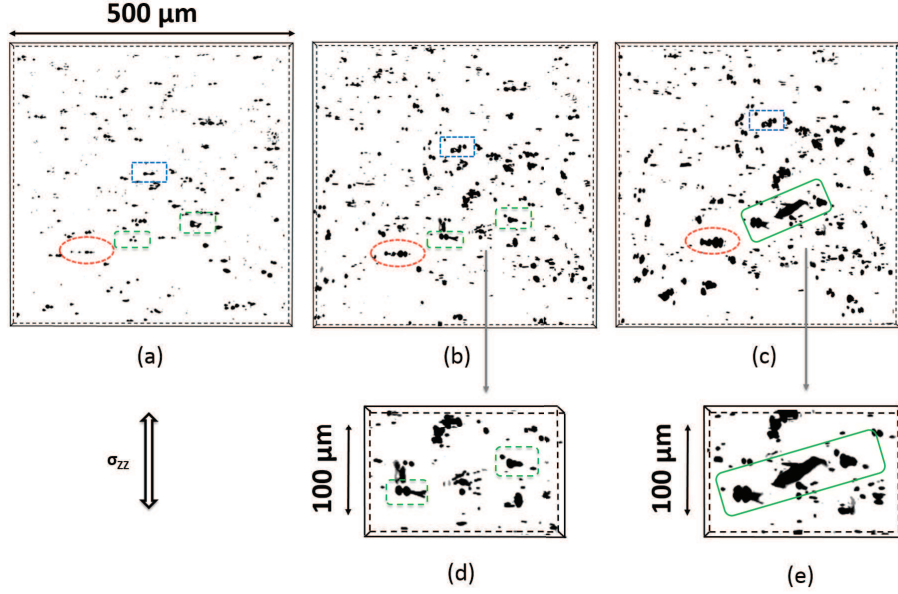


FIGURE 4.6: Void distribution observed during in-situ microtomography in the Al 6056 loaded in the transverse direction at a local strain of (a) $\epsilon_{Loc} = 0.1$, (b) $\epsilon_{Loc} = 0.35$ and (c) $\epsilon_{Loc} = 0.43$. (d) Zoom of figure (b), showing the formation of a second population of voids between two clusters of voids. (e) Zoom of figure (c), showing the intercluster coalescence with the formation of micro shear band.

second population of cavities. These experimental observations are in good agreement with the modeling results from literature, as detailed in the following section.

4.4 Discussion on the origins of the failure anisotropy

First, early coalescence events mainly occur inside particle clusters. Thomson *et al* [130] have shown that coalescence of voids within a cluster, i.e. intra-cluster or void-to-void coalescence, is a stable event which does not directly lead to material failure but is followed by a second void growth process based on these early coalesced

4.4. Discussion on the origins of the failure anisotropy

microcracks. Fig. 4.7 schematically rationalizes the void interactions occurring inside a cluster of particles. The cracking of an intermetallic particle changes the strain field in the surrounding matrix and creates a region of elevated strain in the close neighbourhood, see Fig. 4.7. The process of void coalescence is favoured when the loading is in the transverse direction due to enhanced interaction of the strain fields surrounding closely spaced voids (see Fig. 4.7(a)) facilitating the localization of plasticity in the intervoid ligament. On the contrary, when the loading is in the rolling direction, there is less interaction of the local strain fields associated to the voids due to their spatial distribution (see Fig. 4.7(b)).

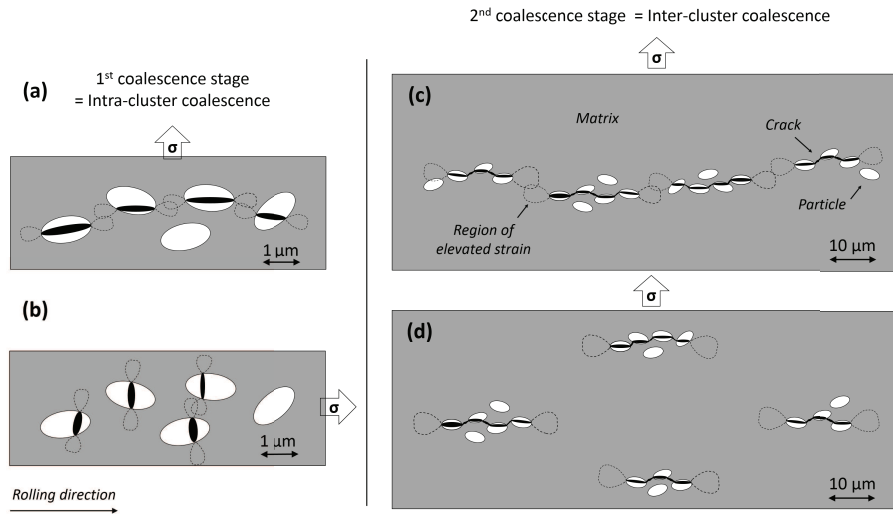


FIGURE 4.7: Schematic of the effect of microstructural anisotropy on the damage percolation process. The effect of the stringer orientation on the first coalescence stage (i.e. intra-cluster) is shown in (a) and (b). The effect of the clusters distribution on the second coalescence stage (i.e. inter-cluster) is shown in (c) and (d). In the case of discontinuous stringer (d), a second void growth process based on the early coalesced voids sets in until second coalescence.

The final fracture is triggered by inter-cluster coalescence which is favoured when the loading is in the transverse direction because clusters are closer to one another (see Fig. 4.7(c) and (d)). Furthermore, this second coalescence stage is likely to occur much faster in the case of an anisotropic large-scale clustering, i.e. linear clusters

forming almost continuous elongated bands (see Fig. 4.7(c)). On the opposite, the effect of particle clustering on the second stage of void coalescence will be weak in case of a more homegenous distribution of clusters (see Fig. 4.7(d)), because their interaction is weaker.

According to the unit cell model of Thomson[130], intra-cluster coalescence occurs by internal necking of the intervoid ligament. In contrast, the second coalescence stage between distant microcracks occurs through void-sheeting, i.e. internal shearing of the ligament, and this is favoured by the nucleation of a secondary void population. Bandstra *et al.* [224] also observed a void-sheet failure mechanism at high triaxiality in a HY-100 steel plate. The void-sheet coalescence mechanism was explained by a strong tendency for intense strain localization between stringers of inclusions despite their large spacing. According to this study, the nucleation of a secondary void population between the primary voids enhances (but does not cause) the plastic strain localization that results in void-sheet coalescence. This is in good agreement with what has been observed in Fig. 4.6.

The anisotropy of ductility observed in the Al 6056 is closely related to stringer-type particle clustering along the rolling direction. However, this type of anisotropic microstructure is also found in the Al 6061 and the Al 6005A for which the fracture strain is close to isotropic (see section. 4.3.2.1). The results of section 4.3.2.3 indicate that the dependence of the ductility on the loading direction in the Al 6056 is linked to the length scale over which the particle distribution exhibits a strong degree of anisotropy. Indeed, the Al 6056 exhibits a strong anisotropic large-scale clustering of particles, i.e. particle clusters are much closer to each other in the rolling direction compared to the transverse direction. This is schematically represented in Fig. 4.8.(a). This proximity of clusters creates an easy percolating crack path along the rolling direction as a crack can extend along this direction throughout the material without the need to cross a tougher matrix material (presenting much less damage), see Fig. 4.8(b) and (c).

On the opposite, particle stringers are more isolated in the case of the alloys Al 6061 and Al 6005A (see Fig. 4.8(d)). The presence of

4.4. Discussion on the origins of the failure anisotropy

tougher matrix between these clusters strongly reduces the detrimental effect of the clusters and the anisotropy of fracture properties by allowing a second stage of stable void growth between clusters (see Fig. 4.8(*e*) and (*f*)). It is thus the degree of continuity of the particle stringers over significant distances, i.e. large-scale clustering, which sets the failure anisotropy in Al 6056.

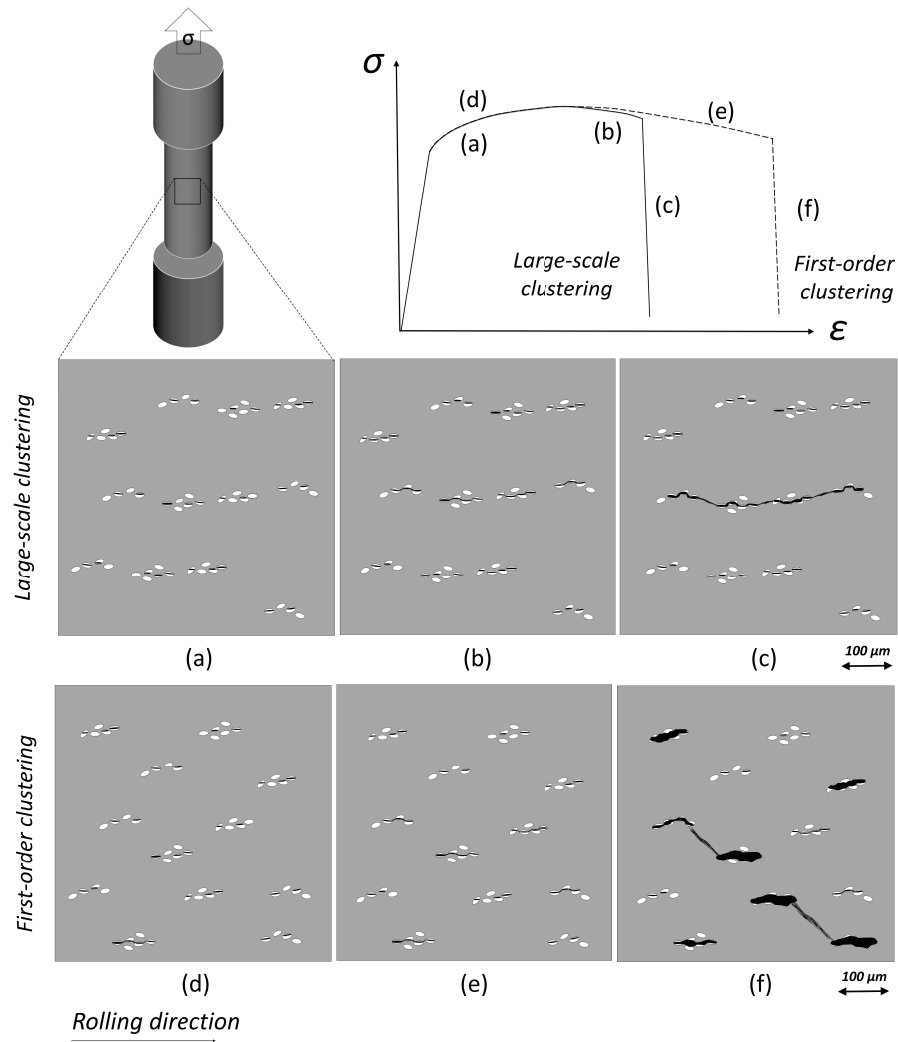


FIGURE 4.8: Schematics of the damage sequence in Al 6056 ((a), (b) and (c)), and in Al 6061 and 6005A ((d), (e) and (f)).

4.5 Conclusion

The hardening and fracture properties of three aluminium alloys of the 6xxx series have been determined for different heat treatment

4.5. Conclusion

conditions and for two different loading directions. The key results of the study are the following:

- The macroscopic tensile tests indicate that the yielding and hardening behaviour is almost isotropic for the three alloys. The fracture strain is significantly larger in the rolling direction compared to the transverse direction in the alloy Al 6056, but not in the alloys Al 6061 and Al 6005A for which ductility is also isotropic.
- In situ tensile tests during X-ray tomography have confirmed the existence of two coalescence stages of ductile damage which consist of intra- and inter-cluster coalescence.
- The Pair Correlation Function (PCF) is used to quantify the effect of the particle spatial distribution on damage. The PCF analysis has been performed both on the 3D tomography data and on SEM images, leading to very similar results.
- The PCF is shown to be an effective way to quantify the degree of first-order and large-scale clustering within a particle field, and to identify the preferred orientation of these clusters, if any. It also allows comparison of the relative amount of clustering between different particle fields.
- The analysis indicates that it is the degree of continuity of the particle stringers over distances comparable to macroscale, i.e. large-scale clustering, which sets the failure anisotropy. If particle clusters appear under the shape of continuous elongated bands, the ductility of the material is likely to exhibit a significant anisotropy. In the case of shorter and isolated particle stringers, the presence of tougher matrix between these clusters strongly reduces the anisotropy of fracture properties by allowing a second stage of stable void growth at the scale of the clusters. Alloy Al 6056 presents continuous elongated bands at the mesoscale explaining its failure anisotropy.

This study illustrates how the PCF can be used to quantify the effect of microstructural anisotropy on macroscopic failure properties. The results demonstrate the strong influence of mesoscale heterogeneities on the damage evolution in case of heterogeneous material. Nowadays, damage models implemented in FE software are still

usually limited to isotropic behaviour. This work emphasizes the need for better representation of the microstructure heterogeneities, such as clustering and microstructure anisotropy. The studied alloys do not exhibit significant anisotropy of yielding which allowed isolating the effect of the particle distribution on the anisotropy of damage properties. However, in many cases, the anisotropy comes from both contributions. In this case, the PCF analysis could be used as a first step for evaluating the degree of anisotropy which is expected for a specific microstructure and for producing statistically representative particle fields. Furthermore, the quantification of the anisotropy of the microstructure plays a key role in manufacturing. For example, the 6005A has been extruded and this could maybe explain why the clustering is the least pronounced in this alloy. Finally, the PCF analysis is not limited to the question of damage propagation as there are many physical properties which depend on the homogeneity of the microstructure. For example, clusters of particles have been shown to form the most severe corrosion pits [225] and to strongly impact the nucleation of recrystallization [197]. Another example is the role of fibre clusters in the problem of internal stresses in composites related to the difference in thermal expansion during processing [195].

4.5. Conclusion

Chapter 5

In situ tailoring of the fracture length scales in aluminium alloy 6056 by friction stir processing

5.1 Introduction

The understanding of the micromechanisms leading to the catastrophic failure plays a key role in the assessment of the mechanical integrity of structures and in the development of manufacturing process which result in damage tolerant-microstructures. In chapter 3, local stirring using a friction stir processing (FSP) tool has been shown to very significantly increase the fracture strain of the Al alloy 6056 while making it more isotropic. Three reasons for the ductilization have been unravelled: (i) FSP breaks the large intermetallic particles into smaller, and thus stronger, fragments, (ii) FSP closes the pre-existing porosity; (iii) FSP randomizes the particle distribution. **The objective of this work is now to quantify the effect of FSP on the fracture toughness of the Al 6056.**

5.1. Introduction

In some applications, only a surface layer is subjected to the FSP thermomechanical cycle. This study focuses on the case for which the full thickness of the plate may be modified by FSP, i.e. relatively thin plates with thickness ranging typically from 1 to 6 mm. The essential work of fracture (EWF) method introduced by Cotterell and Reddel [226] is thus applied in this work as an alternative to the fracture mechanics approach for characterizing the tearing resistance of thin sheets. The EWF method is useful for toughness-oriented material development and improved microstructure design. However, it is of limited use in structural analysis, except if the essential work of fracture (W_e) can be related to the J-integral at cracking initiation (J_{Ic}) [27]. J_R curves have thus also been obtained for the Al 6056.

The results which are presented in this chapter may be divided into four groups :

Macroscopic characterisation of the ductile damage at **low** triaxiality : Macroscopic tensile tests have been characterized for the base material (Al 6056) and for the FSPed material in a previous part of the thesis, see section 3.1. A few selected results will be recalled in this chapter.

Microscopic characterisation of the ductile damage at **low** triaxiality : The impact of FSP on the damage process has already been described in section 3.1 based on ex situ tensile tests. In this chapter, in situ tensile testing in X-ray microtomography is used to validate the effect of FSP on the nucleation, growth and coalescence of voids.

Macroscopic characterisation of the ductile damage at **high** triaxiality : The EWF method and J_R curves are used to characterize the fracture toughness of the base material and the FSPed material.

Microscopic characterisation of the ductile damage at **high** triaxiality : The impact of FSP on crack initiation and propagation is analysed based on in situ CT-like specimens testing in X-ray microlaminography.

Whether the crack results from a void coalescence process (e.g. in tensile specimens) or pre-exists (e.g. in the fracture mechanics specimens), a length scale enters the ductile failure process during crack propagation [7]. Indeed, damage localizes within a narrow band of width being determined largely by the microstructure of the material

[227]. Damage process has traditionally been modelled by assuming a periodic distribution of voids, and it is generally assumed that damage localizes within a band of thickness scaling with the averaged void spacing. The definition of this length scale, i.e. the size of the fracture process zone, plays a key role in ductile failure as it affects the total energy dissipated during the deformation and damage process [7]. In the case of intrinsically heterogeneous materials, such as the studied Al 6056 aluminium alloy, a larger length scale enters the damage process due to particles clustering. A similar result has already been obtained by Achon *et al.* [131], who showed that the characteristic distance which is relevant to the fracture toughness of two Al alloys (7075 and 7475) is the distance between particles clusters in the fracture plane rather than the mean distance between particles.

5.2 Experimental methods

5.2.1 Materials and processing

The Al 6056 alloy was provided in the form of cold rolled plates. The thickness of the plates is equal to 2.5 mm. The plates are initially in T4 condition. Six overlapping FSP passes were performed with a tool rotation rate of 500 rpm and a traverse speed of 200 mm/min along the rolling direction. The tool was composed of a 20 mm diameter scrolled shoulder prolonged by a 2 mm long cylindrical M6 threaded Triflat pin. After FSP, all samples were kept at room temperature for at least 1 month to allow natural ageing. Similar FSP parameters have been shown to improve the fracture strain of the Al 6056 in chapter 3, as already explained in the introduction.

5.2.2 Geometry of the specimens and precracking method

The double-edge-notch-tension (DENT) specimen geometry was used for all fracture tests, see Fig. 5.1(a). All the specimens have a width W equal to 60 mm. The height of the specimen h_0 is 150 mm and the initial ligament length L_0 varies between 5 and 20 mm. The thickness of the specimen t is equal to 2.5 mm. DENT specimens were machined

5.2. Experimental methods

in the Al 6056 plates in two different directions, either perpendicular or parallel to the rolling direction (see Fig. 5.1(b)). In the following, DENT specimens with loading direction (i.e. the direction perpendicular to the ligament) along the rolling and the transverse direction are called DENT-L and DENT-T, respectively. In the case of the FSPed material, the DENT specimen were machined with the notch along the processing direction (see Fig. 5.1(c)) and are named DENT-FSP.

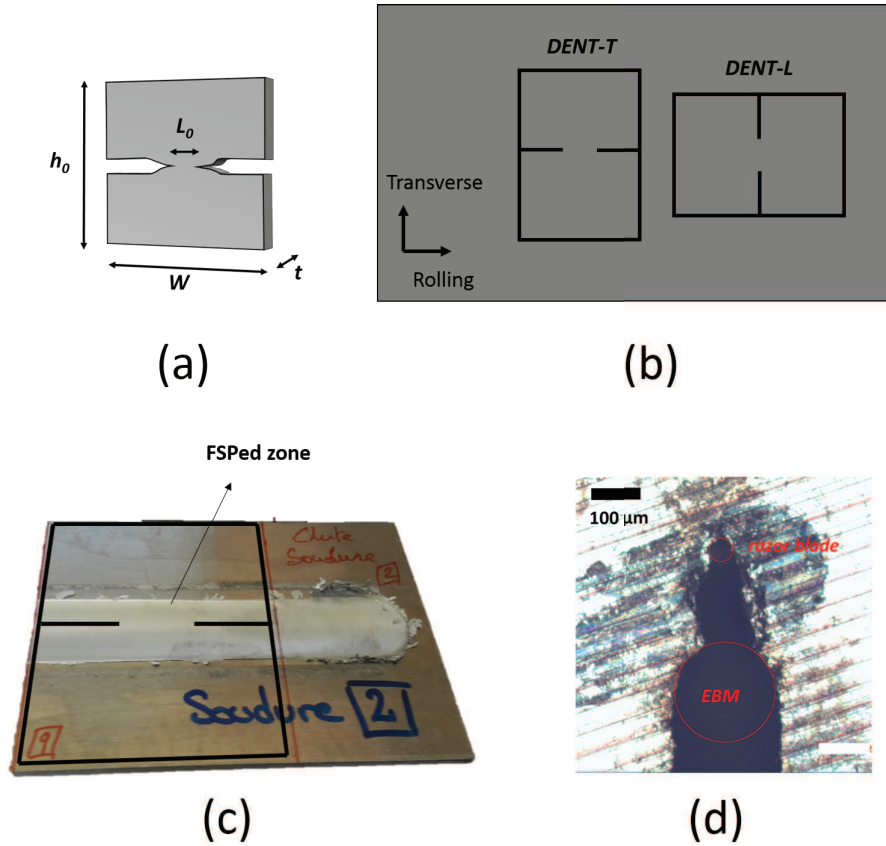


FIGURE 5.1: (a) Geometry of the DENT specimens. DENT configuration of the (b) DENT-T, DENT-L specimens and (c) DENT-FSP specimen. (d) Tip of the notch after electrical discharge machining (EDM) and final machining using fresh razor blades.

The notches of the DENT specimens were machined by electrical discharge machining (EDM). Initial notch tips were then made using

fresh razor blades, leading to notch tip radii of approximately $25 \mu m$, see Fig. 5.1(d). The initial ligament lengths L_0 were measured before testing by use of a travelling microscope. In theory, fracture mechanics requires the precrack tips before the test to be perfectly sharp. In practice, it suffices to have an initial opening of the precracks that is significantly smaller than the crack tip opening displacement at the initiation of cracking [228].

5.2.3 Essential work of fracture

The essential work of fracture (EWF) concept has been introduced by Cotterell and Reddel in their seminal paper published in 1977 [226]. It is based on the idea that the ductile fracture process can be divided in two parts: the fracture process zone (FPZ) at the tip of the crack which is surrounded by a larger outer plastic zone (PZ). Cotterell and Reddel proposed to express the total work of fracture W_f as the sum of an essential work of fracture W_e spent in the FPZ and a plastic work W_p dissipated in the large outer plastic zone (see Fig. 5.2).

The "non essential" plastic work W_p depends on the geometry and the size of the specimen. In principle, the essential work of fracture is independent of specimen geometry [229]. In practice, the double-edge-notch-tension (DENT) specimen is used most of the time. Indeed, the stress state between the notches is tensile and there is no buckling problems [230]. Furthermore, the notch of the DENT specimen constrains the deformation so that the neck spreads along the crack line. For example, there is no such restraint for a deeply centre-notched (DCNT) specimen and necking occurs along lines at an angle of approximately 35° [229].

