

Chapter III :

Influence of the Chemical Composition and Annealing Conditions on the Microstructure of Fe-Mn-Si-Al Steels

III.1 Introduction

Highly alloyed steels have been shown to present excellent mechanical properties such as high work-hardening rate, high ductility and high strength. These superior properties are brought about by mechanically-induced phase transformation. The plasticity brought

about by the progressive transformation of the austenite is called TRIP effect. Indeed, the dilatation and shears provoked by the martensitic transformations may induce some plastic deformations and dynamic softening in the material [Olso78, Naru82]. Moreover, the mechanically-induced phase transformations are comparable to a “dynamic composite effect” due to the progressive formation of the martensite during straining and to the difference of mechanical properties between austenite and martensite. The combination of these effects increase the uniform deformation as well as the strength of TRIP-assisted steels [Bhan72, Tamu92, Jacq98].

As it was presented in Chapter I, the two main parameters which regulate the phase transformations are the relative stability and the stacking fault energy (SFE) of the austenite. On the one hand, the relative stability determines which phase is thermodynamically stable, as a function of the chemical composition and the temperature. In steels presenting mechanically-induced phase transformation, the austenite is metastable, *i.e.* it is stable on cooling to room temperature or lower, but some mechanical load can supply enough mechanical energy to trigger the phase transformation. On the other hand, the SFE controls the crystallographic mechanisms of plastic deformation. Indeed, high SFE will prevent the dissociation of dislocations into partials and the formation of wide stacking faults. However, the nucleation of ϵ -martensite or mechanical twins is brought about by different piling ups of stacking faults in austenite. Moreover, α' -martensite is formed either by direct transformation from the austenite or by indirect transformation, with ϵ -martensite as an

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intermediary phase. Therefore, a low SFE is required to induce mechanically-induced phase transformations, in addition to the low stability of the austenite.

In Fe-Mn alloys, several studies have shown the beneficial influence of silicon and aluminium additions on the mechanically-induced phase transformations [Gräs98, From03]. Indeed, adding aluminium in iron alloys destabilises the austenite and increase the SFE [Ishi74, Chen93]. On the other hand, the addition of silicon also destabilises the austenite, although in a lesser extent, but decreases the SFE [Furu89, Pete97]. Therefore, adjusting the quantities of Si and Al in the alloy influences the proportion of austenite at room temperature and helps tuning the amount of martensitic transformations. Moreover, Chapter I showed that, besides a direct influence on the material mechanical properties, the grain size also affects the stability and SFE of the austenite. Therefore, the microstructure is also a key factor which has an effect on the mechanically-induced phase transformations.

In the present Chapter, two Fe-Mn-Si-Al steel grades which underwent three different annealing treatments were studied, in order to see how the different compositions and annealing conditions modify the microstructure, the austenite stability and the SFE. The influence of the chemical composition was estimated by comparing different calculated pseudo-binary phase diagrams and the SFE predictions of a thermodynamic model. The influence of the annealing treatments is evaluated by microstructure analysis and comparison

with the calculated phase diagrams. These results will be used in the following chapters to investigate the mechanisms of phase transformations and to study the mechanical properties.

III.2 Materials and Experimental Procedures

The exact compositions of the two high Fe-Mn-Si-Al steel grades studied in this Chapter are reminded in Table III.1.

wt. %	C	Mn	Al	Si
Mn15	$1.6 \cdot 10^{-3}$	15.99	3.08	2.80
Mn20	$1.7 \cdot 10^{-3}$	19.66	3.11	2.88

wt. %	Cr	Ni	Mo	N
Mn15	< D. L.	< D. L.	< 0.005	$5.3 \cdot 10^{-5}$
Mn20	< D. L.	< D. L.	< 0.005	$5.7 \cdot 10^{-5}$

*Table III.1 : Chemical compositions of the different steel grades.
(D.L. stands for Detection Limit)*

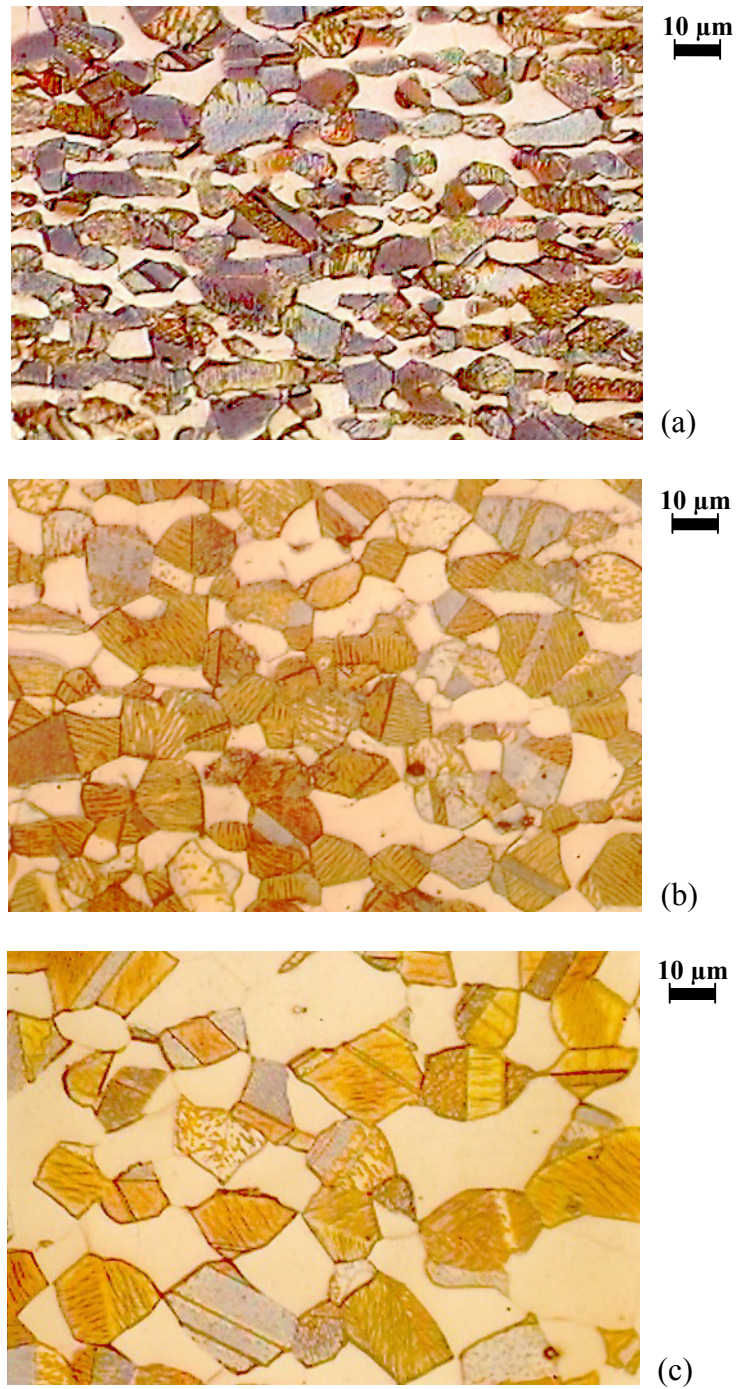
After the last hot-rolling step, the samples were annealed for one hour at 900°C, 1000°C or 1100°C. These annealing temperatures, all high enough to induce a complete recrystallisation after hot rolling, were selected to investigate the influence of the proportion and

chemical composition of the austenite within each material. The samples are named “MnXX YY”, where “XX” refers to the Mn content and “YY” to the annealing temperature (*e.g.* Mn15 1000 stands for a sample of Mn15 steel annealed for 1h at 1000°C). After polishing, the phase proportions were measured by X-ray diffraction and the compositions were determined by WDX-microprobe analysis. The microstructure was examined by optical and transmission microscopy.