If the ligament is completely yielded before fracture initiation and if the plastic zone is confined to the ligament, then the plastic work W_p dissipated during fracture is proportional to the volume of the plastic zone at initiation. On the other hand, the work performed in the FPZ is proportional to the fracture area. The total work of fracture W_f calculated from the area of the force-displacement (F-u) curve is thus given by :

$$W_f = W_e + W_p = w_e \cdot L_0 t + \beta w_p \cdot L_0^2 t \quad (5.1)$$

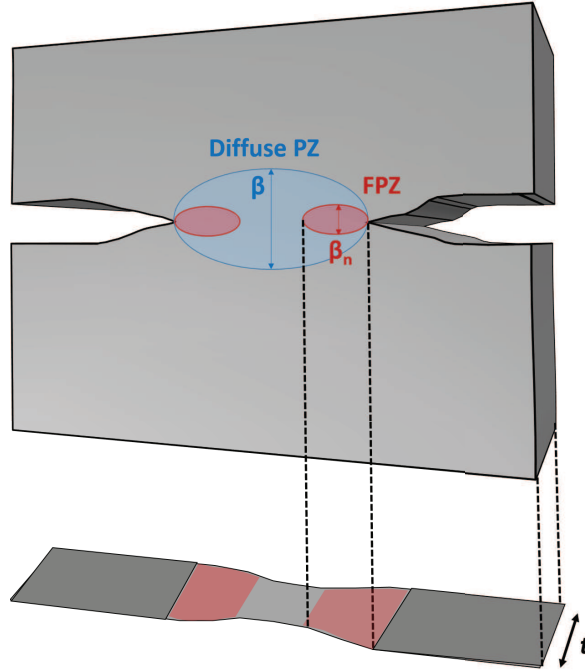


FIGURE 5.2: Schematic of the energy partitioning between the non-essential work of fracture dissipated within the outer plastic zone (PZ) in blue and the essential work of fracture (damage and necking) dissipated in the fracture process zone (FPZ) in red.

where L_0 is the ligament length (see Fig. 5.1(a)), t is the sheet thickness, β is a shape factor related to the form of the plastic zone and w_e and w_p are, respectively, the specific essential work of fracture (J/m^2) and the specific non-essential work of fracture (J/m^3), i.e. related to plasticity in the ligament. By dividing the total work of fracture W_f by the ligament area ($L_0 t$), the specific total work of fracture can be expressed as :

$$w_f = w_e + \beta w_p \cdot L_0 \quad (5.2)$$

The two works of fracture w_e and βw_p are independent of the ligament length and they can thus be separated if a series of geometrically similar DENT specimens of different ligament length are tested. The essential work of fracture w_e is made of two contributions : the necking work and the fracture work spent for damaging.

Some restrictions on the ligament length should be met for the EWF method to be valid :

Lower Bound : The ligament length must be greater than 3 to 5 times the thickness (t) of the plate. Indeed, the reduction in thickness associated with crack propagation is increasing from the tip of the notch over a distance of the order of the sheet thickness [230]. Once necking is fully established, fracture propagation occurs under a quasi-plane stress state. For very small ligaments, the ligament length becomes of the order of this transition distance and the specific work of fracture is not a linear function of the ligament length, i.e. eq. 5.2 does not hold anymore. An alternative criterion for plane stress conditions is based on the Hill criterion [231] which states that fracture occurs just after the limit load, i.e. once the ligament fully yielded. The maximum stress averaged over the ligament is calculated by dividing the maximum force F_{max} by the ligament cross-section, $\sigma_{max}^{Ligament} = F_{max}/(L_0 t)$. According to Hill criterion, the values of $\sigma_{max}^{Ligament}$ should be independent of the ligament and show a constant value equal to $\sigma_{max}^{Ligament} = C_{lig} \sigma_0$, where C_{lig} is the plastic flow constraint factor equal to 1.15 for DENT specimen [231].

Upper Bound : The ligament length must be small enough in order to ensure that it is fully yielded prior to fracture initiation. In other words, the ligament must be smaller than twice the plastic zone size ahead of the crack tip. The size of the plastic zone can be obtained from Irwin's estimate [230]. In this work, digital image correlation (DIC) is used in order to visualize the plastic zone. More details about the DIC software used in this work will be given in section 5.2.6.3. Furthermore, the ligament must also be small enough to prevent edge effects, i.e. to ensure that yielding does not spread to the outer edges of the specimens [232]. This is generally defined by the condition $L_0 < W/3$, where W is the width of the plates.

All DENT specimens used for the EWF method were strained until completion of cracking using a screw driven universal testing machine. The crosshead speed was 1 mm/min. The displacements were measured by means of an extensometer with an initial gauge length of 50 mm. Load/displacement curves were recorded for all specimens.

5.2. Experimental methods

5.2.4 J_R -curves experimental procedure ¹

J_R curves have been measured for a thickness of 2.5 mm and for loading along the rolling and transverse direction, but not for the FSPed material. A dozen of DENT specimens with initial ligament length L_0 equal to 20mm were tested. Each specimen was deformed by a screw driven testing machine, up to a certain crack advance Δa_{Tear} , different for each test. The deformation speed was set to 1 mm/min and then reduced to 0.25 mm/min when the crack started to propagate. The initial gauge length of the extensometer was equal to 50 mm. After unloading, the specimens are machined, embedded in an resin, ground and polished, see Fig. 5.3.

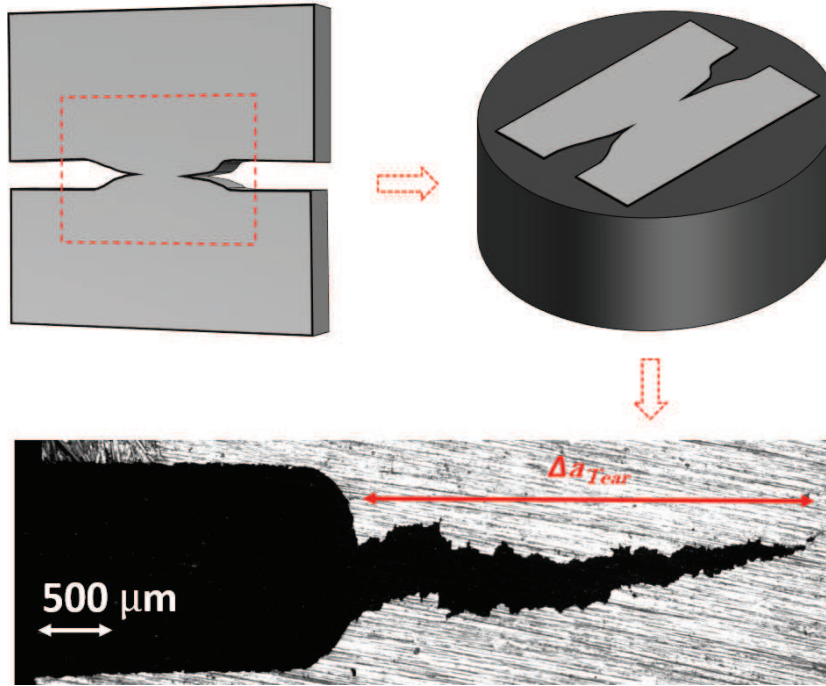


FIGURE 5.3: (a) Machining and (b) embedding of the specimens in the resin in order to measure the crack advance Δa_{Tear} after polishing.

A common phenomenon observed experimentally is the tun-

¹This subsection is mainly based on the methodology proposed in the thesis of F. Hachez [228].

nelling of the crack front, i.e. the crack propagates faster in the center of the plate compared to the surfaces. The tunnelling of the crack has been characterized for DENT cracked plates of different thicknesses made of the alloy Al 6082 [228]. Tunnelling of the crack was observed for plates with a thickness larger than 2 mm and was more and more marked with growing thickness. In order to get a good estimate of the average crack advance, Δa_{Tear} , is measured both in the vicinity of the plate specimen and at the center of the specimen. The crack advance Δa_{Tear} is thus an averaged value over the thickness.

For the DENT geometry, a relationship based on the load-displacement curve has been developed by Rice *et al.* [233] :

$$J_{Rice} = \frac{K_I^2}{E} + \frac{1}{L_0 t} \left(2 \int F. du_p - F u_p \right) \quad (5.3)$$

where K_I is the stress intensity factor ($\text{MPa}\sqrt{m}$), F is the applied load (N) and u_p is the plastic displacement (m). For a DENT specimen, K_I is given by [234] :

$$K_I = \frac{F \sqrt{\frac{\pi a_0}{W}}}{t \sqrt{\frac{W}{2}} \sqrt{1 - \frac{2a_0}{W}}} \left(1.122 - 0.561 \left(\frac{2a_0}{W} \right) - 0.205 \left(\frac{2a_0}{W} \right)^2 + 0.471 \left(\frac{2a_0}{W} \right)^3 + 0.19 \left(\frac{2a_0}{W} \right)^4 \right) \quad (5.4)$$

where a_0 is the initial crack length. The plastic displacement u_{pl} is equal to :

$$u_{pl} = u - u_{el} = u - CF \quad (5.5)$$

where u is the measured displacement, u_{el} is the elastic displacement and C is the compliance of the material. For the DENT geometry, the variation of the compliance C with a change of the specimen dimensions has been fitted as [234]:

$$C = \frac{1}{Et} \left(\frac{H(1 - \nu^2)}{W} + \frac{4}{\pi} \left(0.0629 - 0.061 \left(\cos\left(\frac{\pi a_0}{W}\right) \right)^4 - 0.0019 \left(\cos\left(\frac{\pi a_0}{W}\right) \right)^8 + \ln \left(\sec\left(\frac{\pi a_0}{W}\right) \right) \right) \right) \quad (5.6)$$

5.2. Experimental methods

where H is the initial gauge length of the extensometer (i.e. 50 mm in this case).

5.2.5 Tomography and laminography

5.2.5.1 Tomography

In situ tensile tests coupled to X-ray microtomography have been performed at the TOMCAT beamline of the Swiss Light Source [153]. The mini smooth tensile specimens, with diameter of 1 mm, are deformed up to fracture while the tensile force F is recorded. The data acquisition is made at regular discrete deformation steps. One tensile specimen was machined from the Al 6056 plate along the transverse direction (see Fig. 5.4(b)) and one specimen was machined in the FSPed plate along the processing direction (see Fig. 5.4(c)). The reconstructed volume has a size of $2560 \times 2560 \times 2300$ voxels with a voxel size of $0.6 \mu\text{m}$.

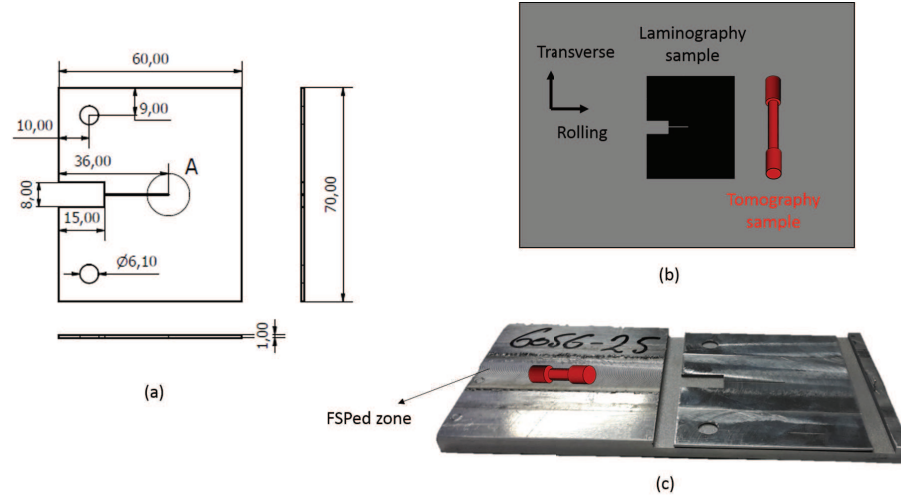


FIGURE 5.4: (a) Geometry of the μCT specimens. (b) Configuration of the $\mu\text{CT-T}$ and $\mu\text{UT-T}$ specimens. (c) Configuration of the $\mu\text{CT-FSP}$ and $\mu\text{UT-FSP}$ specimens.

The mini tensile tests are named $\mu\text{UT-T}$ and $\mu\text{UT-FSP}$ for the Al 6056 specimen loaded along the transverse direction and the as-FSPed

specimen loaded along the FSP direction, respectively. In this case, the prefix μ is used to underline that these tests are performed in situ and imaged with a microscale resolution.

5.2.5.2 Laminography

Synchrotron radiation computed laminography is a 3D non-destructive experimental technique used for scanning laterally extended sheet-like objects [16, 235]. This technique allows to perform damage analysis of the immediate vicinity of the notch root of a flat CT-like specimen. The CT-like specimen geometry is given in Fig. 5.4(a). A dedicated in-situ testing machine described in Ref. [235] has been mounted on the rotation stage of the 3D laminography setup of the ID19 beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The specimens are prepared by using EDM which produce a notch root of radius of about 0.15 mm. One specimen has been extracted in the center of an Al 6056 plate (with the notch along the rolling direction, see Fig. 5.4(b)) and one in the center of a FSPed plate (see Fig. 5.4(c)). These two specimens are called μ CT-T and μ CT-FSP, by analogy with the μ UT specimens. The tensile force F is recorded. The data acquisition is made in a stepwise loading procedure. The reconstructed volume has a size of 2560x2560x2300 voxels with a voxel size of 0.65 μ m. More details on the in situ laminography procedure can be found in Refs [16, 235].

5.2.6 Projection Digital Image Correlation (P-DIC) methodology

5.2.6.1 Principle of the P-DIC

Buljac *et al.* [235, 236] have studied the ductile tearing of thin sheet made of AA 2198 aluminium alloy via in-situ laminography (see Fig. 5.7(a)). The in situ strain fields have been analysed via Digital Volume Correlation (DVC), see Fig. 5.5. The nominal strain profiles in the crack propagation direction (L) show negative levels ($\epsilon_{LL} \approx -0.04$) in the

5.2. Experimental methods

close vicinity of the notch root ($d_{notch}=140\text{ }\mu\text{m}$) and increases up to approximately $300\text{ }\mu\text{m}$ from the notch. Further away from the notch (d_{notch} larger than $\approx 300\text{ }\mu\text{m}$), the strain mean values are close to zero (close to noise level). This behaviour is observed from the very beginning of the loading process (loading steps (0)-(1)) and remains over the loading history (loading steps (1),(2) and (3)). In other words, the region further away from the notch is shown to be in plane strain condition in the crack propagation direction [235]. The observed plane strain condition compares well with previous numerical simulations [237].

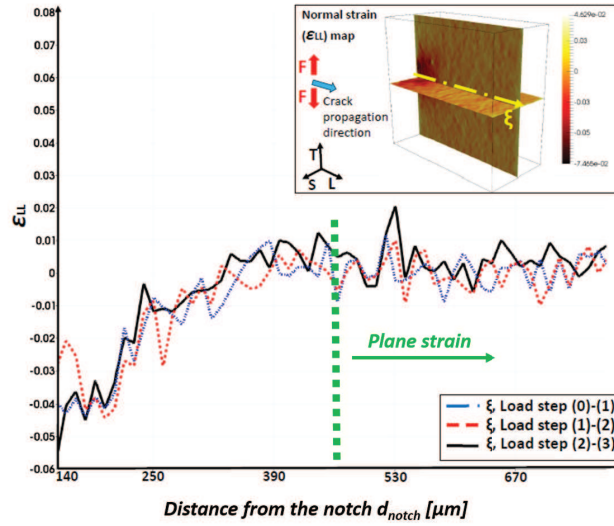


FIGURE 5.5: Nominal strain profiles (DVC measurements), in the AA2198 alloy in T8 state, in the crack propagation direction (L) as a function of the distance from the initial notch root (d_{notch}) for increasing loading steps (0),(1),(2) and (3). Adapted from [235].

This observation led to the development of a new Projection Digital Image Correlation (P-DIC) technique, which constitute an alternative to Digital Volume Correlation (DVC) under certain conditions [236, 238]. The principal idea is the following. In regions far away from the notch (i.e. in plane strain condition), the contrast provided over a certain thickness in the propagation direction is projected onto one single slice of material. Indeed, the plane strain condition in the propagation direction implies that the strain field in a plane perpendicular to the propagation direction is independent of the distance from the ini-

tial notch root d_{notch} , at least for small variation of d_{notch} far away from the notch. The “pseudo” 2D images obtained by projection of the contrast can thus be used to perform in-plane correlation, i.e. classical 2D Digital Image Correlation (DIC).

Fig. 5.6 compares the normal strain field, i.e. strain field in a plane perpendicular to propagation direction, obtained by using DVC (left) and by using P-DIC (right) for the AA2198 alloy in T3 state [236]. The strain fields are very similar and this indicates that the P-DIC procedure is valid. Furthermore, the P-DIC was also successfully applied on the AA2139 alloy in T8 state [236].

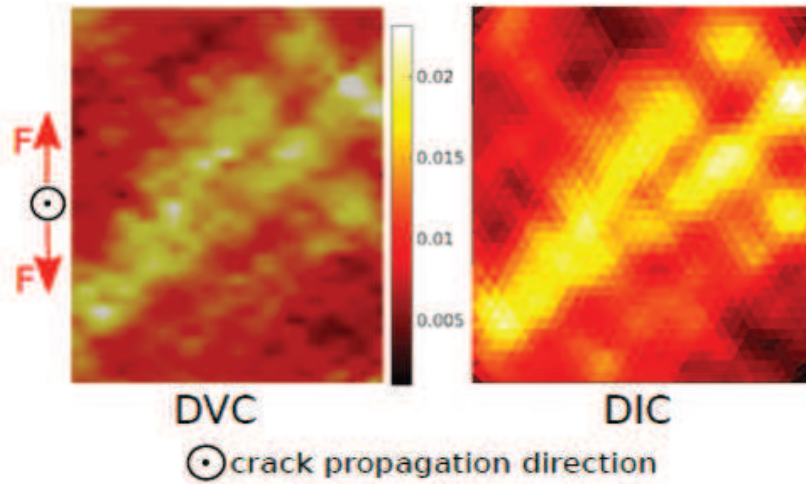


FIGURE 5.6: Comparison of normal strain fields, in the AA2198 alloy in T3 state, in the loading direction obtained by DVC (left) and “projection DIC” (right) on projected 3D data [236]

In this work, the P-DIC procedure has been applied on the laminography data detailed in section 5.2.5. Fig. 5.7(a) shows the geometry and the configuration of the CT-like samples in the loading device. The loading direction is along the transverse direction for the Al 6056 T4 (base material) and along the processing direction for the FSPed Al 6056. Fig. 5.7(b) shows the configuration of the thick slice (80 voxels, i.e. $\approx 50 \mu m$) which has been used for the contrast projection.

Furthermore, the P-DIC has also been applied on tomography data (see section 5.2.5). Fig. 5.7(c) shows the geometry and the con-

5.2. Experimental methods

figuration of the mini tensile specimen in the loading device. Since a cylindrical mini tensile specimen has been used, the deformation field is axisymmetric and there is no angular displacement. In other words, the displacement field within a 2D cross-section through the center of the specimen depends upon the radial and axial coordinate only. The contrast can thus be projected for the slice configuration shown in Fig. 5.7(d). The small angular displacement is neglected by considering the very small thickness of the slice ($50\mu m$) compared to the specimen diameter (1mm).

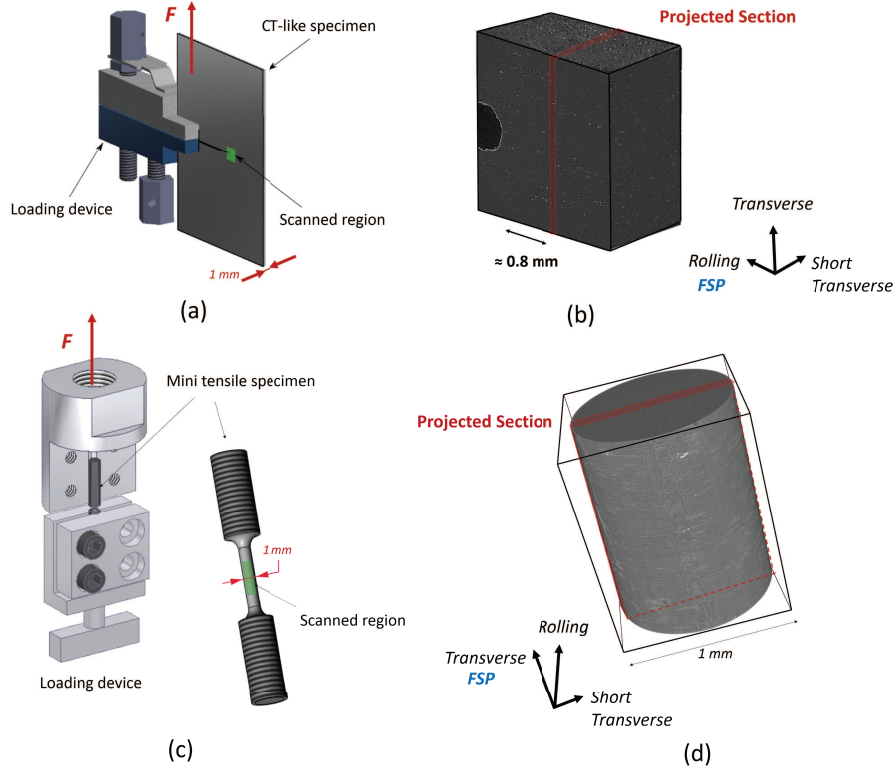


FIGURE 5.7: Laminography experiment : (a) Loading device with the specimen [238], (b) reconstructed volume with positions of the projected section used in P-DIC. Tomography experiment : (c) Loading device with the specimen (adapted from [81]), (d) reconstructed volume with positions of the projected section used in P-DIC.

The “pseudo” 2D images used for the P-DIC procedure are

obtained by keeping only the maximum gray level values (*MAX*-projection). These *MAX*-projections contains mainly the matrix, inter-metallic particles and some white phase contrast fringes around voids, see Fig. 5.8. The *MAX*-projection improves the contrast compare to a single slice (i.e. a section of the volume with a thickness of one voxel) both for laminography and tomography data.

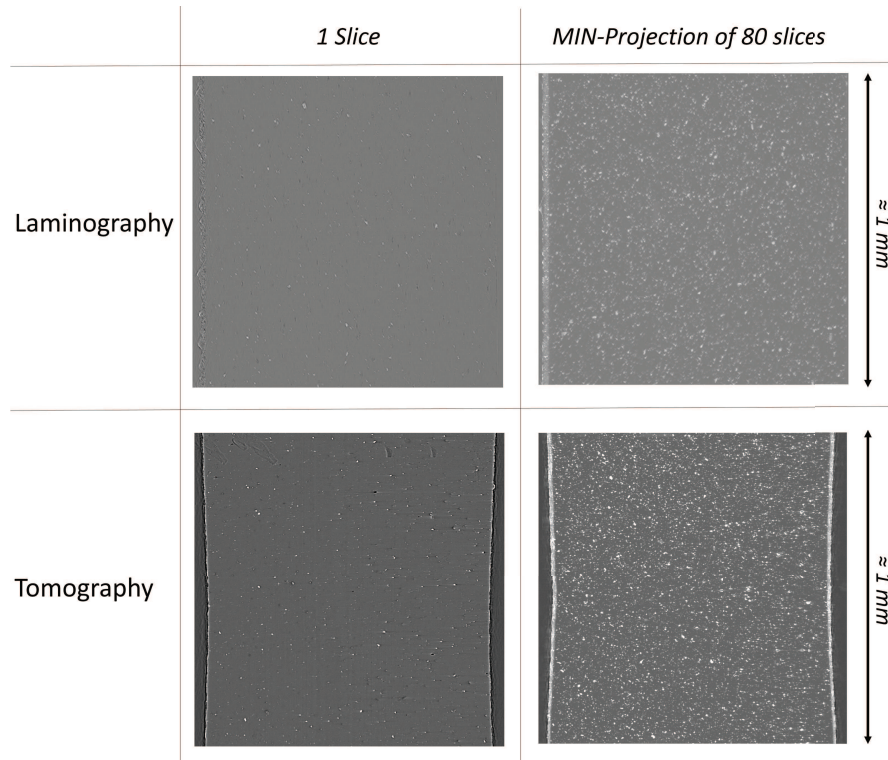


FIGURE 5.8: Comparison between a single slice and a *MIN*-projection of 80 slices for laminography data and tomography data obtained on the FSPed Al 6056. The *MIN*-projection enhance the contrast compared to one slice.

The normal strain fields obtained based on *MAX*-projections will be plotted over the corresponding *MIN*-projection, which is performed by keeping only minimum gray level values, i.e. keeping only the voids (see the following section 5.2.6.2). In other words, the particles are used as natural markers to performed the P-DIC analysis and the result (i.e. the normal strain field) is superposed on the voids configuration.

5.2.6.2 Local distribution of porosity

MIN-projection (performed by keeping only minimum gray level value) results in a "pseudo" 2D image containing the void distribution within the matrix. An example of *MIN*-projection is shown in Fig. 5.9(a). Note that the thickness of the projected slice (i.e. $50\mu\text{m}$) is much smaller than the size of the projection (i.e. $\approx 1\text{mm}$ side). The map is thus an indicator of the "in-plane" value, i.e. the fluctuations are not due to the projection. It is clear that some regions of the matrix contain higher local volume fraction of voids. In order to visualize better the degree of inhomogeneity of the voids distribution, the *MIN*-projection has been segmented and a "finite-body" tessellation has been performed. Details of the "finite-body" tessellation are given in section 3.3.1.2. This procedure allows to compute a local measurement of the void volume fraction F_V^{local} . A zone of the matrix with a small number of voids will have a small F_V^{local} . On the opposite, an elevated value of F_V^{local} corresponds to an element of the matrix in which voids are concentrated. These local values are then interpolated in order to compute a continuous map of the local porosity values, see Fig. 5.9(b). The color bar scale of the F_V^{local} map is systematically adjusted between 0 and $6F_V^{global}$, where F_V^{global} corresponds to the global void volume fraction. In other words, a zone in blue/red corresponds to a zone of the matrix with a small/large local porosity, respectively.

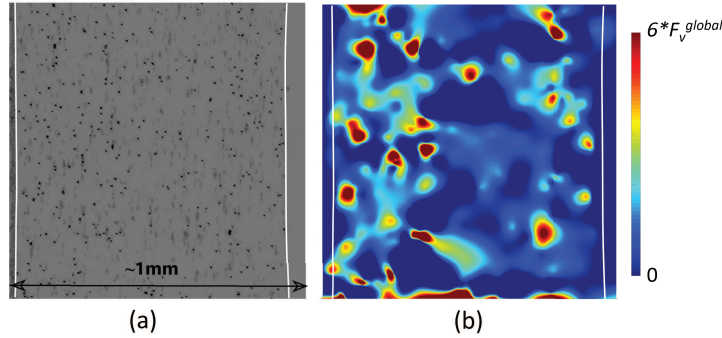


FIGURE 5.9: Example of the "finite-body" tessellation procedure applied on the *MIN*-projection shown in (a). The corresponding map of F_V^{local} is shown in (b). A zone in blue/red corresponds to a zone of the matrix with a small/large local porosity, respectively.

5.2.6.3 Digital Image Correlation (DIC) procedure

Digital image correlation (DIC) has been performed using the open-source *Ncorr* software [239]. The basic idea of the *Ncorr* DIC algorithm is to obtain a one-to-one correspondence between small subsections of the reference image (called subsets) in the reference (initial undeformed picture) and current (subsequent deformed pictures) configurations. For each subset, the displacement and strain information can be obtained through the transformation used to match the location of the subset from the reference in the current configuration. Increasing the subset or step sizes effectively decreases the intensity of the strain noise, but also decreases spatial resolution [240]. This is associated with the classical trade-off between spatial resolution and measurement uncertainties.

Carter *et al.* [240] have observed that increasing the subset size to reduce signal noise intensity produces less spatial resolution loss than increasing the step size. The objective of the analysis is to identify a potential plastic strain localization within the material and this requires a good spatial resolution. Therefore, the step size has been kept as low as possible while using a moderate subset size in order to minimizing signal noise and also maintaining the best spatial resolution. A subset size of 50 pixels and a step size of 4 pixels were selected because they allow a reasonable compromise between noise and resolution. In the following sections, these parameters have always been kept constant. Segmentation and counting of the white features (or markers) in the MAX-projection shown in Fig. 5.8 has been performed. The result indicate that a subset size of 50 pixels contains an averaged of ≈ 9 markers which, at least in part, justifies this subset size. The "optimization" of subset and step size remains quite subjective, but the strain uncertainty that they set has been carefully studied, as detailed in the next section.

Fig. 5.10(a) shows the vertical and horizontal displacement field computed between two MAX-projections. The displacement gradients obtained through the correlation are always somewhat noisy and must be "smoothed" in some way before calculating the strain fields. In the software *Ncorr*, a "strain window" is used for calculating the strain field. It uses a least squares fit on a subset of displacement data to find the best-fitting plane, see Fig. 5.10(b). The size of the subset which is used to "smoothen" the displacement field is named the strain radius.

5.2. Experimental methods

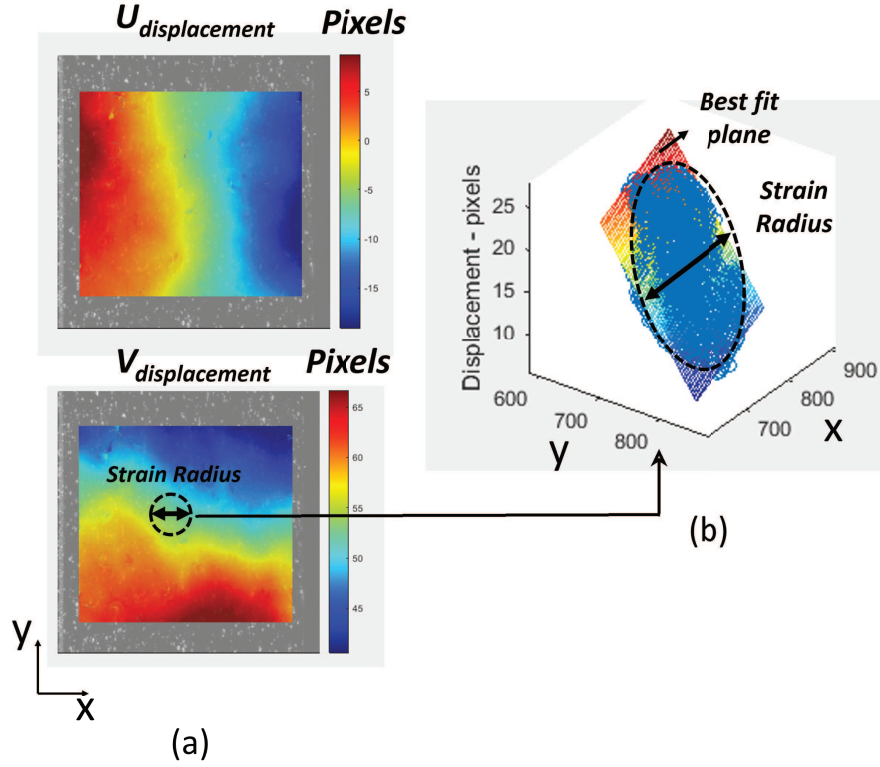


FIGURE 5.10: (a) Example of vertical (V) and horizontal (U) displacement fields. (b) 3D view of the strain radius used to find the "best fit" plane. The strain field is then computed based on this "best fit" plane.

Again, there is a trade-off between spatial resolution and measurement uncertainties. The strain radius used in the strain calculation acts as a smoothing operation; the larger the window, the larger the smoothing effect [239]. The software *Ncorr* allows to switch between a Lagrangian perspective (using Green-Lagrangian strains) or an Eulerian perspective (using Eulerian-Almansi finite strains tensors).

5.2.6.4 Evaluation of Measurement Uncertainties of Projected-DIC applied to Laminography Data

The procedure used to evaluate the uncertainty associated with P-DIC is presented in this section. Buljac *et al.* [241] have performed a detailed analysis of the effect of different data acquisition parameters on the displacement and strain uncertainties associated with Digital Volume Correlation (DVC) applied to three-dimensional (3D) data sets imaged by synchrotron laminography. Their analysis showed that the best procedure to fully evaluate measurement uncertainties, i.e. evaluate the more conservative measurement uncertainty values, is the "rigid body motion" (*rbm*) method. The *rbm* method consists in analysing two consecutive scans of the sample in the same given loading state with a deliberately introduced small rigid body motion between the two acquisitions. Since the two scans of the sample are obtained at the same loading state, the strain field within the material is unchanged ². Correlation is then applied on an overlapping zone between the two scans. In theory, the measured field should correspond exactly to the rigid body motion applied to the specimen. The uncertainty associated with the measurement and the correlation can thus be evaluated from the "non-physical" strain field which is measured. The strain uncertainty is defined as the mean of the absolute value of this calculated strain field. The *rbm* procedure is the most conservative because the uncertainty encompass both the effect of noise associated with the laminography technique and the correlation procedure. Furthermore, the reconstruction artefacts (e.g., rings) do not follow the motion, i.e. the reconstruction axis has moved with respect to microstructure features, which has a clear effect on the measurement uncertainties. Other parameters such as image digitalization, beam properties, detector characteristics and the geometry of the scanned specimen may also affect the measurement uncertainty.

The *rbm* procedure is used here to evaluate the uncertainty associated with the P-DIC measurement. Fig. 5.11 shows a slice in the 3D reconstructed volume of the reference and the *rbm* configuration. The overlapping zones resulting from the rigid body motion are shown in order to indicate the horizontal and vertical displacements between

²Note that the *rbm* procedure is performed at a very limited load (the specimen may even be unloaded) in order to avoid stress relaxation.

5.2. Experimental methods

the two configurations. The thick slice of volume used for the MAX-projection, i.e. the projected section, has always the same thickness in this work (i.e. thickness of 80 voxels = $50\mu m$). This is very important because the quality of the correlation is highly dependant on the "amount" of natural markers, i.e. gray levels contrast from the MAX-projection. If the thickness of the projected slice is increased, the amount of markers is increased and the uncertainty of the P-DIC measurement is likely to decrease. The MAX-projections shown in Fig. 5.11 give an idea of the natural marker pattern. Furthermore, the uncertainty analysis is performed on the base material (Al 6056), and not on the FSPed material. However, as explained in chapter 3, the large second phase particles are broken into smaller fragments during FSP. The volume fraction of markers is thus larger for the FSPed material and assessing the uncertainty from the base material is conservative.

Fig. 5.12(a) shows the vertical displacement field obtained for the P-DIC between the two MAX-projections shown in Fig. 5.11. If the measurement was perfect, the displacement field would have the same value everywhere (i.e. the *rbm* displacement along the y axis). The maximum deviation remains low (≈ 3 pixels) and this is a first hint that the P-DIC uncertainty is not too large. Fig. 5.12(b) shows the evolution of the strain uncertainty as a function of the strain radius which is used to compute the strain field. The strain along the x and y axis are noted ϵ_{xx} and ϵ_{yy} , respectively. Fig. 5.12(b) shows that the strain uncertainty is larger for ϵ_{yy} compare to ϵ_{xx} . A possible explanation is linked with the strong anisotropic clustering of particles, i.e. particle clusters are much closer to each other in the rolling direction compared to the transverse direction. These particles are precisely the ones used as markers. In other words, the spatial distribution of markers used for correlation is anisotropic. As already explained, the strain uncertainty decreases when the strain radius increases due to the "smoothing" effect. In the following sections, a strain radius equal to 25 pixels has systematically been used because it represents a good trade-off between spatial resolution and measurement uncertainties. Fig. 5.12(b) shows that a strain radius of 25 pixels yields a strain uncertainty lower than 0.01 in both direction. This value represents the limit below which the estimated strain levels are not trustworthy.

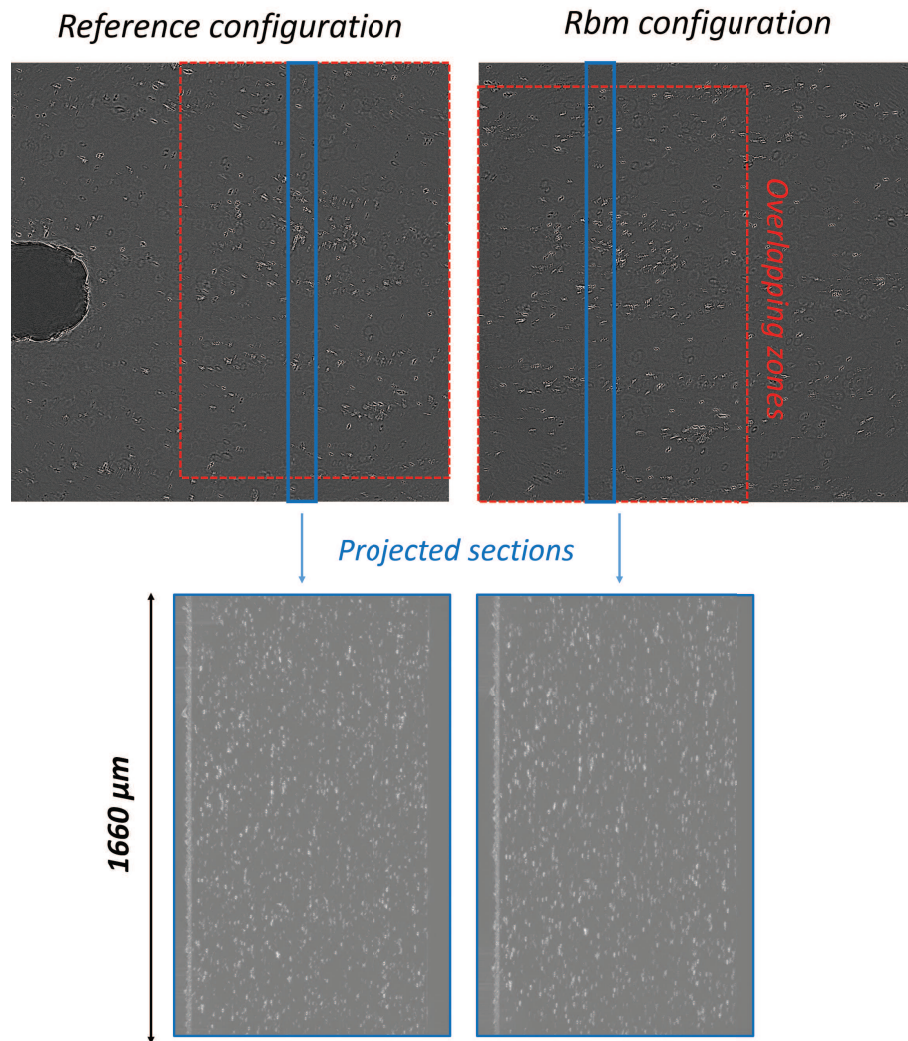


FIGURE 5.11: Region of interest considered in the uncertainty analysis of the volume made of Al 6056. The overlapping zones resulting from the rigid body motion are shown (in red) in order to indicate the horizontal and vertical displacements between the two configurations. The sections used for projection are shown in blue and the projections are shown in the bottom of the image.

5.2. Experimental methods

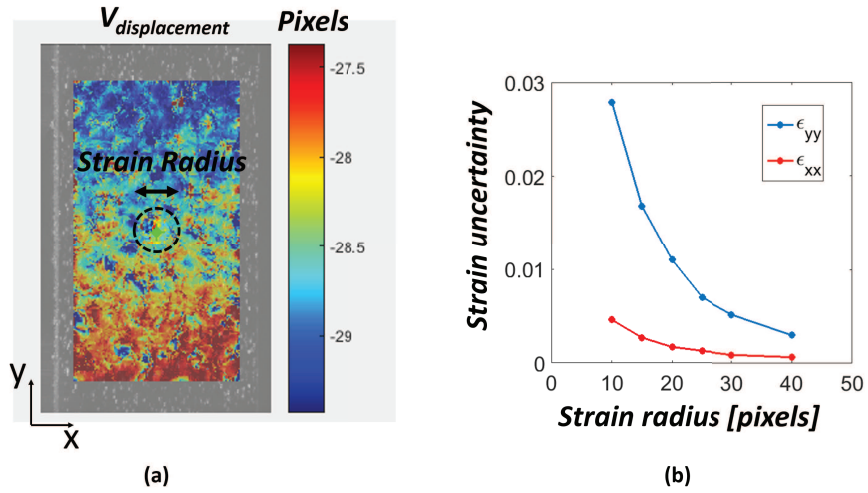


FIGURE 5.12: (a) Vertical displacement field resulting from the correlation between the two projections shown in Fig. 5.11. (b) Evolution of the strain uncertainty with the size of the strain radius.

5.3 Results

5.3.1 Uniaxial tensile test

A few selected results from macroscopic uniaxial tensile tests (cylindrical specimens, 4 mm in diameter) obtained in chapter 3 and used in this work are recalled in this short subsection. The true stress-true strain curves of the Al 6056 (T4 condition) loaded along the rolling (UT-L) and along the transverse direction (UT-T) are shown in Fig. 5.13. Low plastic anisotropy is observed, i.e. the tensile curves are very similar for both loading directions. However, the fracture strain (defined as $\varepsilon_f = \ln(A_0/A_f)$, where A_0 and A_f are the initial and final cross-section area) is significantly larger when loading along the rolling direction ($\varepsilon_f=0.78$) compared to loading along the transverse direction ($\varepsilon_f=0.4$). In the as-FSPed material, the yield stress is slightly decreased but remains similar to the base material, i.e. similar to a T4 condition. However, the FSP increases the ductility and $\varepsilon_f \approx 1$ after 6 passes of FSP. Note that the strain at necking, image of the strain hardening ability, is slightly reduced. Furthermore, FSP leads to an isotropic fracture behaviour, i.e. ε_f is very similar regardless of the FSP direction (see chapter 3 for more details).

5.3.2 EWF results

5.3.2.1 Validity of the EWF method

First, the validity conditions required to apply the EWF method (as explained in section 5.2.3) are checked.

1) Digital image correlation (DIC) is used in order to characterize the plastic zone at the onset of cracking. Fig. 5.14 shows the strain fields in loading direction (ε_{yy}) observed for DENT-L and DENT-T specimens with different ligament lengths L_0 . The strain fields are shown for the last image before the onset of crack propagation is observed. For a ligament length L_0 of 5.6 and 14 mm, the ligament is fully yielded before crack propagation. However, for large ligament length, the value of ε_{yy} at the center of the ligament is very low. This procedure has been

5.3. Results

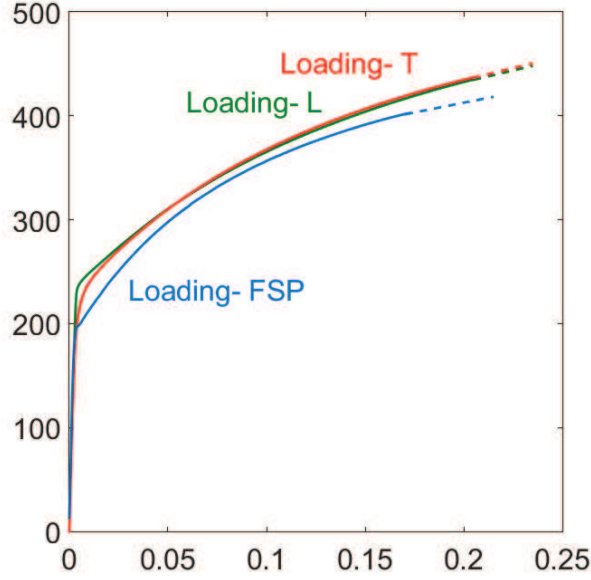


FIGURE 5.13: True stress-true strain curves for the Al 6056 in T4 condition loaded along the rolling (UT-L) and along the transverse direction (UT-T) and the as-FSPed material loaded along the FSP direction (UT-FSP).

performed for each fracture test and the ligament was considered completely yielded at crack initiation if the value of ε_{yy} at the center of the ligament was larger than 0.03. This value is conservative compared to the theoretical value for the elastic strain at crack initiation, i.e. $\varepsilon_{el} = \sigma_{max}^{Ligament} / E \approx 0.005$). It was chosen considering the DIC strain uncertainties and the fact that crack initiation has been detected on the surface of the sample (i.e. neglecting the tunnelling effect).

2) Furthermore, the plane stress condition has been assessed based on the Hill criterion. The values of $\sigma_{max}^{Ligament}$ are plotted against the ligament lengths L_0 and compared with the theoretical value in plane stress ($\sigma_{max}^{Ligament} = 1.15\sigma_0$ for DENT specimen). Fig. 5.15 shows the evolution of $\sigma_{max}^{Ligament}$ for a serie of DENT-T specimens. For large ligament lengths, $\sigma_{max}^{Ligament}$ tends to the value $1.15\sigma_0$. However, $\sigma_{max}^{Ligament}$ increases with decreasing ligament length due to a mixed mode stress state (plane stress/plane strain) and the EWF method becomes less valid. In the

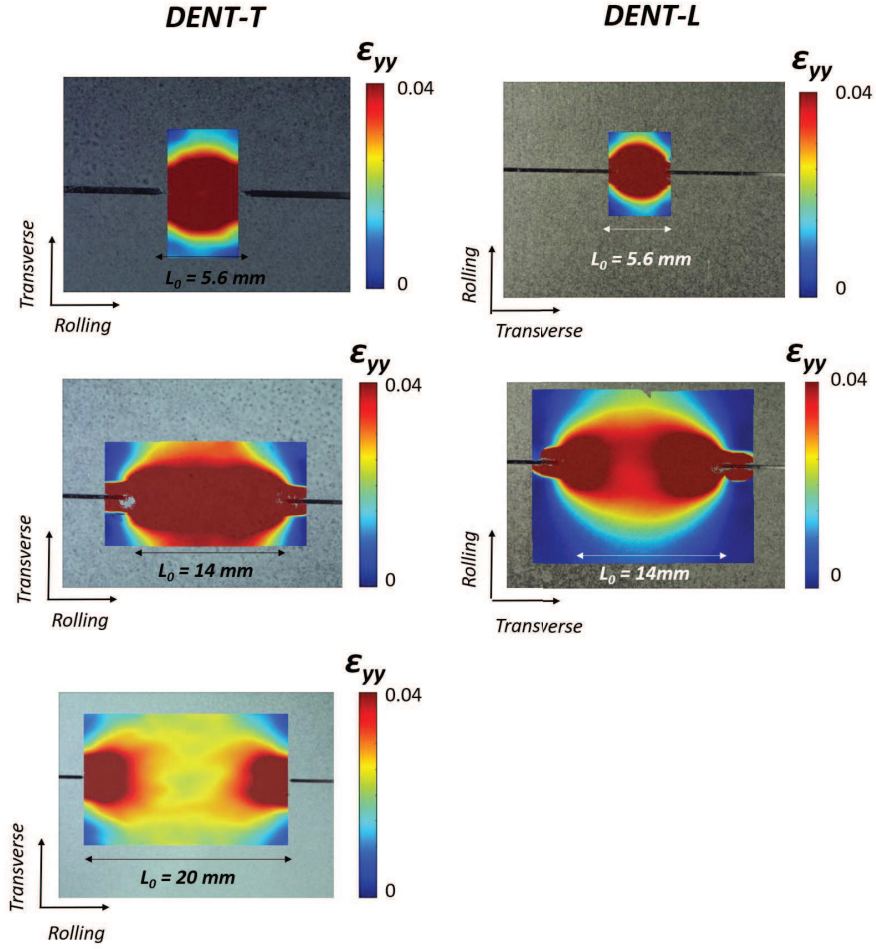


FIGURE 5.14: ε_{yy} strain fields for (Left) DENT-T and (Right) DENT-L specimens just before the onset of crack propagation. The initial ligament length L_0 is indicated on each figure.

following, specimens associated with a large increase of $\sigma_{max}^{Ligament}$ have been excluded and the $\sigma_{max}^{Ligament}$ is given for the specimens which have been used.