III.3 Results

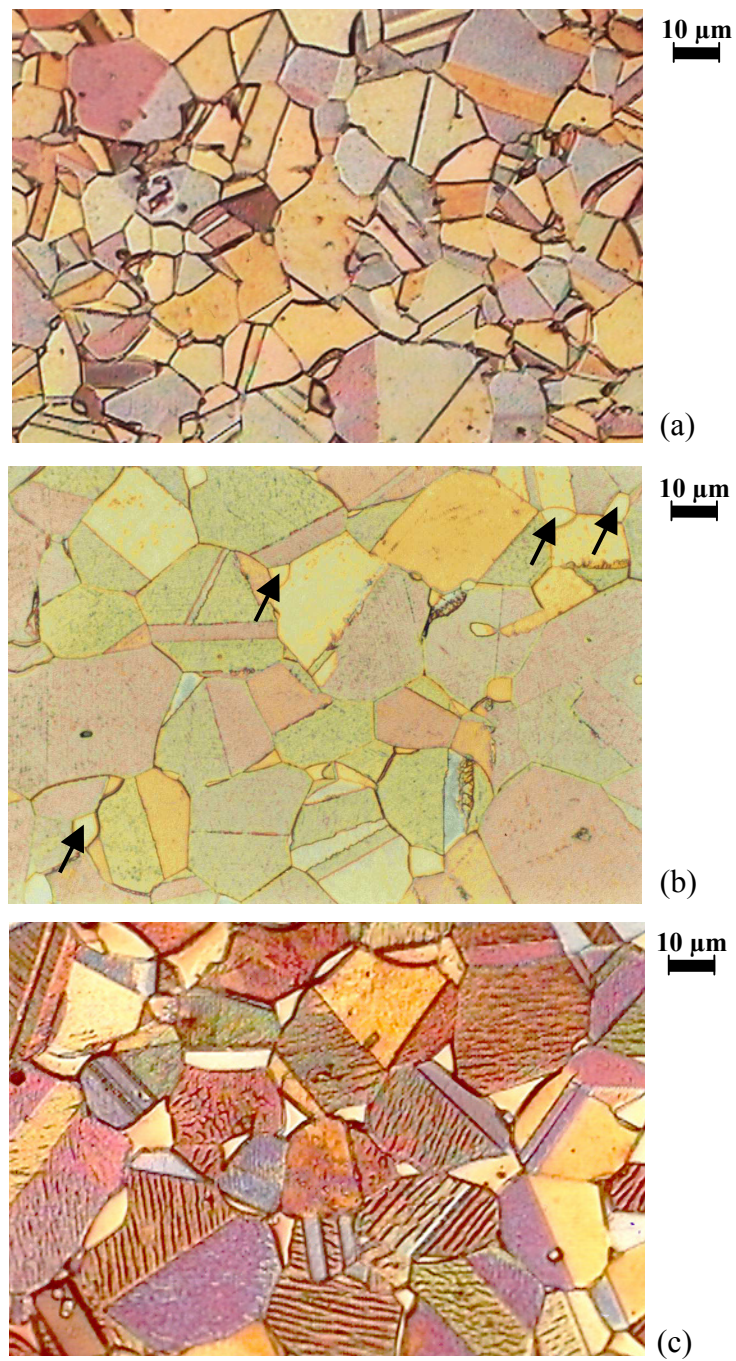
Figures III.1 and III.2 present the microstructures of steels Mn15 and Mn20 annealed for 1h at either 900°C, 1000°C or 1100°C, respectively. The ferritic phase appears in white. First, it must be noticed that the proportion of austenite is higher in steel Mn20 than in steel Mn15. In steel Mn15, former elongated grains resulting from hot rolling can still be seen for the samples annealed at 900°C (Figure III.1 (a)). However, for higher annealing temperatures, this structure transforms into equiaxed grains (Figure III.1 (b) and (c)). In steel Mn20, the microstructure presents equiaxed grains for all annealing conditions. Moreover, the small ferrite grains are mainly located at the austenite triple points.