5.3. Results

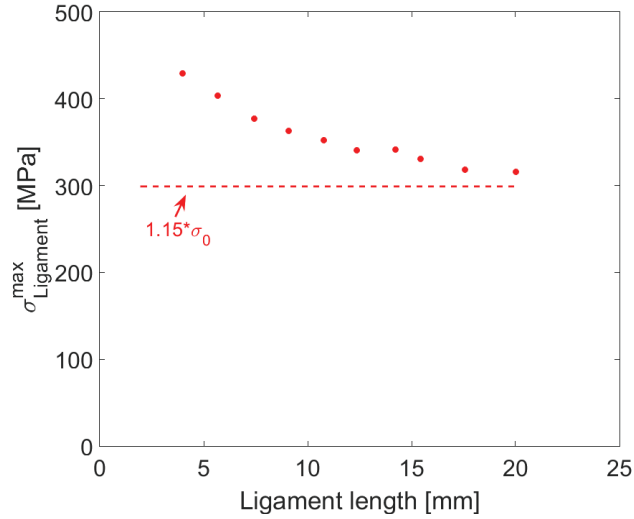


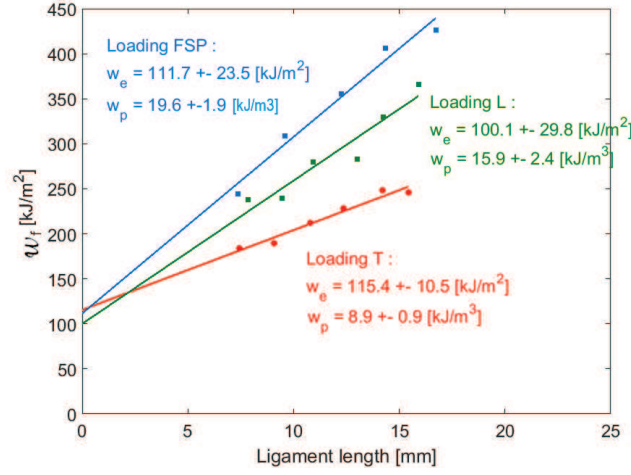
FIGURE 5.15: Effect on the ligament length L_0 on the averaged stress acting on the ligament $\sigma_{max}^{Ligament}$.

5.3.2.2 Determination of the essential work of fracture w_e

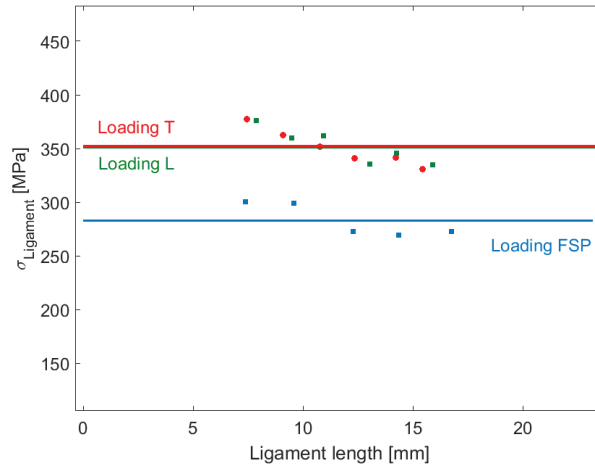
Fig. 5.16(a) shows the linear regressions on the values of the specific total work of fracture w_f for the DENT-T, DENT-L and DENT-FSP specimens. All the samples have a thickness equal to 2.5mm. The extrapolated values to a zero ligament length gives the specific essential work of fracture w_e for these three conditions and w_e is equal to $100 \pm 30 \text{ kJ}/m^2$ for DENT-T specimens, $115 \pm 10 \text{ kJ}/m^2$ for DENT-L specimens and $112 \pm 23 \text{ kJ}/m^2$ for DENT-FSP specimens. It can thus be concluded that the essential work of fracture w_e is similar for all three conditions, with value ranging between 100 and 115 kJ/m^2 .

5.3.2.3 Fractographic analysis

In order to describe the macroscopic geometry of the fracture surface, it has been systematically analysed using a SEM microscope (top view), see for exemple Fig. 5.17(a). Necking develops progressively at the crack tip and after a transition zone, a constant thickness reduction is reached in the fracture process zone (FPZ). This transition length is



(a)



(b)

FIGURE 5.16: (a) Effect of loading direction and effect of FSP on the essential work of fracture for 2.5mm thick Al 6056 sheets. (b) Evolution of the averaged stress acting on the ligament $\sigma_{\text{Ligament}}^{\text{Ligament}}$ for the DENT specimens which have been used in (a).

usually roughly equal to the thickness of the plate [230], in good agreement with what is observed in Fig. 5.17(a). The thickness in the region

5.3. Results

of constant thickness is denoted t_f . The thickness reduction factor at fracture is defined as the ratio t_f/t , where t is the initial thickness of the sheet. The evolution of the thickness reduction factor, i.e. the degree of necking, is shown in Fig. 5.17(b) for the DENT-T, DENT-L and DENT-FSP. The dashed line indicates the averaged value for each specimen type. The degree of necking is similar for all conditions with an averaged value between 80 and 85%.

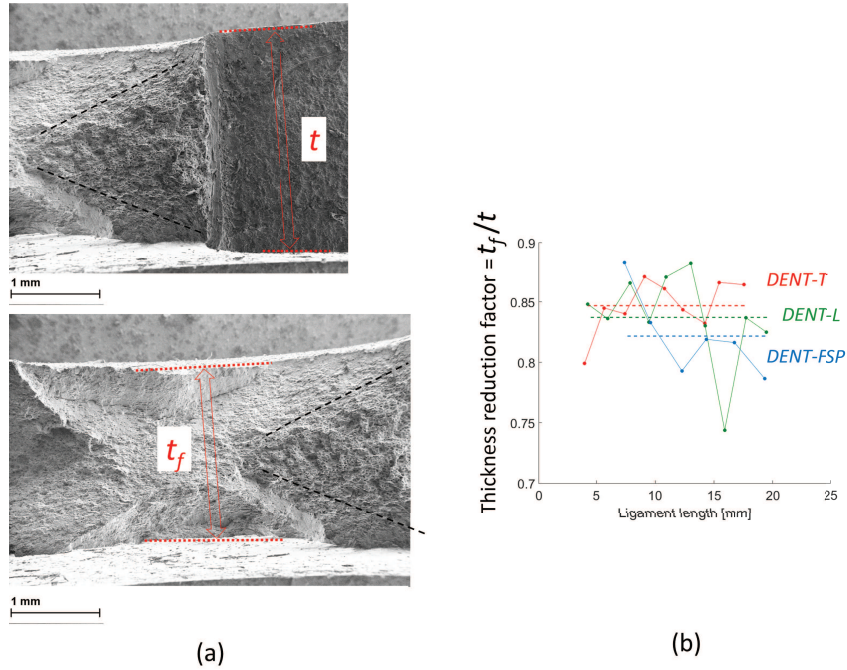


FIGURE 5.17: (a) Method used to measure the thickness reduction factor. (b) Variation of the thickness reduction factor for loading along the transverse and rolling direction.

Fracture surfaces of one DENT-T, one DENT-L and one DENT-FSP specimen (with ligament length L_0 equal to 14mm in each case) are shown in details in Figures 5.18, 5.20, 5.21, respectively. For all samples, the final crack starts to propagate in a flat mode, then tilts and continues to propagate in a slant manner. This is more easily observed in Fig. 5.19, which indicates the height of the fracture surface from the initial notch for the DENT-T specimen of Fig. 5.18. This classical behaviour is consistent with the evolution of the triaxiality factor. Damage growth is more rapid in the flat initiation region than in the propagation region. This

is directly related to the higher stress triaxiality in the initiation region. In the slant zone, the stress triaxiality is lower and the Lode parameter is close to 0. Both of these conditions tend to favour strain localization within a band making an angle with the loading direction equal to 45° [242].

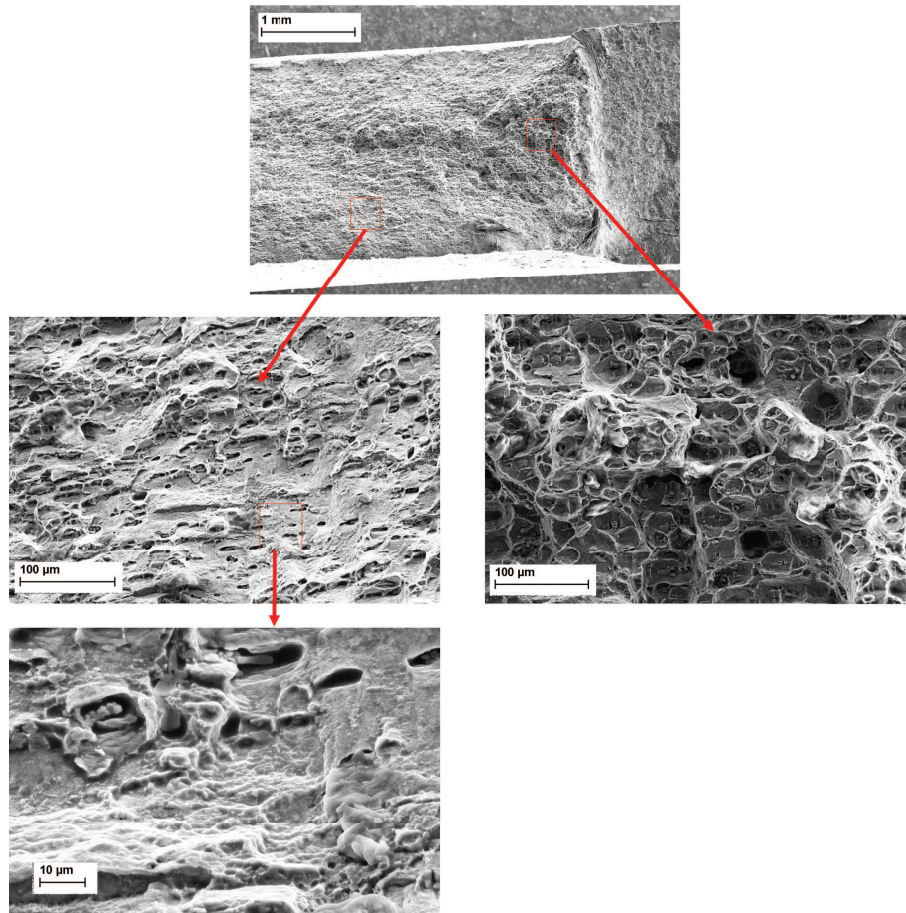


FIGURE 5.18: Fractography of DENT-T specimen in the T4 condition.

In all specimens, large primary dimples are observed in the flat region. The number of large dimples per area unit is larger in the DENT-FSP specimen (see Fig. 5.21), compared to the DENT-T and DENT-L specimen. Furthermore, the size distribution of the dimples is broader in the DENT-FSP, see Fig. 5.21. Both of these observations are linked with the fact that FSP breaks the large intermetallic particles

5.3. Results

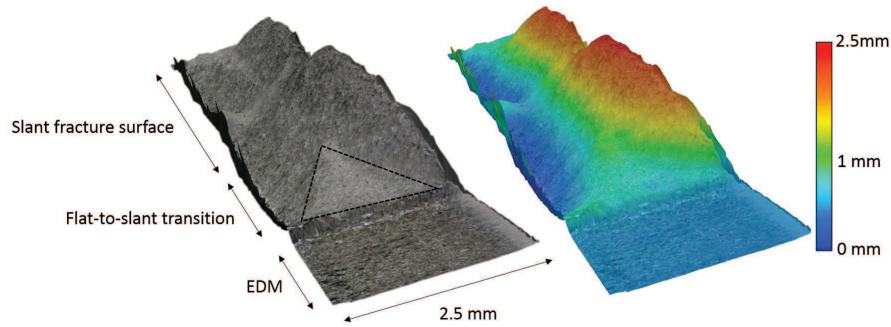


FIGURE 5.19: Fracture surface of an Al 6056 DENT-T specimen indicating the initial flat fracture surface and the subsequent slant fracture under monotonic loading. A transition region connects the flat and the slant parts. The color map indicates height from the initial notch. The height was obtained using a 3D optical microscope.

into smaller fragments. The limited number of large dimples (void diameters of the order of $10\text{-}40\mu\text{m}$) observed in the DENT-FSP fracture surface corresponds to the small fraction of large intermetallic particles which were not broken during FSP.

On the other hand, the fracture surface in the macroscopically slant region presents, in all cases, a relatively low coverage of large dimples (void diameters of the order of $10\mu\text{m}$). Indeed, in the DENT-T and DENT-L specimens, primary dimples are accompanied with some additional regions which are planar, tilted and covered with fine dimples (diameters of the order of $1\mu\text{m}$). This type of fracture surface in the slant region has previously been observed in the 6x56 serie Al alloys [121]. These planar features correspond to a intergranular fracture mechanism, due to void sheets formed by decohesion at dispersoid particles on the grain boundaries. The void sheets act as a rapid coalescence mechanism lowering fracture toughness. In the slant region of the base material, void growth is thus apparently limited by the occurrence of the void sheeting mechanism. In the case of the DENT-FSP specimen, the majority of the slant fracture surface is also covered by small voids. However, in this case the planar features indicating the intergranular fracture mechanism are not observed. This might be due to the grain refinement associated with FSP. Furthermore, the grain boundary coverage decreases somewhat on increasing the severity of

the quench [121], and it is thus likely that there is an effect of the fast cooling rate during FSP.

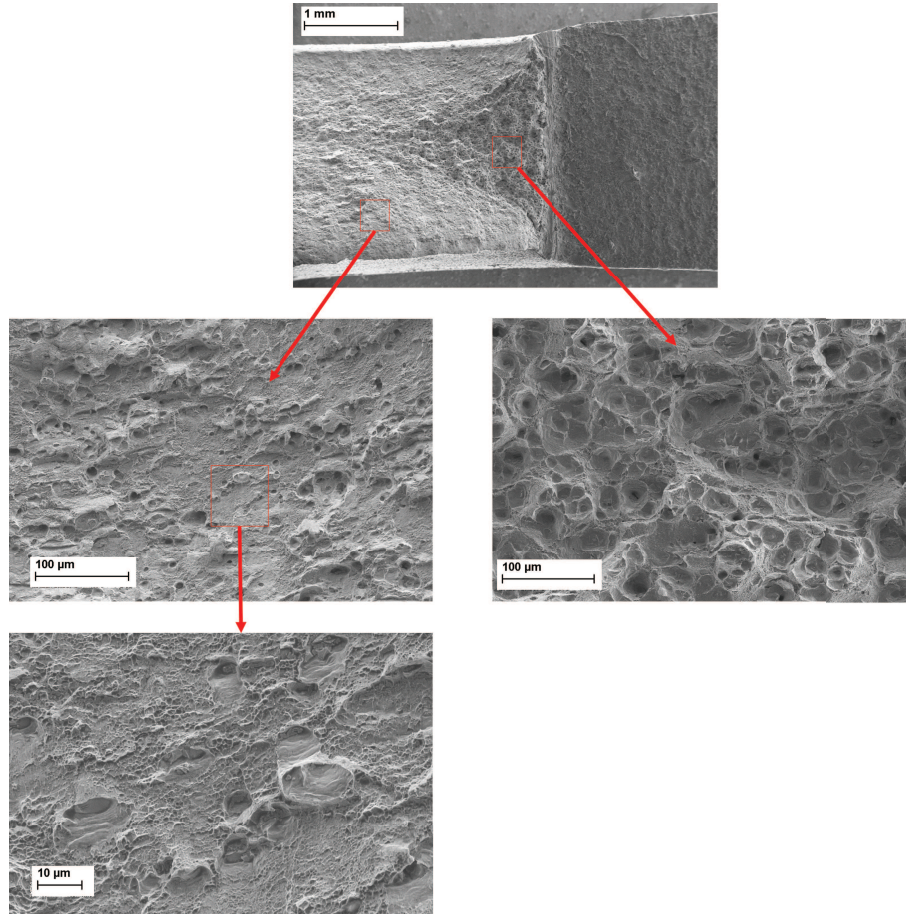


FIGURE 5.20: Fractography of DENT-L specimen in the T4 condition.

5.3.3 J_R curve results

The J_{Rice} integral has been estimated based on the Rice's analytical expressions given by eqs. (5.3, 5.4, 5.5, 5.6). Hachez *et al.* [228] have compared the value of the J_{Rice} integral obtained based on these analytical approximations with the exact J_{Rice} integral value based on 3D finite element simulations. The difference between the two values

5.3. Results

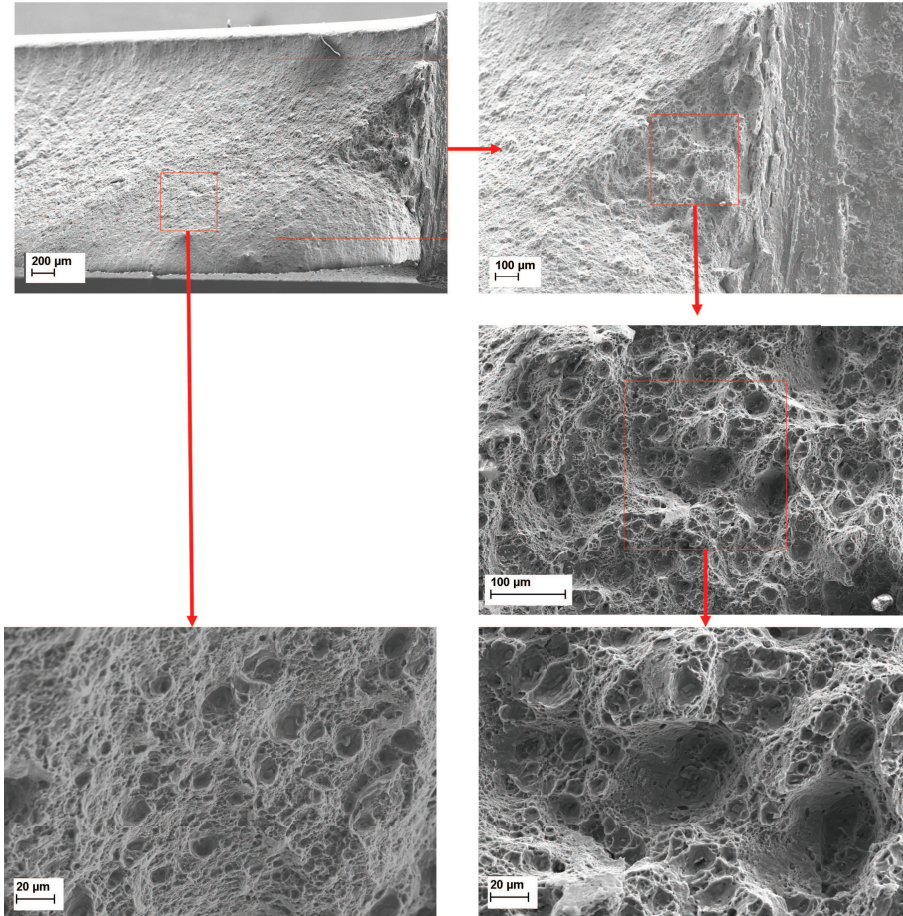


FIGURE 5.21: Fractography of DENT-FSP specimen in the as-FSPed condition (i.e. close to a T4 condition).

was shown to be lower than 10%. The analytical approach will thus be applied.

Multiple DENT specimens have been loaded up to different levels of crack advances, see Fig. 5.22(a). For each test, the area under the load-displacement curve is integrated in order to compute the analytical expression of J_{Rice} . Furthermore, the crack advance Δa_{Tear} is also measured for each specimen, as explained in section 5.2.4. The J_R curve obtained based on DENT-L and DENT-T specimens have been plotted in Fig. 5.22(b).

Unfortunately, in the case of DENT-L specimens, no sample were unloaded sufficiently close to cracking initiation and results are only available for Δa_{Tear} larger than about 1.5 mm. The intersection of the blunting line ($J_{Blunting} = 2\sigma_0 \Delta a_{Tear}$ [243]) is traditionally used to estimate the critical value of the J_{Rice} integral at crack initiation, i.e. J_{Ic} . Due to the limited number of points at small crack advance, it is not possible to give an exact value for J_{Ic} . However, extrapolation of the J_R curve to zero crack advance indicates that J_{Ic} is approximately equal to 100 kJ/m² for the DENT-T specimen. This value is similar with the essential work of fracture obtained in the previous section for DENT-T specimens, i.e. $w_e = 115 \pm 10$ kJ/m².

5.3.4 In situ tensile testing in X-ray tomography

Fig. 5.23(d) and Fig. 5.23(e) shows a 2D cut, just a little before fracture, through the middle of the μ -UT-T and μ -UT-FSP specimen, respectively. The evolution of the minimum cross-sectional area in the necking region (A_{loc} , see Fig. 5.23(b)) provides an estimate of the local true tensile strain $\epsilon_{loc} = \ln(A_0/A_{loc})$ and of the true macroscopic axial stress $\sigma_{zz} = F/A_{loc}$. The tensile stress-strain curves extracted from the in situ data is given in Fig. 5.23(c). It shows that the effect of FSP on the plasticity of the base material remains limited, as already concluded from the macroscopic tensile tests.

The influence of the intermetallic particle spatial distribution can be quantitatively discussed based on Fig. 5.23(d). When loading in the transverse direction, intermetallic particles, which are preferentially aligned along the processing direction, creates an easy damage percolation path. This effect has already been largely discussed in chapter 4. Furthermore, looking at the zoom in Fig. 5.23(c), (d), coalescence events are observed inside clusters (i.e. intracuster coalescence) at deformation levels similar to the uniform elongation (i.e. well before fully developed necking). Intracuster coalescence is a stable event which does not directly lead to material failure but is followed by a second void growth process based on these early coalesced microcracks. In other words, the final fracture is triggered by a second stage of inter-cluster coalescence which leads to the collapse of the matrix ligament between microcracks formed by the intra-cluster coalescence stage. It is thus

5.3. Results

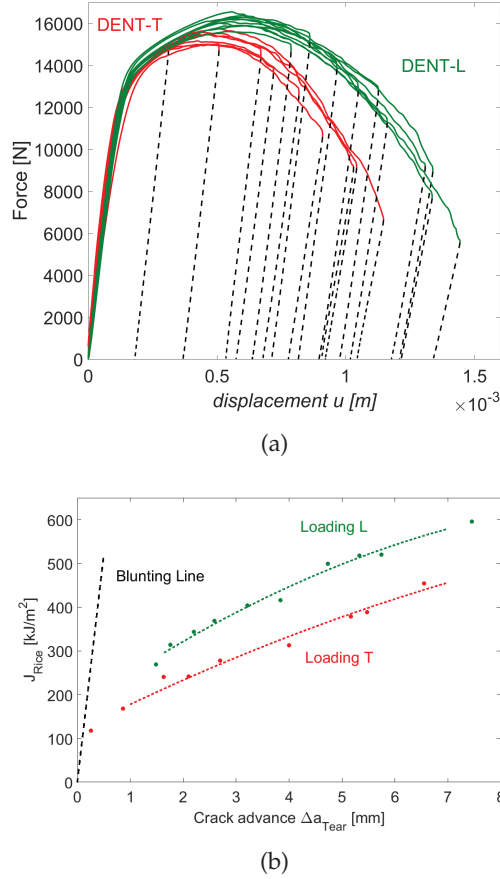


FIGURE 5.22: (a) Loading (plain line) and unloading (dotted line) curves of the multiple DENT specimens. (b) J_R curves for the DENT-T and DENT-L cracked plates made of the alloy Al6056.

the distance between clusters which has to be considered in order to explain the fracture behaviour of the Al 6056.