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*Figure III.1 : Light micrographs of steel Mn15 after annealing for 1h
at 900°C (a) ; 1000°C (b) or 1100°C (c). (the ferrite appears in white)*

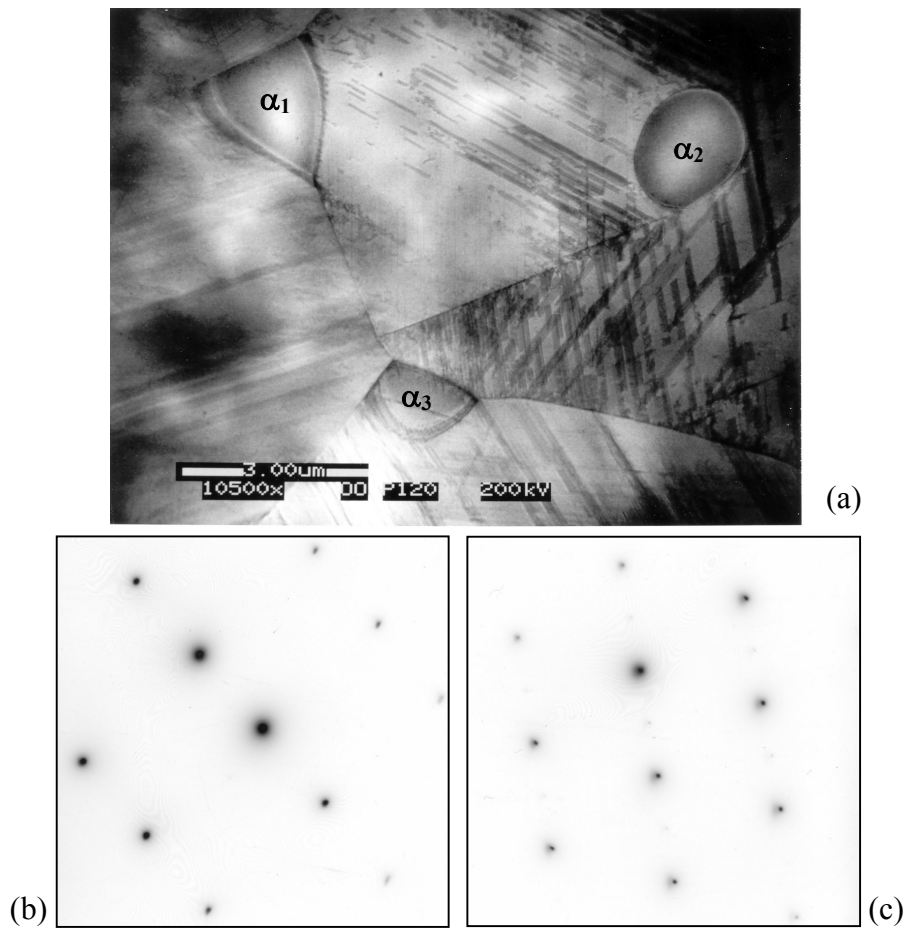
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*Figure III.2 : Light micrographs of steel Mn20 after
annealing for 1h at 900°C (a) ; 1000°C (b) or 1100°C (c).
(ferrite grains indicated by arrows on (b))*