On the opposite, in the FSPed material (see Fig. 5.23(e)), damage is limited even after significant amount of necking. A very limited number of voids are observed within the FSPed material compared to the base material. As already discussed in details in chapter 3, this ductilization is explained by the fact that: (i) FSP breaks the large inter-

metallic particles into smaller, and thus stronger, fragments, (ii) FSP closes the pre-existing porosity; (iii) FSP randomizes the particle distribution.

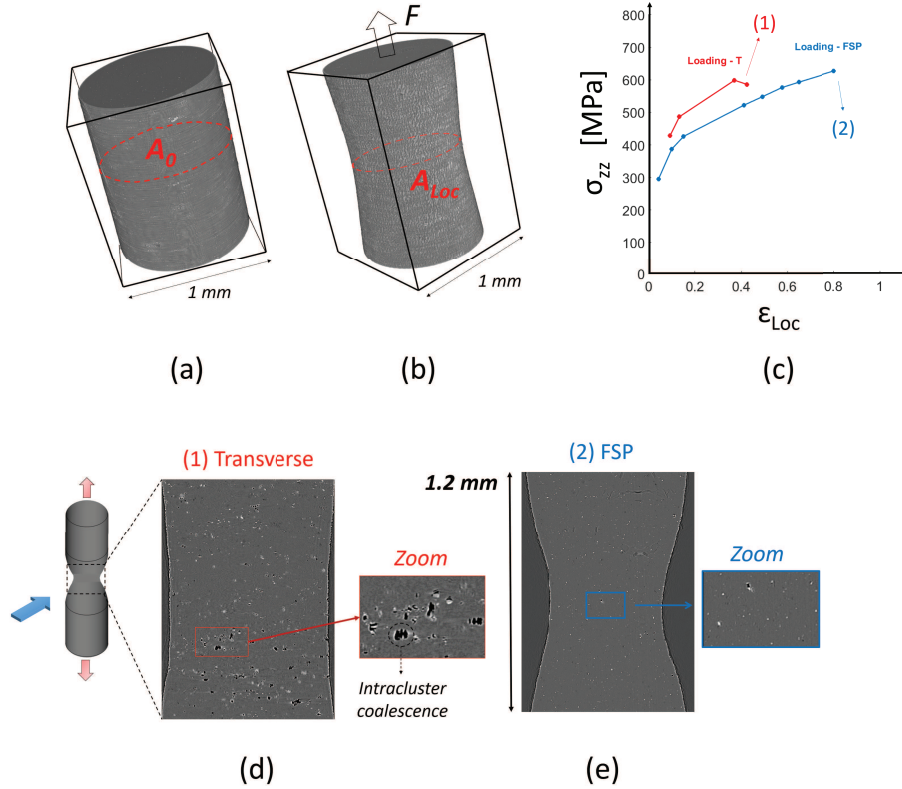


FIGURE 5.23: X-ray microtomography reconstructed images just before fracture, of the tensile test performed on the (d) μ UT-T and (e) μ UT-FSP specimens. The tensile curves in (c) are obtained by dividing the force by the minimum cross-section A_{Loc} of the specimens (see (a) and (b)).

Cracking initiation has been traditionally defined in the past as when a couple of voids have coalesced locally in the most loaded region of a specimen [7]. In the case of an homogeneous particle distribution, such as in the FSPed material, this definition is not too far from what is observed experimentally (see Fig. 5.23(e)). However, it is clear that in the case of particles clustering, it is the distance between clusters which is the relevant length scale.

5.3.5 CT-like sample testing in laminography

5.3.5.1 Visualization of the damage evolution

The reconstructed volume from laminography, see Fig. 5.24, is represented by a 3D array of voxel (i.e. 3D pixel) associated with a gray level. The aluminum matrix is shown in gray, initial porosities in black and intermetallics are white. The damage evolution from one load step to another is visualized by using "2D projection". The 2D projection is performed by keeping only the minimum gray level values (i.e., voids which are black) which are projected along one direction onto one plane, as shown in Fig. 5.24. The projections along the three directions shown in Fig. 5.24 allow thus to fully characterize the 3D damage evolution. The three sub-volumes which have been used for projection are also indicated in Fig. 5.24. At this point, it is important to note that the initial notch of the μ CT specimens were not reduced with razor blades. The notch tip radii (about 0.15mm) is thus large compared to the crack tip radii of the DENT specimens (approximately 25 μ m, see section 5.2.2). Nevertheless, the μ CT specimens can be used to identify the failure mechanisms which must occur in the vicinity of the crack tip in the macroscopic DENT specimens.

For each loading step, the notch opening displacement (NOD) and the force are measured (see Fig. 5.25(a)). The evolution of the force with the NOD is shown in Fig. 5.25(b) for both μ CT-T and μ CT-FSP specimens.

Fig. 5.26 and Fig. 5.27 shows 2D projections of the laminography data obtained for the μ CT-T specimen. The voids are in black and the matrix is in gray. It should be noted that the intrinsic void content of the matrix is exaggerated by the projection of a 3D thick slice of the material onto a 2D plane. First, it is clear that a lot of voids are observed in the vicinity of the notch, even in the first loading steps. Furthermore, it is clear that the voids are preferentially aligned along the rolling direction. This is due to the fact that particle clusters (not visible in these projections) appear under the shape of continuous elongated bands. Since void nucleation is due to the fracture of these particles, the void configuration exhibits the same type of "banded configuration". It is particularly interesting to focus on the "type 2" projections for the

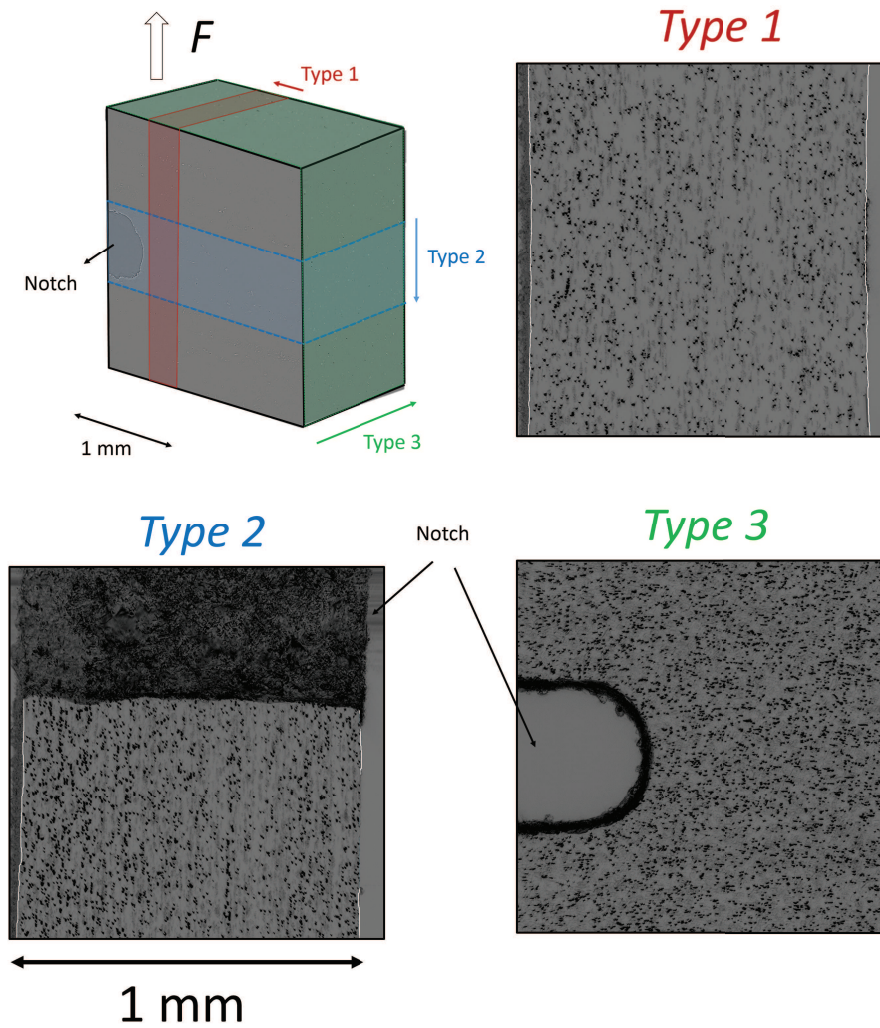


FIGURE 5.24: (a) Reconstructed volume from laminography. The 2D projections ((b), (c), (d)) along the three directions allow to fully characterize the 3D damage evolution.

loading step (6),(7) and (8). In stage (6), "intracluster" coalescence is observed, i.e. void coalescence occurs between closely spaced void inside a same cluster. However, this intracluster coalescence stage does not correspond to the initiation of crack propagation and a second void

5.3. Results

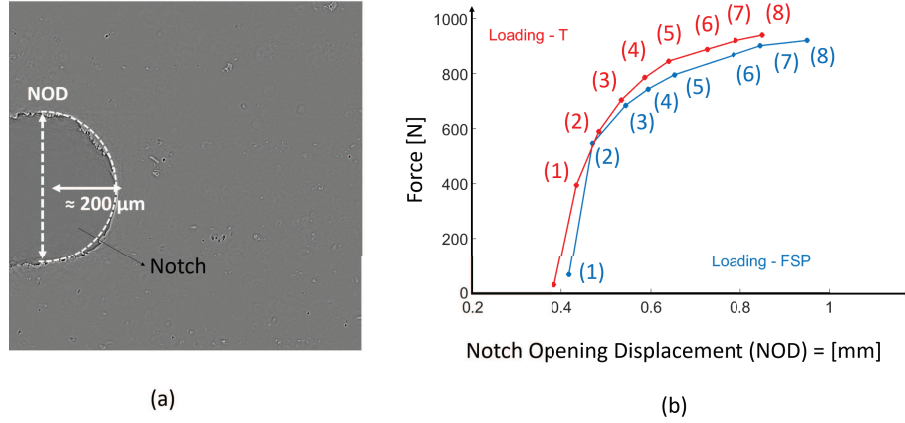


FIGURE 5.25: (a) Notch opening displacement (NOD) measurement and (b) Force-NOD curves for for both μ CT-T and μ CT-FSP specimens.

growth process based on these early coalesced microcracks is observed in stage (7). Finally, coalescence of microcracks in the direction transverse to the crack propagation is observed in stage (8) and the crack starts to propagate. These observations are consistent with the damage percolation process observed in the in situ tensile test (specimen μ UT-T) from which it was concluded that it is the second inter-cluster coalescence which trigger the catastrophic failure of the specimen.

Fig. 5.28 and Fig. 5.29 shows 2D projections of the laminography data obtained for the μ CT-FSP specimen. First, it is observed that, for a similar NOD, significantly less damage is observed in the FSPed material compared to the base material. In the FSPed material, voids are more uniformly dispersed in the matrix and it is only in the last loading stages (stages (5),(6) and (7)) before crack propagation (stage (8)) that voids are significantly observed in the matrix. Again, these observations are in good agreement with in situ tensile testing (specimen μ UT-T) of the FSPed material; voids nucleate at larger strain (and thus larger stress) compared to the base material and a stable void growth is observed. It is only at the end of the damage process that void coalescence is observed, which triggers the crack propagation.

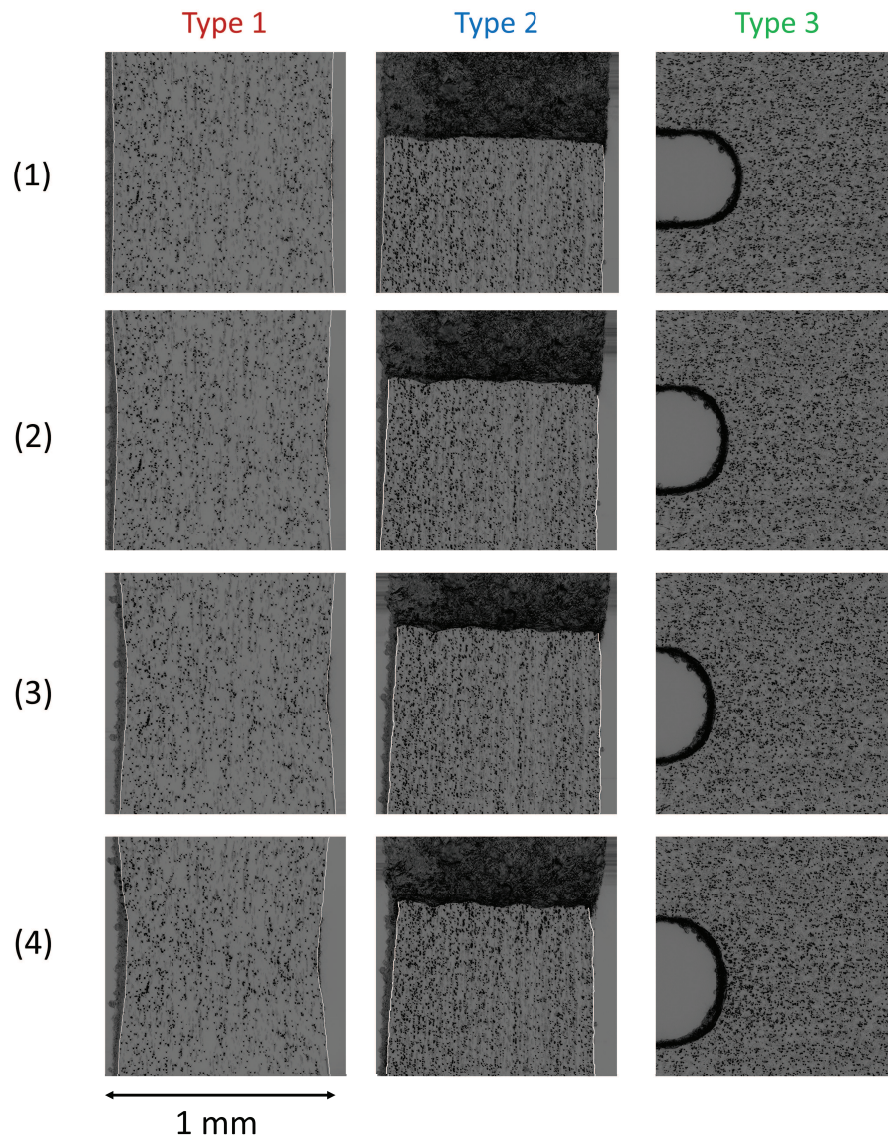


FIGURE 5.26: Damage evolution for the μ CT-T specimen. The loading steps (1), (2), (3), (4) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.

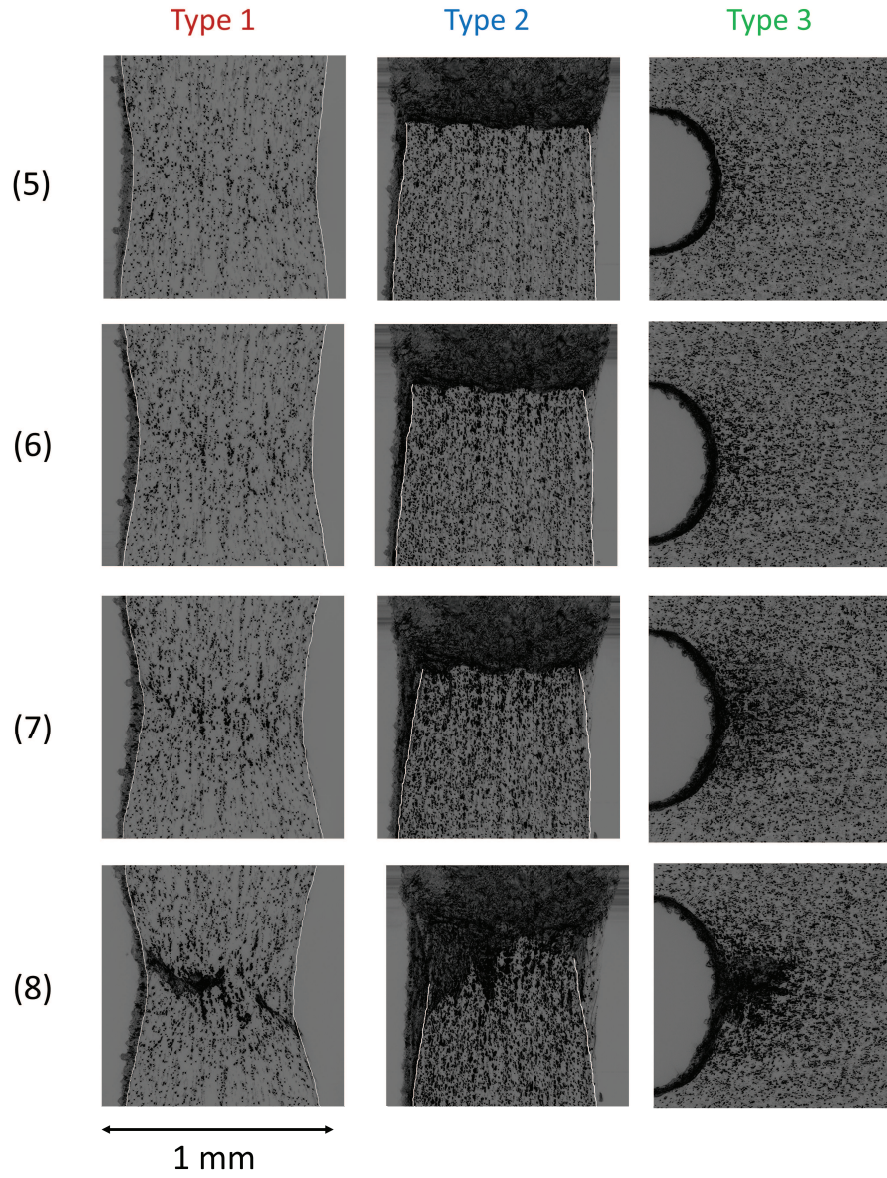


FIGURE 5.27: Damage evolution for the μ CT-T specimen. The loading steps (5), (6), (7), (8) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.

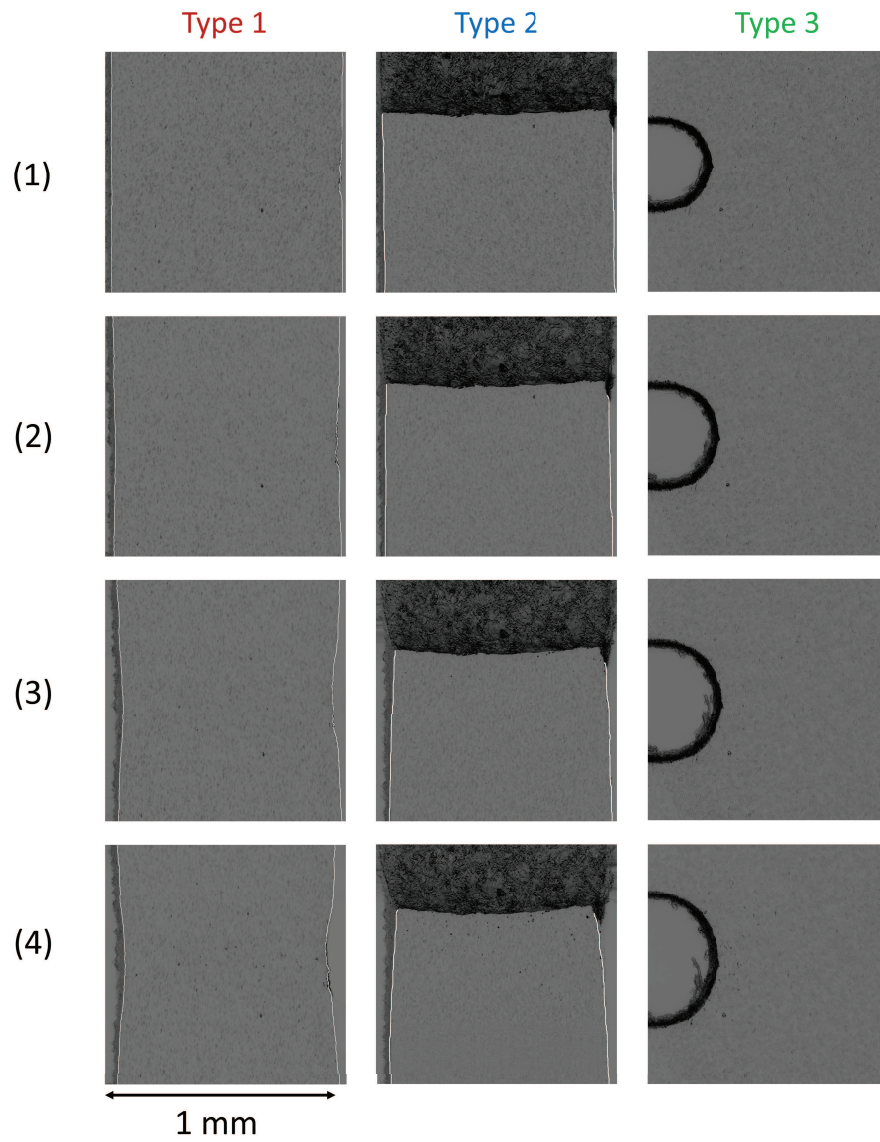


FIGURE 5.28: Damage evolution for the μ CT-FSP specimen. The loading steps (1), (2), (3), (4) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.

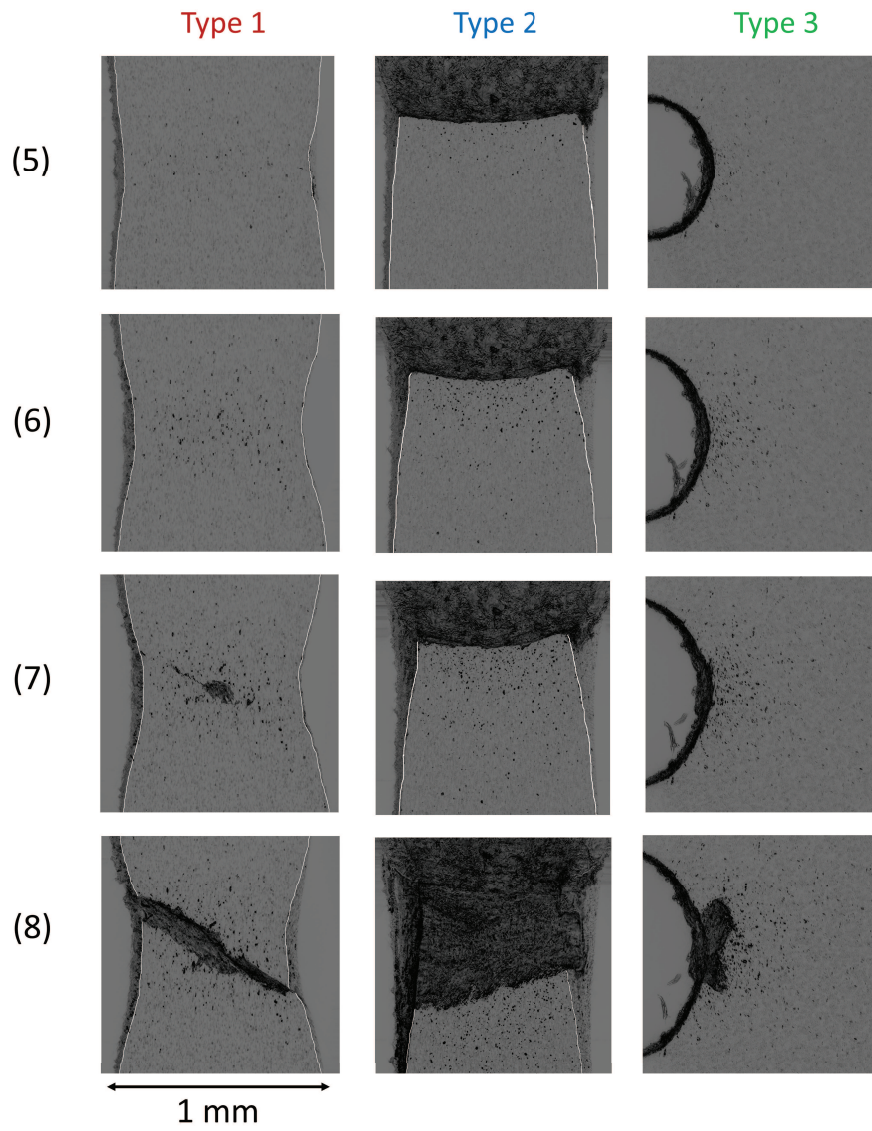


FIGURE 5.29: Damage evolution for the μ CT-FSP specimen. The loading steps (5), (6), (7), (8) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.

5.3.5.2 Fractographic analysis of CT-like specimens

The fracture surface of the two CT-like specimens has been analysed using a SEM microscope. The general observations are very similar with what has been observed on the fracture surface from the DENT-T and DENT-FSP specimens (see section 5.3.2.3). These fracture surfaces correspond obviously to the final stage of the damage evolution described in the previous paragraph.

It is interesting to compare the thickness reduction factor measured from these two fracture surfaces with the thickness of the corresponding CT-specimens observed in situ, just before the initiation of crack propagation. In both the μ CT-T and μ CT-FSP, the thickness reduction observed (in situ) in the last scan before crack propagation is around 0.7. Furthermore, the thickness reduction factor measured (post-mortem) on the fracture surface is also about 0.7 for both specimens. First, note that this value is smaller compared to the macroscopic thickness reduction factor of the DENT-T and DENT-FSP (around 0.85, see section 5.3.2.3). This is not surprising as the thickness and the geometry of these specimens are different. Second, the fact that the thickness reduction factor measured both from in situ and post-mortem images indicates that necking has already fully developed at cracking initiation. Since the sheet thickness of the macroscopic DENT specimens (2.5 mm) is not much larger than the μ CT specimen thickness (1mm), it is thus likely that necking has also fully developed (or has almost fully developed) at cracking initiation in the macroscopic DENT specimen. This explains why, at least in part, the essential work of fracture w_e and the critical J_{Ic} have similar values (around $100 \text{ kJ}/\text{m}^2$ in both cases). Indeed, the EWF method provides a global determination of fracture toughness which does not distinguish between the work needed to initiate the crack and the work needed to propagate the crack. In this case, the resistances to fracture initiation and to fracture propagation are similar because necking has already nearly fully developed at cracking initiation. In other words, w_e constitutes a convenient way for characterising the fracture toughness. This one-to-one relationship between w_e and J_{Ic} has been observed previously [244] but it is not always true [245].

5.3. Results

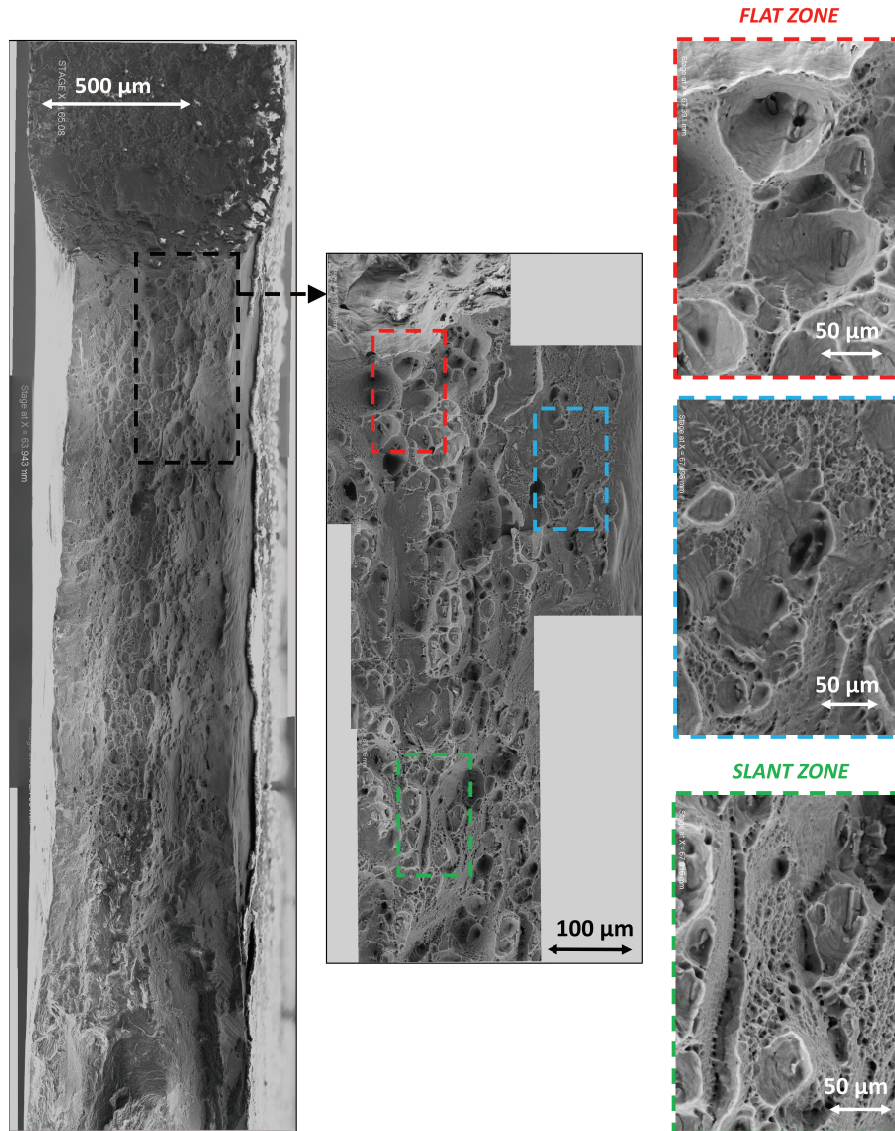


FIGURE 5.30: Fracture surface of the μ CT-T specimen.

5.3.6 Results of the P-DIC procedure

The results of the P-DIC procedure are presented in the following section. Strain fields are displayed together with stress-strain curves or

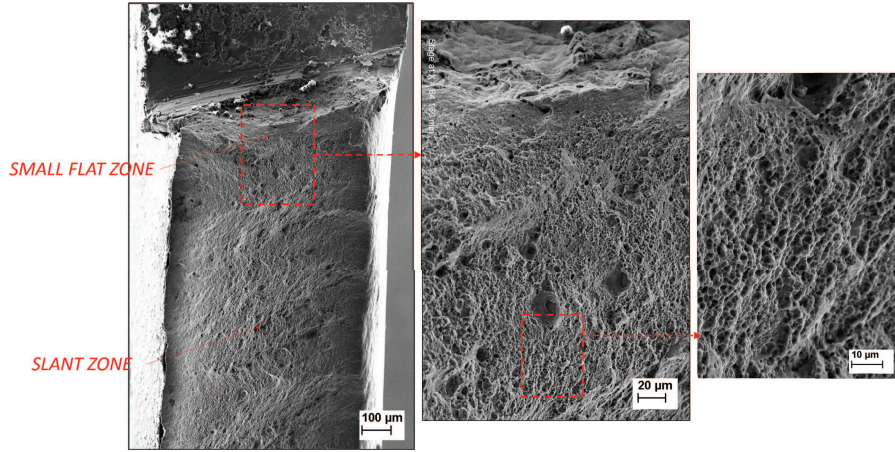


FIGURE 5.31: Fracture surface of the μ CT-FSP specimen.

Force-NOD (notch opening displacement) curves for the tomography or laminography data, respectively. Arrows on these curves are used to indicate that the P-DIC analysis has been performed between a reference state (beginning of the arrow) and a subsequent loading step (end of the arrow). The color bar scale of the strain fields is systematically adjusted to the range of values for the corresponding section. It is always the strain in loading direction (ϵ_{yy}) which is displayed within the strain fields. The strain fields are plotted in a Lagrangian way, i.e. in the reference state. Furthermore, the strain fields are plotted over the *MIN*-projection of the reference state, which contains the initial porosities in black.

5.3.6.1 Tomography

Al 6056 (base material) Fig. 5.32 shows the results of the P-DIC procedure applied on the mini tensile specimen of the Al 6056 (base material). The local variations of the initial porosity (F_V^{local}) corresponding to the reference state is shown in Fig. 5.32(0). Fig. 5.32 shows the strain fields obtained for correlation between the reference state (0) and the loading step (1) and (2). From the first loading step (0)-(1), the field is not uniform but exhibits regions of strain concentration. Furthermore, the shape of the strain field observed in state (2) is quite similar but with cumulation of additional strains compared to state (1).

5.3. Results

In the F_V^{local} map, high values of F_V^{local} indicate regions of the material in which initial porosities are concentrated. A correlation between the zones of strain concentration (i.e. softer zones) and regions with high local F_V^{local} would explain the softening of the material by the presence of these initial voids. Dashed lines have been drawn in state (0) and (1) for easier comparison. Strain concentration zones seem effectively to correlate with some zones of higher F_V^{local} . However, multiple zones of high F_V^{local} are also found outside of these strain localization regions.

The strain field obtained for the final loading step (3), which corresponds almost to the failure of the specimen, has been plotted in an Eulerian perspective in order to be compared easily with the F_V^{local} map of the same state. Again, the zone of high strain values has been encircled (in white) in both representations for easier comparison. Almost all the high porosity regions (in the final state, not initial porosities in this case) are located inside the large encircled soft zone. Since strain concentration zones are indeed expected to be more damaged, this is a first hint that the P-DIC analysis is valid.

In summary, it is observed that :

- There is, at least in part, a correlation between the zones of strain concentration and regions which contain a higher initial porosity (F_V^{local}).
- The location of the strain concentration are similar for all loading steps ((1),(2) and (3)).
- In the last loading step before failure (3), the damage distribution is concentrated within this soft zone.

It is thus concluded that the initial strain map, and thus the initial porosity distribution, contains , at least to some degree, the final fracture path.

As-FSPed Al 6056 Fig. 5.33 shows the results of the P-DIC procedure applied on the mini tensile specimen of the FSPed material. In this case, the reference state (1) is not the initial and unloaded material. Indeed,

the specimen had to be shifted (during in situ testing) because necking was occurring outside of the field of view. Furthermore, the void volume fraction in the reference state (1) is still very limited. The correlation procedure has been applied between state (1) and state (2). In this case, there is only a large and central zone of strain concentration due to the smooth evolution of a diffuse neck. Correlation has also been performed for the other intermediate loading steps between state (1) and state (2). The strain fields (not shown here) display the same shape. The state of damage is shown for the loading step (4) close to specimen failure. Voids are quite homogeneously distributed within the diffuse neck.

5.3.6.2 Laminography

Al 6056 (base material) Fig. 5.34 shows the results of the P-DIC procedure for two different slices of the reconstructed volume. The first projection is positioned relatively close from the notch root ($d_{notch} \approx 300 \mu\text{m}$) and the second projection is positioned far from the notch ($d_{notch} \approx 1 \text{ mm}$). It can be safely assumed that the material "far" from the notch is in plane strain condition, as discussed in section 5.2.6 and Fig. 5.5. However, for the projection positioned relatively close from the notch root ($\approx 300 \mu\text{m}$), results should be viewed with caution as it corresponds to the transition zone towards plane strain condition. The reference state (0) is not the initial and unloaded material (due to difficulty in achieving correlation from this state). The F_V^{local} map is thus composed of two contribution in this case; the initial porosity and early void nucleation.

Strain field close to the notch

The strain fields for load step (0)-(1) and (0)-(2) are shown in Fig. 5.34. First, it can be observed that the shape of both strain fields is very similar. This suggests that the P-DIC procedure is valid despite the relative proximity to the notch root. Furthermore, similarly with what has been observed in tomography (see section 5.3.6.1), the strain fields are not uniform but exhibit regions of strain concentration. Finally, the projection of the final loading step (3), i.e. approximately at the initiation of crack propagation leading to final failure, shows a good

correspondence between the final crack path and the configuration of these "soft zones".

These results can now be compared with the fractography of the specimen after final failure (see Fig. 5.30 in section 5.3.5.2). Close from the initial notch ($\approx 300 \mu\text{m}$), the fracture surface is flat in the central part of the specimen with shear lips on the border of the specimen (see Fig. 5.30). This is in good agreement with the crack path in state (3). Furthermore, the large dimples observed in this area are explained by the "soft" zones which are more damaged and create a preferred path for void coalescence.

Strain field far from the notch

Fig. 5.34 shows, again, that the shape of the strain field is very similar for state (1) and state (2). However, in this case, the strain localize within a slant band which is already visible at the beginning of the deformation process. This is in good agreement with the study of Buljac *et al.* [236, 238] which showed that the plane strain condition is accompanied with the existence of slant strained bands.

The fractography of the specimen (see Fig. 5.30 in section 5.3.5.2) shows that far from the initial notch ($\approx 300 \mu\text{m}$), the fracture surface is slant and covered by small voids. This compares well with the damage projection in the loading step (3) which shows a limited amount of large voids. This suggest that the sudden creation of a slanted crack requires little energy to be added to the system by loading [16] and is due to the nucleation of a second population of voids.

As-FSPed Al 6056 Fig. 5.35 shows the results of the P-DIC procedure applied on the CT-like specimen of the FSPed material. Correlation has been performed between the state (0) and the loading stage (1) and between state (1) and state (2). The strain field for the loading step (0)-(1) is shown in state (1). For the loading step (0)-(1), the incremental strain field shows a large and central zone of limited strain concentration due to the necking of the specimen. The incremental strain field of the following loading step (1)-(2) contains a slant band with significant strain concentration. However, the damage is still very limited at this stage, even within the strain localization band. In the last loading step (3),

the distribution of the porosity is then highly concentrated within the strain localization band.

The fractography of the specimen (see Fig. 5.31 in section 5.3.5.2) shows that the fracture surface is slant and covered by small voids. Again, this suggest that the local stress reached within the strain localization induce the nucleation of a second population of voids.

5.3. Results

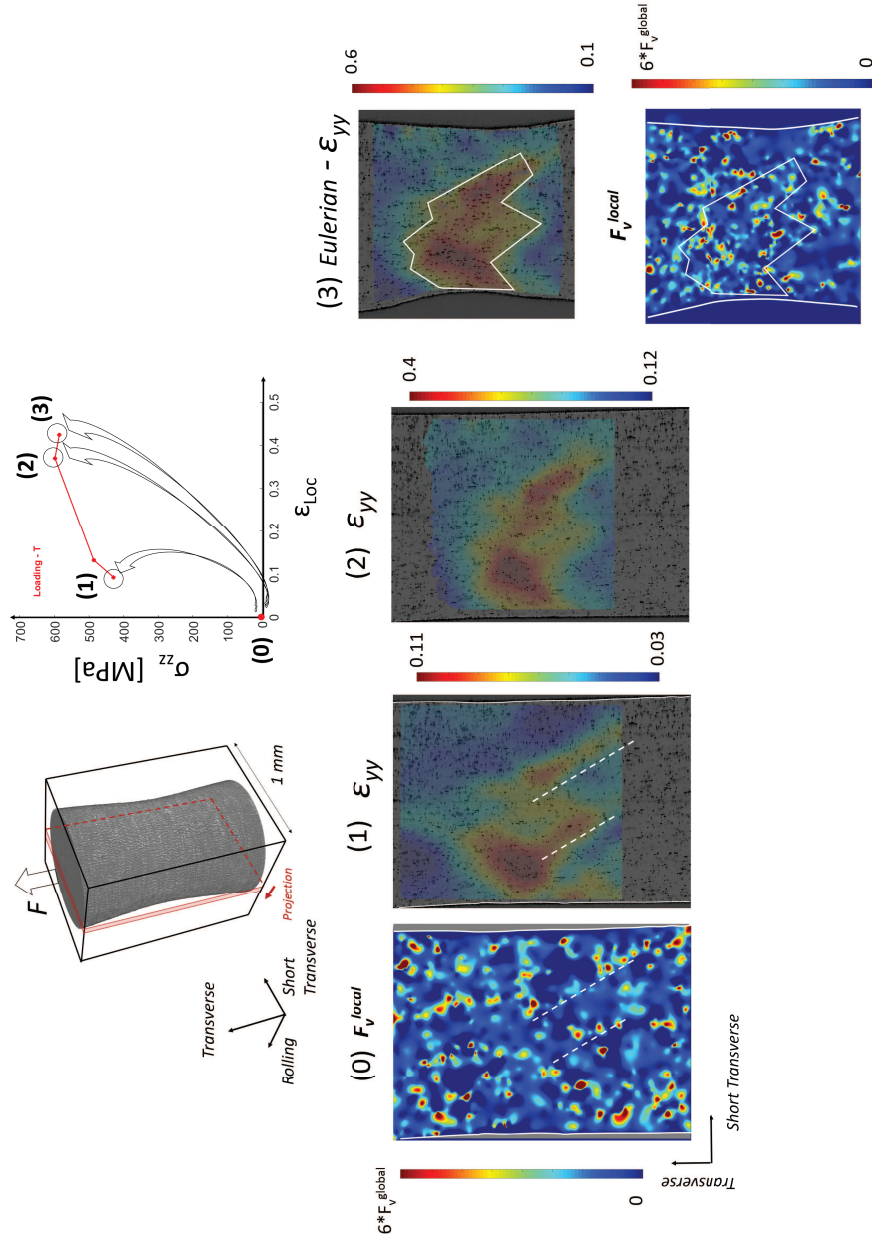


FIGURE 5.32: (Top) Reconstructed volume of the μ UT-T specimen with the position of the projected sections used in P-DIC procedure and stress-strain curve. The arrows indicate the correlation steps which have been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the correlation steps.

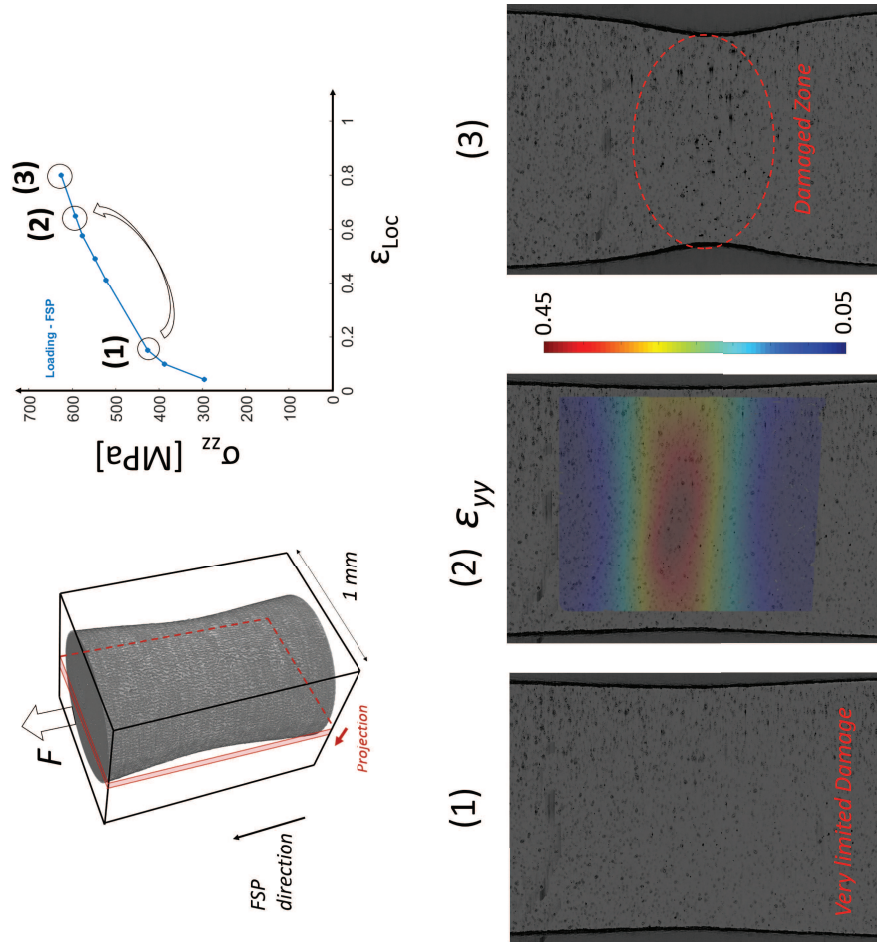


FIGURE 5.33: (Top) Reconstructed volume of the μ UT-FSP specimen with the position of the projected sections used in P-DIC procedure and stress-strain curve. The arrow indicates the correlation step which has been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the loading steps.

5.3. Results

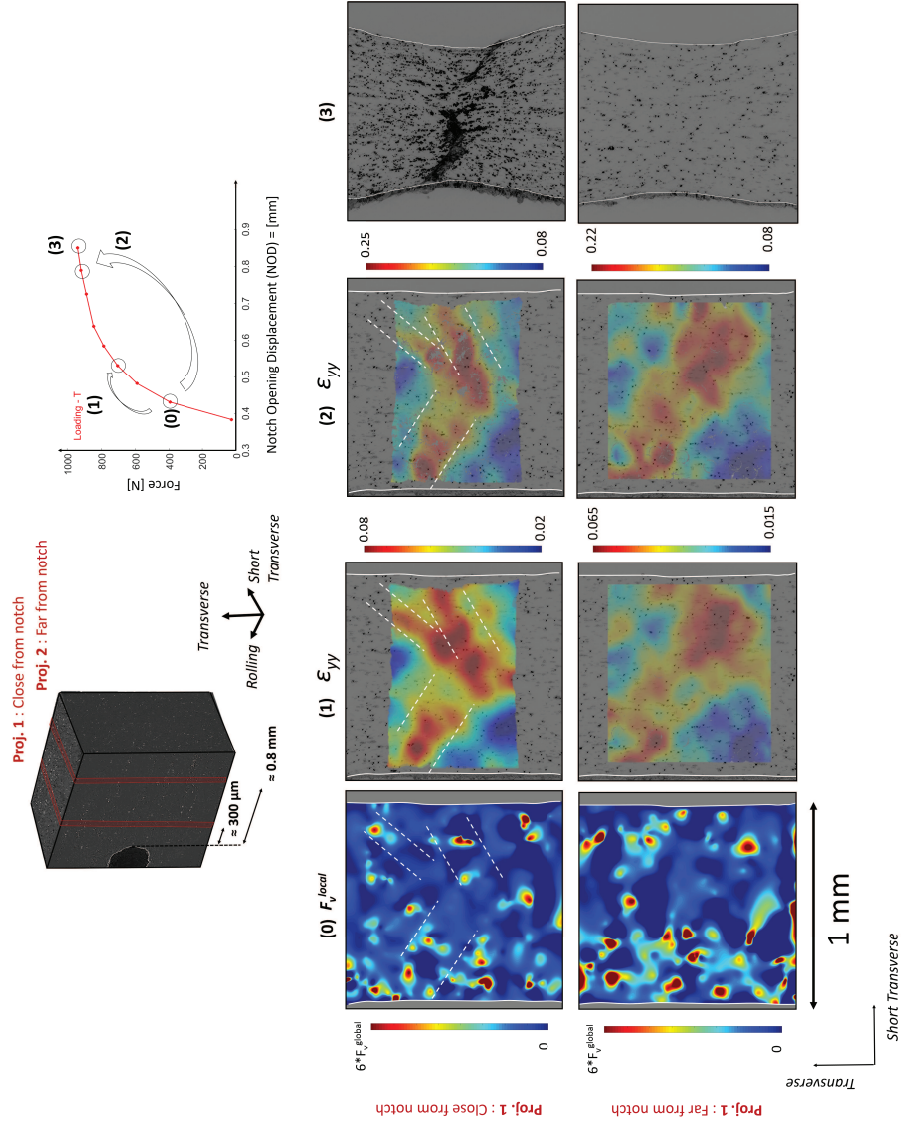


FIGURE 5.34: (Top) Reconstructed volume of the $\mu\text{CT-T}$ specimen with the positions of the two projected slices used in P-DIC procedure and Force-NOD curve. The arrows indicate the correlation steps which have been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the correlation steps.

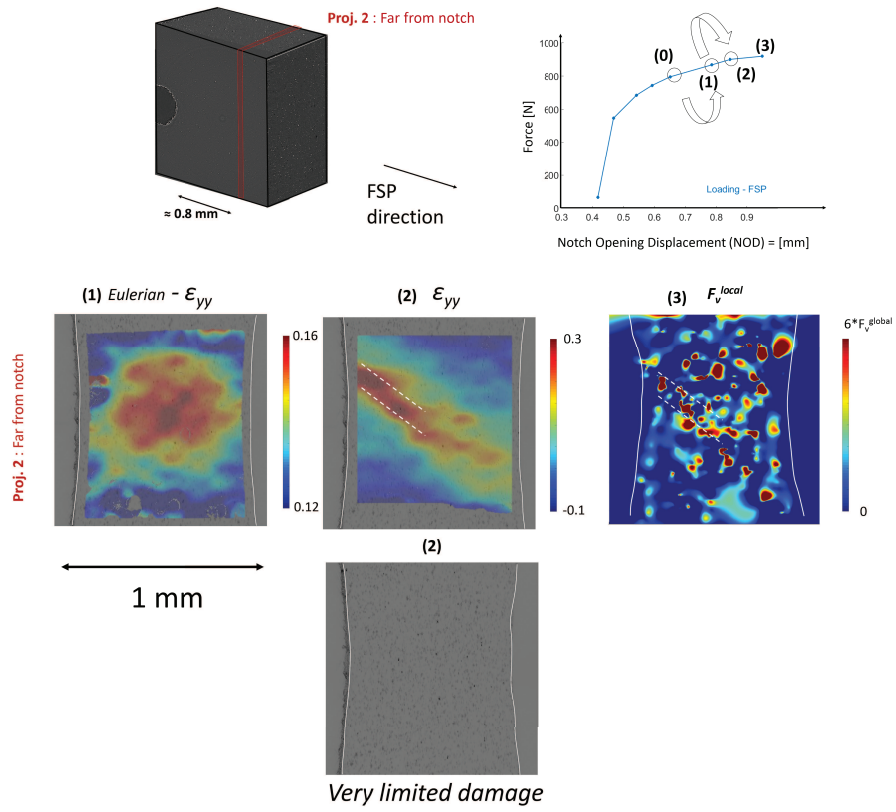


FIGURE 5.35: (Top) Reconstructed volume of the μCT -FSP specimen with the position of the projected slice used in P-DIC procedure and Force-NOD curve. The arrows indicate the correlation steps which have been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the correlation steps.

5.4 Discussion

The major experimental results of this chapter may be summarized as follow :

- The ductility of the FSPed material is significantly improved compare to the base material. This was already explained in chapter 3 and has been confirmed with in situ tensile tests in this chapter.
- The essential work of fracture w_e is roughly equal to $100 \text{ kJ}/m^2$ for all the conditions. The fracture toughness (characterized by w_e) is thus relatively independent of the loading direction. Furthermore, the fracture toughness is not significantly modified by FSP.
- The degree of necking, characterized by the thickness reduction factor of the DENT specimens, is similar for all conditions with an averaged value between 80 and 85%.
- The essential work of fracture w_e is related to the J_{Ic} parameter. This is due to the fact that necking is fully developed when the crack starts to propagate, as observed in situ in the μ CT-like specimens.
- In the Al 6056, particles are concentrated into clusters. It is the distance between particle clusters which is the relevant fracture length scale. Indeed, final fracture is triggered by the collapse of the matrix ligament between microcracks formed by the intra-cluster coalescence stage. On the opposite, in the FSPed material, the degree of particle clustering is strongly reduced and it is the mean particle spacing which is the relevant fracture length scale.
- In all the DENT specimens, the final failure is characterized by the sudden creation of a slanted crack. The high stresses reached within this strain localization band induce the nucleation of a second population of voids. In the slant region, primary void growth is thus limited by the occurrence of the void sheeting mechanism.

All these experimental facts can be rationalized based on Fig. 5.36.

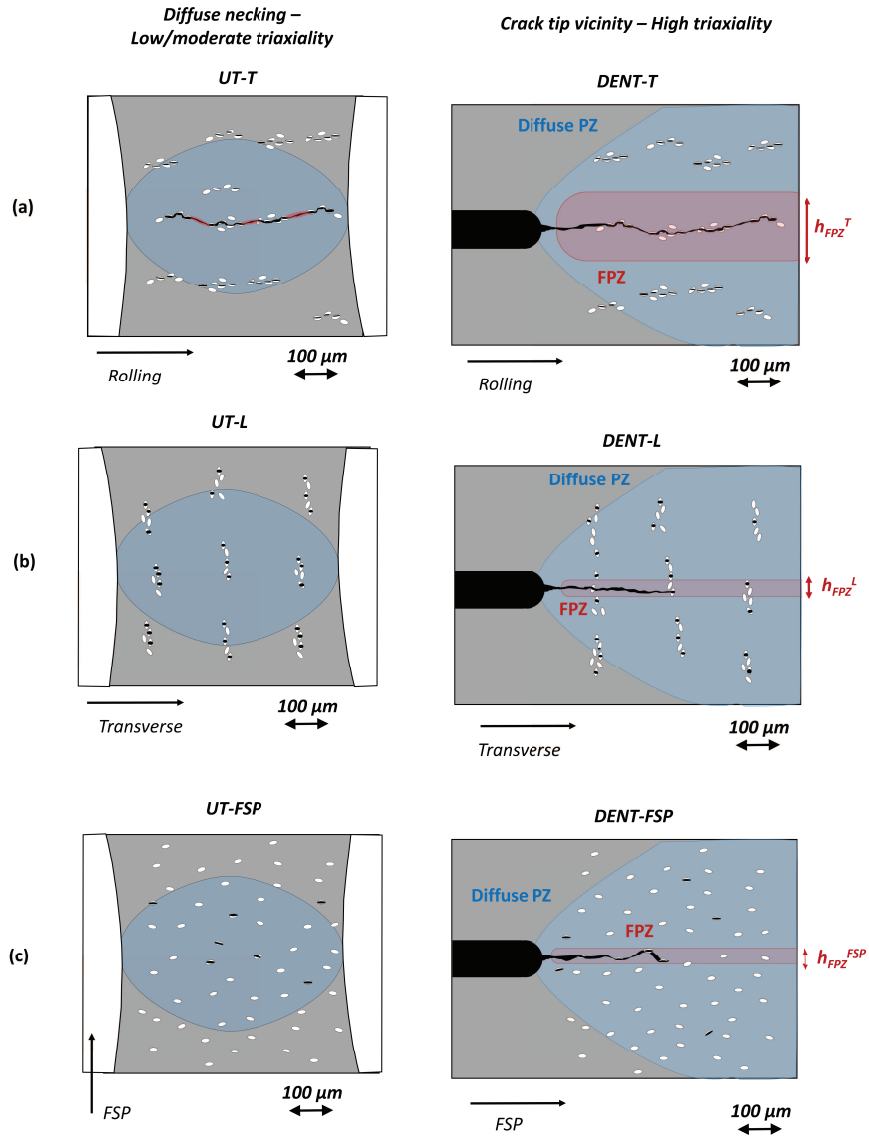


FIGURE 5.36: Schematics of the damage sequence in : (Left) Uniaxial tensile tests specimens and (Right) DENT specimens.

Low to moderate triaxiality : In uniaxial tension, void nucleation, growth and coalescence occur within a large plastic zone due to the diffuse necking of the specimen. In the base material, particle clusters

appear under the shape of continuous elongated bands. The ductility of the material exhibits a significant anisotropy due to the easy percolation path when loading is along the transverse direction. On the other hand, when loading is along the rolling direction, the presence of tougher matrix between these clusters increases the fracture strain of the material.

In the base material, there is, at least in part, a correlation between the zones of strain concentration and regions which contain a higher initial porosity. Indeed, a high volume fraction of initial porosity introduce a local softening effect which can explain the local strain localization. Furthermore, several studies have reported that initial porosities (which are probably hydrogen micropores) initiate preferentially on intermetallic particles during the homogeneization process [246]. This means that regions of high initial porosity correspond to particle clusters. Fig. 5.37 shows a 3D microtomography perspective of particles (blue) and initial porosities (red) distribution in the Al 6056. It appears indeed that initial porosities are preferentially located within particle clusters. Higher void nucleation is associated with particle clusters and this also contributes to the strain localization within these "soft zones".

In the FSPed material, FSP breaks the large intermetallic particles into smaller, and thus stronger, fragments, closes the pre-existing porosity and randomizes the particle distribution. The fracture strain is thus significantly improved. Furthermore, in the FSPed material, the particle distribution is homogenized and the fracture strain is isotropic.

At low triaxiality, diffuse necking is observed and the strain concentration due to this localization phenomenon remains limited. The large stresses required to initiate a second population of voids are not reached. The ductility depends mainly on the primary population of voids, i.e. on the intermetallic particles, and is significantly improved by FSP.

High triaxiality :

In fracture of thin specimens (2.5mm), the development of localized necking zone is systematically observed in the near-crack-tip region, as schematically shown in Fig. 5.36. This localized necking region is considered to be the fracture process zone (FPZ). The overall energy absorption of thin plates (w_e) results from plastic energy spent in the

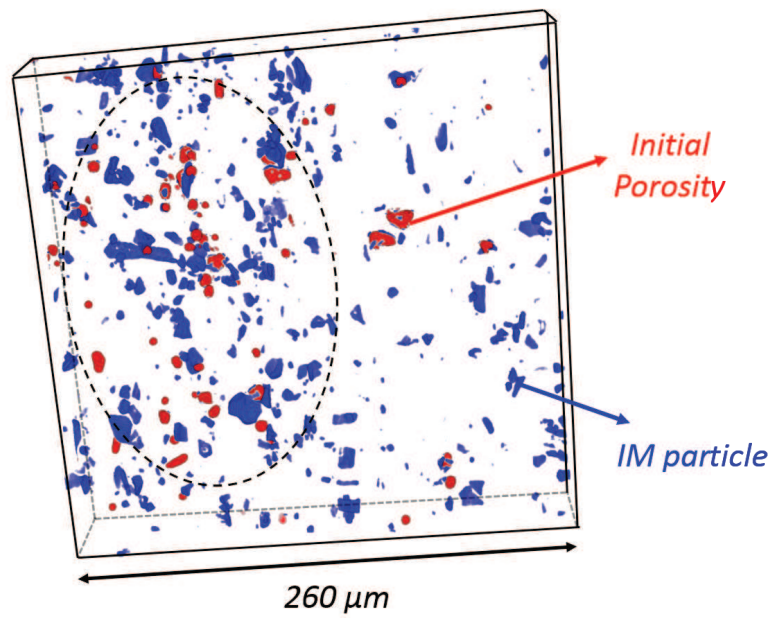


FIGURE 5.37: 3D microtomography perspective of particles (blue) and initial porosities (red) distribution in the Al 6056. Microtomography data comes from chapter 2 with a resolution of 325 nm.

5.4. Discussion

neck (w_n) and from the energy spent for damaging the material (w_0) in the fracture process zone [245]. In all fracture specimens (DENT-T, DENT-L and DENT-FSP), void growth is limited by the occurrence of the void sheeting mechanism in the slant zone. The primary void growth is postponed by FSP when compared to the base material, as observed with the in situ μ CT specimens. However, the sudden creation of a slanted crack, due to the nucleation of a second population, requires little energy to be added to the system [16]. This slant transition swamps the potential beneficial effect of a lower primary void growth. The energy absorption (toughness) is thus limited by the nucleation of the second population and is not improved by FSP, while the ductility is increased because it is controlled by the primary void growth.

Furthermore, the height of the FPZ (h_{FPZ}) is determined by the microstructure of the material [227] and this could also explain why the toughness is not lower for the DENT-T specimens. In the case of the Al 6056, it is the clusters configuration which sets the height of the FPZ. Particle clusters appear under the shape of continuous elongated bands. In the case of the DENT-T specimens, the height of the FPZ (h_{FPZ}^T) corresponds thus to the large distance between clusters along the transverse direction. On the other hand, in the DENT-L specimen, it is the distance between voids inside a cluster along the rolling direction which sets the FPZ height (h_{FPZ}^L). In the FSPed material, the particle distribution has been homogenized and the height of the FPZ (h_{FPZ}^{FSP}) corresponds to the mean distance between voids in the vicinity of the crack tip. The FPZ size is thus larger for the DENT-T configuration compared to DENT-L and DENT-FSP configurations. This could also explain why, despite a significantly lower ductility observed for the UT-T configuration, the fracture toughness of the DENT-T configuration is similar to the fracture toughness of the DENT-L and DENT-FSP configurations.

Finally, the results of the P-DIC procedure on the micro-tensile specimens indicate that the strain field exhibits regions of strain concentration in the base material (see Fig. 5.32) while it is very homogeneous in the FSPed material (Fig. 5.33). The average grain size in the base material is approximately equal to 100 μ m and is reduced to 10 μ m after FSP, see section 3.6.3. Since the mini tensile specimens have a diameter equal to 1mm, the number of grains along the thickness di-

reaction is much larger for the FSPed material (≈ 100 grains) compared to the base material (≈ 10 grains). This might thus also explain why the strain field observed in the base material is heterogeneous compared to the FSPed material.

Conclusions and perspectives

The main objective of this work was the 3D characterization and quantification of the microstructural heterogeneity effects on damage evolution of 6xxx aluminium alloy. A cellular automaton model has been developed to predict fracture strain using only the flow properties and knowledge of its particle distribution. The 3D damage model enables to uncouple the effect of particle size, particle spatial distribution and initial porosities. Furthermore, a quantitative approach has been proposed to relate the propensity to fracture anisotropy to a simple microscopic parameter characterizing the degree of anisotropy in the spatial distribution of second phase particles. Based on these results, friction stir processing (FSP) has been performed on a 6056 rolled Al alloy in order to improve the microstructure and the ductility. Furthermore, it was shown that FSP could potentially be used to locally enhance the formability of aluminium sheet where forming involves large strains. Finally, the microstructural heterogeneity effects on cracking initiation in thin plates has been performed, at the macro and the microscale. The most important findings are summarized below. Remaining questions and perspectives are discussed thereafter.

Major findings

The major conclusions regarding the microstructural heterogeneity effects on the ductility (i.e. the fracture strain) of 6xxx aluminium alloy are :

- The fracture strain is primarily dictated by the effect of the second phase particle size distribution with the alloy exhibiting the smallest particles being the most ductile.
- The effect of particle spatial distribution plays also a first-order effect on controlling the ductility. It is the degree of continuity of the particle stringers over large distances, i.e. large-scale clustering, which explain the detrimental effect of particle clustering. Furthermore, if particle clusters appear under the shape of continuous elongated bands, the ductility of the material is likely to exhibit a significant anisotropy. In the case of shorter and isolated particle stringers, the presence of tougher matrix between these clusters strongly reduces the anisotropy of fracture properties by allowing a second stage of stable void growth at the scale of the clusters.
- The presence of pre-existing voids has also a first order effect on the predicted fracture strains.

Friction stir processing positively impacts these three causes of ductility loss :

- FSP breaks the large intermetallic particles into smaller, and thus stronger, fragments.
- FSP closes the pre-existing porosity.
- FSP randomizes the particle distribution and reduces the degree of clustering.

Finally, the major conclusions regarding the microstructural heterogeneity effects on the toughness of 6xxx aluminium alloy are :

- For all fracture tests, the final crack starts to propagate in a flat mode, then tilts and continues to propagate in a slant manner.

Conclusions and perspectives

- In the slant fracture region, a bulk-DIC analysis has been used and strained bands appear before any significant damage development in this region.
- Void nucleation and growth of a second population of particles occur in this band of high level of strain. These small void sheets act as a rapid coalescence mechanism.
- A similar toughness has been observed for a very heterogeneous (Al 6056) and a very homogeneous (Al 6056 after FSP) intermetallic particle spatial distribution. Furthermore, the toughness of the Al 6056 is quite isotropic. This behaviour is due to the second population of voids. The growth of the large primary voids is limited by the occurrence of the void sheeting mechanism.

Perspectives

Several elements studied in this work should be further investigated :

- The nucleation of a second population of voids has been shown to have a major influence on toughness. More work could thus be done on expressing an adequate condition for void nucleation by decohesion at these small particles.
- The scanning resolution of the laminography results was $0.65\mu\text{m}$. A good characterization of the large intermetallic particles (typically larger than $1\mu\text{m}$) is thus possible. However, it is not been possible to characterize the effect of dispersoids (typically ranging between 0.02 and $0.5\mu\text{m}$) on the damage evolution with this resolution. First, but significant, steps toward this achievement have been made recently [106]. In-situ laminography has been performed at beamline ID 16b at ESRF, Grenoble. Scans with intermediate resolution (240 nm) and scans with high resolution (100 nm) were obtained during the experiment. Thanks to this technique, the voids and intermetallic particles of nanometer size are more easily distinguished from their surroundings. This new technique allows to study the evolution and the interaction be-

Conclusions and perspectives

tween microstructure, damage and plasticity at nanometer resolution. With this resolution, the characterization of voids, inter-metallic particles and dispersoids can be achieved. This brings new possibilities to study the effect of a second population of voids and how this second population limits the primary void growth.

- The apparent similar toughness observed for the Al 6056 and for the FSPed material has been linked with the nucleation and growth of a second population of voids within the slant zone. More advanced fracture specimens could be used in order to eliminate this flat-to-slant transition and measure the resistance of the material to flat, ductile fracture.
- The tool for tracking the cavities which has been used in this work could be coupled with the new Projection-DIC (or with DVC) procedure. Indeed, tracking allows the quantitative study of the evolution of a cavity, from nucleation to coalescence. On the other hand, the P-DIC (or DVC) procedure allows the quantitative study of the strain field around the voids. Such results could help for the understanding and the modelling of the interaction between the damage and the deformation process.
- Model materials, whose microstructure is usually enlarged and controlled as much as possible, represent a simplified microstructure of engineering materials. Such model materials could be used, for example, to facilitate the quantitative study of the effect of void clustering on the damage evolution. Furthermore, model material could be used to elaborate fracture mechanism maps for metals and alloy. These maps would show how the fracture mechanisms changes with some microstructural parameters and flow properties. One example which has already been treated in this work is the link between the degree of particles clustering and the fracture anisotropy.

List of Figures

1	Evolution of the number of publications involving (1) ductile damage and (2) microstructural heterogeneity effects on ductile damage and related to materials science applications (according to Elsevier Scopus, www.scopus.com , consulted in July 2017).	12
2	Structure of the thesis, as detailed in the text.	14
1.1	The five generic scenarios of ductile fracture in single phase polycrystals at temperatures far below melting point temperature. Schematics adapted from [14]. Experimental figures (a), (b), (d), (e) are reproduce from Refs. [15],[16],[18],[17], respectively. More details are given in the text.	18
1.2	Schematic description of the microstructural features and length scales entering a generic damage process. . .	20
1.3	Effect of the particle shape on the different void nucleation criteria. For k_C , the points correspond to unit-cell computation results and the dashed line has been fitted ($k_C = 0.061W_p^2 + 0.72W_p - 0.067$).	25
1.4	Fig.(b) shows the evolution of the stress concentration factor k_C . The elasto-plastic properties which have been used for the matrix are shown in Fig.(a)	26
1.5	Principal stress distribution inside and at the interface of a particle, normalised by their respective fracture criteria, just before nucleation. Adapted from [41].	27
1.6	Comparison between the stress concentration factor obtained by the Beremin model (k_b') and obtained by the Lee and Mear analysis (κ_P).	29

List of Figures

1.7	Review of the critical stress found in the literature for void nucleation by particle fracture. Points 1 to 13 come, respectively, from Refs.[52, 53, 34, 43, 54, 50, 55, 46, 56, 57, 58, 59, 60]	32
1.8	Effect of the particle spatial distribution on void nucleation by interfacial debonding. Adapted from [62].	33
1.9	Effect of the particle spatial distribution on void nucleation by particle fracture. Adapted from [63]	34
1.10	Evolution of the (a) void volume fraction and of (b) the void aspect ratio for different stress triaxialities and for initial void volume fraction $f_0 = 10^{-8}$ and cell aspect ratio = 1. The plain line indicate the unit cell results of Tvergaard (Redrawn from [75]) and is compared with the evolution predicted by the R&T model (dotted line, see eq. 1.18).	38
1.11	Unit cell results for the evolution of an initially penny-shaped crack (blue, green line) from Ref. [75]. The black line shows the void shape evolution given in eq. (1.19).	39
1.12	Left : FE simulations [77] of the growth of a spherical void (dotted line) and of a void nucleated from a spherical rigid particle with no cohesion with the matrix (plain line) for $T=0, 1/3$ and $2/3$. Right : Experimental observation of void locking by a particle inside a void a low (from [79]), intermediate (from [79]) and high triaxiality (from [80]).	40
1.13	FE simulations of the growth of a spherical void and of a void nucleated from a spherical rigid particle with no cohesion with the matrix, for $T=1/3$. Adapted from [78].	41
1.14	Effect of particle fragments on the growth of initially penny-shaped voids : (a) unit cell results of Tvergaard (redrawn from [75]) compared with the evolution predicted by the (c) R&T model. The unit cell used with/without the particle fragments is shown in (b). The initial void aspect ratio W_{void}^0 is 0.091 and the particle aspect ratio W_p is 5. In both (a) and (c), plain/dotted line corresponds to void growth with/without void locking effect. Figure (d) shows void locking observed by X-ray tomography (reproduced from [81]).	42

1.15	Schematic description of the competition between the two modes of plasticity. In the early stages of deformation, the growth is stable. At some point, the deformation localized within the void ligament. Reproduced from [24].	45
1.16	(a) Effect of the voids configuration (W_{void} and χ) on the plastic constraint factor C_f . (b) Unit cells used by Thomason [85, 86] and by Benzerga [25].	46
1.17	(a) Deformation sequence of a laser drilled model material made of an Al 5052 plate (with thickness around 100 μm) containing a random array of holes (with diameter around 10 μm). Reproduced from [89]. (b) A red line indicates location where void coalescence is likely to occur first, according to the coalescence indicator I_{coal}	49
1.18	(b, c, d) Deformation sequence for a random multivoid cell. Reproduced from [90]. (a) A red line indicates location where void coalescence is likely to occur first, according to the coalescence indicator I_{coal}	50
1.19	(a), (b) Comparison between the HC-criteria (with randomly picked parameter $a_{HC}=1, c_{HF}=0.2, \sigma_{coh}=2.7\sigma_0$) and the void nucleation, growth and coalescence approach in plane stress condition. (c) Evolution of T_{crit} with the volume fraction of void at nucleation f_0 for a penny-shaped void ($W_0=0.01$) and spheroidal void ($W_0=1$).	53
1.20	Schematics illustrating (a) cone-beam and (b) parallel-beam projection systems. Reproduced from [102].	58
1.21	Evolution of the number of publications involving X-ray tomography related to materials science applications (according to Elsevier Scopus, www.scopus.com , consulted in July 2017).	59
1.22	Tomography and laminography experimental setup. Reproduced from [106].	60
1.23	The black sphere is obviously on top of the blue ellipsoid. However, a view from another angle shows that the blue object is actually not an ellipsoid.	61
1.24	Example of mental interpretation of structures and objects. Reproduced from [99].	62

List of Figures

1.25	Toughness tree representation of microstructural features that affect toughness in age-hardenable aluminium alloys [115].	66
1.26	A schematic drawing of friction stir processing. Courtesy of N.J. Mena.	69
1.27	An overview of the evolution of microstructural features and its linkage to various emerging FSP technologies. Reproduced from Ref.[126].	70
2.1	Variation of the different parameters extracted from the uniaxial tensile tests on the three alloys as a function of the yield stress corresponding to the different heat treatments. (a) True fracture strain of three Al alloys; (b) true strain at the onset of necking; (c) dislocation storage rate θ_0 of the Voce law; (d) dynamic recovery parameter β . . .	76
2.2	Schematic representation of the method used to analyse the probability of fracture of intermetallic iron-rich particles at various distances from the fracture surface of broken tensile samples polished down to mid-thickness; for each particle, record position, dimensions and broken or not broken (here a 6056 alloy specimen), micrograph obtained by FEG-SEM. The color map shows the finite element simulation predictions of the tensile test.	79
2.3	(a) Void nucleation on particle. (b) Result of the growth model at different $\varepsilon_{Growth} = \varepsilon_{Loc} - \varepsilon_{Nucleation}$. (c) Definition of the effective parameters used to apply the coalescence criterion. (d) Equivalent geometry. (e) Result of the merging process : minimum ellipsoid that envelops both coalescing voids. (f) Exemple of a whole RVE at a local strain $\varepsilon = 0.25$. (a – f) The particle is in blue, the voids in red and the coalesced voids in grey.	85
2.4	Overview of the modelling strategy	88
2.5	Characterization of intermetallic particles sizes and initial porosity by X-ray microtomography and SEM. (a) 3D microtomography perspective of particles (blue) and pores (red) distribution in all three alloys. Distribution of equivalent diameter for the intermetallic particles; (b) based on the 3D X-ray microtomography images (c) based on the SEM micrographs.	90

2.6	Yield stress σ_0 corresponding to the various heat treatments performed on the three alloys.	91
2.7	Comparison between the experimental and modelled (best identification) fraction of broken particles as a function of the particles equivalent diameter in the three alloys for all heat treatment conditions.	94
2.8	(a) Evolution of the rates of change of the equivalent radius of every void $\frac{R_{eq}}{R_{eq,0}}$ as a function of strain after nucleation; (b) Evolution of the mean rate of change $\frac{R_{eq}}{R_{eq,0}}$ of all voids; (c) Evolution of the mean void aspect ratio W_{void}	95
2.9	(a) Comparison between the variations of the void volume fraction (f) measured during in-situ microtomography and the void volume fraction predicted by the model as a function of the local strain in the necking zone; (b) Zoom in figure (a).	97
2.10	Results of the model applied to the Al 6056 alloy for three heat treatments (namely T6, T4 and overaged with $\sigma_0 \simeq 60$ MPa). Comparaison between : (a) the variations of the void volume fractions; (b) fraction of broken particles extracted from the model; (c) number of coalescence events; (d) total number of voids. All the data are extracted from the model and their evolution are presented as a function of the local strain in the necking zone. The point on each curve corresponds to the predicted fracture strain.	99
2.11	Comparison between the experimental fracture strain and the fracture strain of the model as a function of yield stress. The plain line corresponds to the linear regression of experimental values. The dotted line corresponds to the linear regression of the values from the model.	100
2.12	Sensitivity analysis on different contributions of the cellular automaton model. The "full model" corresponds to the predictions of the model derived in the present paper and used in Fig. 2.10 and Fig. 2.11 for comparison to experimental data. The "modified models" are degraded versions of the model.	103
2.13	Definition of the effective void dimensions used in the growth model.	105

List of Figures

2.14	Variation of the true strain at the onset of necking ε_u extracted from the uniaxial tensile tests on the three alloys as a function of the yield stress corresponding to the different heat treatments. Plain line, dashed line and dotted line correspond to heat treatments which are performed at 180°C, 300°C and 350°C, respectively. Stage <i>a</i> , <i>b</i> and <i>c</i> indicate the evolution with increasing heat treatment time.	106
2.15	Definition of the strain hardening parameters (Θ_0 , β , Θ_{IV}) of the Voce law involving both stages III and IV based on a hardening rate versus flow stress plot. . . .	107
2.16	Variation of (a) Θ_0 and (b) β extracted from the uniaxial tensile tests on the alloy Al 6056 as a function of the yield stress corresponding to the different heat treatments. . .	108
2.17	(a) Stress-strain curves corresponding to points (1), (2) and (3) in Fig. 2.14. (b) and hardening rate versus flow stress plots corresponding to these points.	110
2.18	3D microtomography perspectives of the damage evolution for the Al 6056.	112
2.19	3D microtomography perspectives of the damage evolution for the Al 6005A.	113
2.20	Vizualisation of the RVE used in the automaton cellular and the projection procedure used to compare it with experiments.	115
2.21	Comparison between the damage evolution observed by in situ tomography and the evolution predicted by the model. The matrix is in the overaged state. For the model images, only cavities that have already been involved in a coalescence process are shown (in order to highlight the percolation process).	117
2.22	Comparison between the damage evolution observed by in situ tomography and the evolution predicted by the model. The matrix is in the T6 state. For the model images, only cavities that have already been involved in a coalescence process are shown (in order to highlight the percolation process).	118
2.23	Example of the void tracking procedure. Each cavity which has been tracked is shown with the same color in (a),(b) and (c). A cross-section of the tomography volume is shown in (d).	120

2.24	Vizualisation of the tracking procedure in the Al 6056. Initial voids are shown in red and nucleated voids are shown in grey.	122
3.1	(a) Schematic of the FSP configuration and the geometry of extracted tensile and tomography specimens, (b) micrograph of the cross-section of the weld in a plane perpendicular to the processing direction with visualization of the tensile (red) and tomography (green) specimens location, the SEM micrographs are taken in the RD-TD plane represented by the blue line in the cross-section. (c) Angle between the major axis of the particle and the transverse direction and (d) equivalent diameter of the particles observed on the SEM micrographs.	126
3.2	Schematic of a tensile sample loaded in the RD up to 420 MPa (left figure) and visualization of a sub-volume (right figure) extracted from the total volume scanned by tomography in the base material after automatized particle reconstruction. The unbroken particles are in blue and the fragments corresponding to a fragmented and further reconstructed particle are shown with the other colors (one colour per particle).	129
3.3	3D microtomography perspectives of intermetallic particles distribution for the 6056 base material and for 1, 3 or 6 passes of FSP. White particles correspond to isolated particles, and particles with the same color belong to the same cluster according to the definition given in section 3.1. RD = rolling direction, STD = short transverse direction.	130
3.4	Cumulative particle size distribution (volume-weighted) for the intermetallic particles based on the 3D X-ray microtomography images.	131

List of Figures

3.5	Morphological parameters of the particles for different number of FSP passes: (a) Distribution of the aspect ratio; (b) Equivalent diameter (D_{eq}) as a function of the aspect ratio (W_p); (c) Equivalent diameter (D_{eq}) as a function of the angle between the major axis of the particle and the transverse direction (TD); (d) Aspect ratio as a function of the angle between the major axis of the particle and the transverse direction (TD). BM = base material.	132
3.6	Example in 2D of the (a) segmented particles field, (b) distance map of the labelled particles with the distance visualized by greyscale, and (c) watershed cells and the particles superposed. (d) Visualization of the result of the procedure applied in 3D to a sub-box of the microtomography image of the base material where particles are in blue and cells are in purple.	134
3.7	Distribution of (a) nearest-neighbour distances and (b) nearest-neighbour distances as a function of the number of FSP passes. Distances have been normalized by $\rho^{-1/3}$, with ρ the number of particles per unit volume. The “occurrence” is calculated as the ratio of the population in the class divided by the total number of particles.	136
3.8	(a) Principle (left) and result (right) of the PCF analysis for the base material. On the left figure, intermetallic particles are presented in blue and voids in red. (b) Pair correlation functions (PCFs) from experimental images for the RD-TD plane through the center of the sub-volume for the base material and FSPed samples. PCF value smaller or larger than one corresponds respectively to a smaller or larger probability to find a neighbor in the vicinity of a particle compared to a perfectly homogeneous distribution.	139
3.9	Evolution of (a) porosity volume fraction and (b) mean equivalent diameter of the voids as a function of the number of FSP passes. 3-D views of (c) an initially broken particle (blue) in the BM, (d) initial porosity in the BM and (e) voids observed after 1 pass of FSP. Cavities are in red.	140
3.10	Schematic scenario of the microstructural evolution with the number of FSP passes.	142

3.11	(a) Tensile curves of the base material loaded in the rolling and transverse direction, and after 6 FSP passes in the rolling direction. The base material is in the T4 state, and the FSPed material has been aged for 1 hour at 180°C. Each point corresponds to an interrupted tensile test and the fraction of damaged particles is given in (b). (c) X-ray microtomography reconstructed images of the two samples (A) and (B).	143
3.12	Results obtained on the interrupted tensile test on BM, in the T4 state, loaded in RD up to 420 MPa and characterized by microtomography: (a) Fraction of broken particles as a function of the equivalent particle diameter (i.e. size) obtained from SEM analysis (over 2,000 particles analyzed) and microtomography (over 10,000 particles analyzed). (b) Number of observed fragments as a function of the size of the broken particles.	151
3.13	Variation of the (a) true fracture strains and (b) engineering fracture strain extracted from the uniaxial tensile tests as a function of the yield stress corresponding to the different heat treatments.	152
3.14	Fraction of broken particles as a function of the macroscopic flow stress. Comparison of experimental results (points) with the nucleation model (lines).	153
3.15	Results of the model applied on the T4 state. Ratio between the number of cavities couples satisfying the coalescence criterion and the number of particles, for the model (a) with and (b) without the effect of particle size on void nucleation. The vertical lines indicate the experimental fracture strain (ε_f).	154
3.16	(a) Principle of the RDF analysis for a random distribution. (b) Radial distribution function $g(r)$	156
3.17	Principle of the PCF analysis (here in 2D) for a random distribution. The particles and the measurement frame are shown on the left figure.	157
3.18	Principle of the PCF analysis (here in 2D) for a random distribution. Counting procedure after 2 particles.	157
3.19	Principle of the PCF analysis (here in 2D) for a random distribution. Counting procedure after 15 particles.	158

List of Figures

3.20	Principle of the PCF analysis (here in 2D) for a random distribution. Results of (a) the counting procedure, (b) local volume fraction and (c) PCF.	159
3.21	Comparison between the PCF and the RDF analysis applied on a periodic, a random and a clustered distribution.	160
3.22	Results of the clustering analysis performed on the example image shown in Fig. 3.23. Points A,B,C correspond with the images A,B and C in Fig. 3.23.	162
3.23	Example of a "test image" and results corresponding to points A, B and C of Fig. 3.22. White particles correspond to isolated particles, and particles with the same color belong to the same cluster.	163
3.24	Micrography of the grain structure in (a) the base material and (c) the FSPed material. Measurements for grain size have been performed as shown in Fig.(b) and (d). . .	164
3.25	Micrography of the cross-section of the weld in a plane perpendicular to the processing direction	165
3.26	Micrography of the grain structure in (a) the as-FSPed material and (b) after 20min at 475/475°C. The initiation of abnormal grain growth is observed in (b).	165
3.27	Fracture surface of a FSP tensile specimen (a) without and (b) with post-weld annealing heat treatment. The fracture mode changes from (a) ductile transgranular to (b) intergranular fracture. The color map indicates the height.	166
3.28	(a) Bending test set-up, (b) cracking initiation in the bent sheet and (c) concept of "Selective Formability".	169
3.29	(a) Force-punch displacement curves from bending tests. (b) Specimens BM-L and FSP at the initiation of cracking. (c) Measure of the bending radius R_b . (d) Comparison between experimental results and eq. (3.5)	171
4.1	Variation of the different parameters extracted from the uniaxial tensile tests on the three alloys as a function of the yield stress corresponding to the different heat treatments: (a) tensile strength of three Al alloys; (b) true fracture strain; (c) true strain at the onset of necking.	180

4.2	SEM micrographs taken in the processing-transverse plane showing the distribution of intermetallic particles (in white) in the Al 6056, Al 6061 and Al 6005A.	181
4.3	(<i>Left</i>) 3D microtomography perspective of particles distribution in (a) 6056, (b) 6061 and (c) 6005A aluminium alloys and (<i>Right</i>) corresponding pair correlation functions (PCFs) for the RD-TD plane through the center of the sub-volume. A PCF value smaller or larger than one corresponds respectively to a smaller or larger probability to find a neighbor in the vicinity of a particle compared to a perfectly homogenous distribution.	183
4.4	(<i>Left</i>) Visualisation of a few clusters from particle distribution shown in the SEM images of Fig. 4.2. (<i>Right</i>) PCF analysis performed on clusters from SEM images for the (a) Al 6056 and (b) Al 6005A.	185
4.5	X-ray microtomography reconstructed images just before fracture, of the tensile test performed on Al 6056-T4 loaded in (a) the transverse direction and (b) the rolling direction. Voids appear in black in the grey matrix and IM particles appear in white. The tensile curves in (c) correspond to the in situ tensile tests shown in (a) and (b).	188
4.6	Void distribution observed during in-situ microtomography in the Al 6056 loaded in the transverse direction at a local strain of (a) $\epsilon_{Loc} = 0.1$, (b) $\epsilon_{Loc} = 0.35$ and (c) $\epsilon_{Loc} = 0.43$. (d) Zoom of figure (b), showing the formation of a second population of voids between two clusters of voids. (e) Zoom of figure (c), showing the intercluster coalescence with the formation of micro shear band.	189
4.7	Schematic of the effect of microstructural anisotropy on the damage percolation process. The effect of the stringer orientation on the first coalescence stage (i.e. intra-cluster) is shown in (a) and (b). The effect of the clusters distribution on the second coalescence stage (i.e. inter-cluster) is shown in (c) and (d). In the case of discontinuous stringer (d), a second void growth process based on the early coalesced voids sets in until second coalescence.	190
4.8	Schematics of the damage sequence in Al 6056 ((a), (b) and (c)), and in Al 6061 and 6005A ((d), (e) and (f)).	193

List of Figures

5.1	(a) Geometry of the DENT specimens. DENT configuration of the (b) DENT-T, DENT-L specimens and (c) DENT-FSP specimen. (d) Tip of the notch after electrical discharge machining (EDM) and final machining using fresh razor blades.	200
5.2	Schematic of the energy partitioning between the non-essential work of fracture dissipated within the outer plastic zone (PZ) in blue and the essential work of fracture (damage and necking) dissipated in the fracture process zone (FPZ) in red.	202
5.3	(a) Machining and (b) embedding of the specimens in the resin in order to measure the crack advance Δa_{Tear} after polishing.	204
5.4	(a) Geometry of the μ CT specimens. (b) Configuration of the μ CT-T and μ UT-T specimens. (c) Configuration of the μ CT-FSP and μ UT-FSP specimens.	206
5.5	Nominal strain profiles (DVC measurements), in the AA2198 alloy in T8 state, in the crack propagation direction (L) as a function of the distance from the initial notch root (d_{notch}) for increasing loading steps (0),(1),(2) and (3). Adapted from [235].	208
5.6	Comparison of normal strain fields, in the AA2198 alloy in T3 state, in the loading direction obtained by DVC (left) and “projection DIC” (right) on projected 3D data [236].	209
5.7	Laminography experiment : (a) Loading device with the specimen [238], (b) reconstructed volume with positions of the projected section used in P-DIC. Tomography experiment : (c) Loading device with the specimen (adapted from [81]), (d) reconstructed volume with positions of the projected section used in P-DIC.	210
5.8	Comparison between a single slice and a MIN-projection of 80 slices for laminography data and tomography data obtained on the FSPed Al 6056. The MIN-projection enhance the contrast compared to one slice. . .	211

5.9	Example of the "finite-body" tessellation procedure applied on the <i>MIN</i> -projection shown in (a). The corresponding map of F_V^{local} is shown in (b). A zone in blue/red corresponds to a zone of the matrix with a small/large local porosity, respectively.	212
5.10	(a) Example of vertical (V) and horizontal (U) displacement fields. (b) 3D view of the strain radius used to find the "best fit" plane. The strain field is then computed based on this "best fit" plane.	214
5.11	Region of interest considered in the uncertainty analysis of the volume made of Al 6056. The overlapping zones resulting from the rigid body motion are shown (in red) in order to indicate the horizontal and vertical displacements between the two configurations. The sections used for projection are shown in blue and the projections are shown in the bottom of the image.	217
5.12	(a) Vertical displacement field resulting from the correlation between the two projections shown in Fig. 5.11. (b) Evolution of the strain uncertainty with the size of the strain radius.	218
5.13	True stress-true strain curves for the Al 6056 in T4 condition loaded along the rolling (UT-L) and along the transverse direction (UT-T) and the as-FSPed material loaded along the FSP direction (UT-FSP).	220
5.14	ϵ_{yy} strain fields for (<i>Left</i>) DENT-T and (<i>Right</i>) DENT-L specimens just before the onset of crack propagation. The initial ligament length L_0 is indicated on each figure.	221
5.15	Effect on the ligament length L_0 on the averaged stress acting on the ligament $\sigma_{max}^{Ligament}$	222
5.16	(a) Effect of loading direction and effect of FSP on the essential work of fracture for 2.5mm thick Al 6056 sheets. (b) Evolution of the averaged stress acting on the ligament $\sigma_{max}^{Ligament}$ for the DENT specimens which have been used in (a).	223
5.17	(a) Method used to measure the thickness reduction factor. (b) Variation of the thickness reduction factor for loading along the transverse and rolling direction.	224
5.18	Fractography of DENT-T specimen in the T4 condition.	225

List of Figures

5.19	Fracture surface of an Al 6056 DENT-T specimen indicating the initial flat fracture surface and the subsequent slant fracture under monotonic loading. A transition region connects the flat and the slant parts. The color map indicates height from the initial notch. The height was obtained using a 3D optical microscope.	226
5.20	Fractography of DENT-L specimen in the T4 condition.	227
5.21	Fractography of DENT-FSP specimen in the as-FSPed condition (i.e. close to a T4 condition).	228
5.22	(a) Loading (plain line) and unloading (dotted line) curves of the multiple DENT specimens. (b) J_R curves for the DENT-T and DENT-L cracked plates made of the alloy Al6056.	230
5.23	X-ray microtomography reconstructed images just before fracture, of the tensile test performed on the (d) μ UT-T and (e) μ UT-FSP specimens. The tensile curves in (c) are obtained by dividing the force by the minimum cross-section A_{Loc} of the specimens (see (a) and (b)).	231
5.24	(a) Reconstructed volume from laminography. The 2D projections ((b), (c), (d)) along the three directions allow to fully characterize the 3D damage evolution.	233
5.25	(a) Notch opening displacement (NOD) measurement and (b) Force-NOD curves for for both μ CT-T and μ CT-FSP specimens.	234
5.26	Damage evolution for the μ CT-T specimen. The loading steps (1), (2), (3), (4) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.	235
5.27	Damage evolution for the μ CT-T specimen. The loading steps (5), (6), (7), (8) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.	236
5.28	Damage evolution for the μ CT-FSP specimen. The loading steps (1), (2), (3), (4) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.	237

5.29	Damage evolution for the μ CT-FSP specimen. The loading steps (5), (6), (7), (8) correspond with the Force-NOD curve shown in Fig. 5.25. The projection method is shown in Fig. 5.24.	238
5.30	Fracture surface of the μ CT-T specimen.	240
5.31	Fracture surface of the μ CT-FSP specimen.	241
5.32	(Top) Reconstructed volume of the μ UT-T specimen with the position of the projected sections used in P-DIC procedure and stress-strain curve. The arrows indicate the correlation steps which have been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the correlation steps.	246
5.33	(Top) Reconstructed volume of the μ UT-FSP specimen with the position of the projected sections used in P-DIC procedure and stress-strain curve. The arrow indicates the correlation step which has been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the loading steps.	247
5.34	(Top) Reconstructed volume of the μ CT-T specimen with the positions of the two projected slices used in P-DIC procedure and Force-NOD curve. The arrows indicate the correlation steps which have been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the correlation steps.	248
5.35	(Top) Reconstructed volume of the μ CT-FSP specimen with the position of the projected slice used in P-DIC procedure and Force-NOD curve. The arrows indicate the correlation steps which have been performed. (Bottom) Strain fields and map of the local volume fraction F_v^{Local} of the correlation steps.	249
5.36	Schematics of the damage sequence in : (Left) Uniaxial tensile tests specimens and (Right) DENT specimens. . .	251
5.37	3D microtomography perspective of particles (blue) and initial porosities (red) distribution in the Al 6056. Microtomography data comes from chapter 2 with a resolution of 325 nm.	253

List of Figures

List of Tables

2.1	Chemical composition measured by ICP-OES of the 6005A, 6061 and 6056 Al alloys [wt.%] Al content is the balance.	77
2.2	Mean volume fraction, shape factor, diameter (based on the surface area) of the intermetallic particles in the three aluminium alloys; results based on FEG-SEM image analysis; observations performed at mid-thickness viewed from the top of the plates.	91
2.3	Mean volume fraction and equivalent diameter (based on X-ray microtomography images) of the initial porosities.	91
3.1	Local volume fraction resulting from the finite body tessellation ("All particles") and the clustering analysis ("Particles within clusters" and "isolated particles").	135
3.2	Indicators resulting from the clustering analysis.	137
4.1	Indicators resulting from the clustering analysis.	186

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