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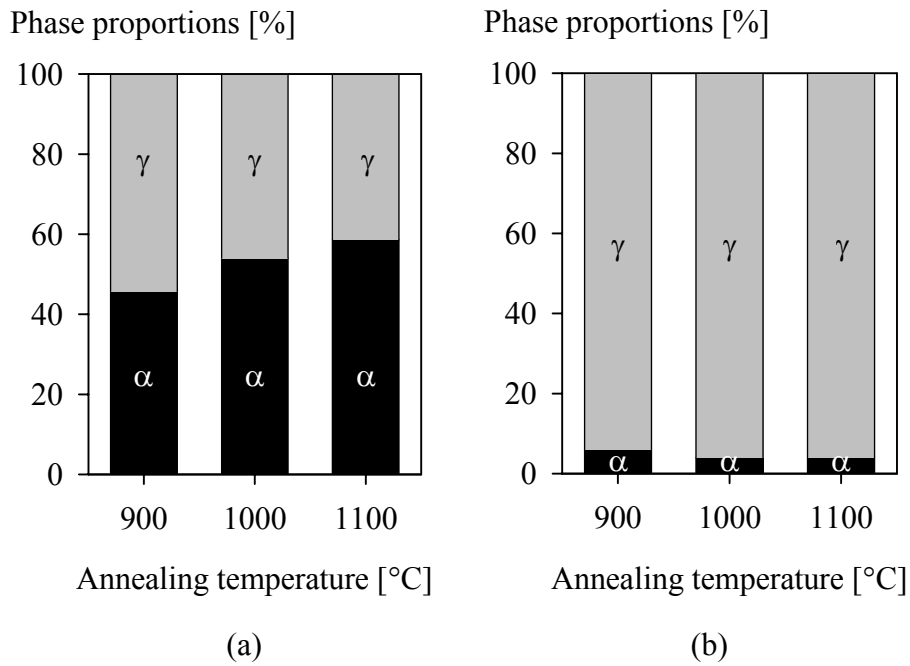
The TEM micrographs of Figure III.3 (a) correspond to the sample Mn20 900. As indicated by the diffraction patterns, the three small grains (α_1 , α_2 and α_3) are made of ferrite (Figure III.3 (b)), while the bigger grains are austenitic (Figure III.3 (c)). The sets of parallel lines in the austenite grains may indicate stacking faults or ϵ -martensite, which could have appeared during the sample preparation.



*Figure III.3 : TEM micrographs of sample Mn20 900 :
(a) bright field image ; (b) diffraction pattern of a ferrite grain
(zone axis = $[0\ 1\ 2]$) ; (c) diffraction pattern of an austenite grain
(zone axis = $[1\ 1\ 2]$).*

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Figure III.4 shows the proportions of phases present in steels Mn15 and Mn20 annealed for 1h at 900°C, 1000°C or 1100°C, determined by X-ray diffraction. This Figure shows that a higher annealing temperature brings about a larger ferrite content in steel Mn15 while the proportions remain almost constant in steel Mn20.

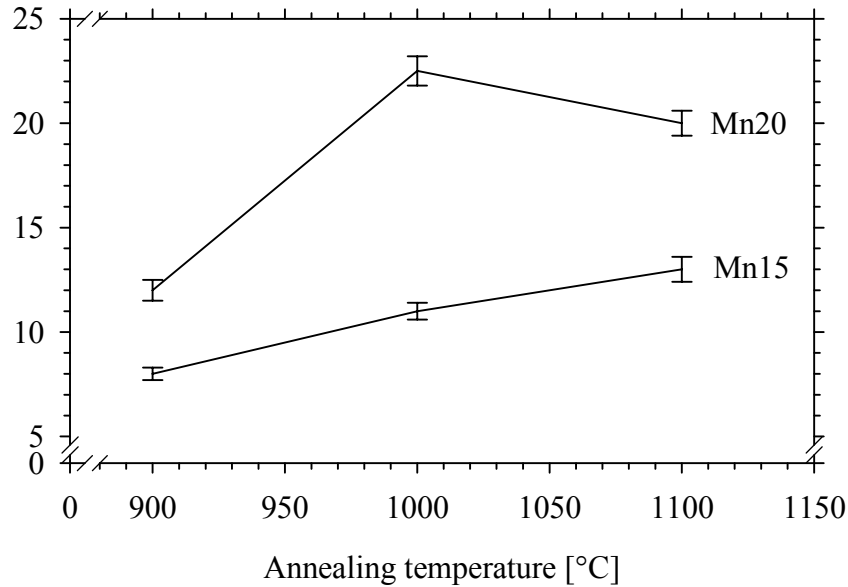


*Figure III.4 : Phase proportions in steels Mn15 (a) and Mn20 (b)
annealed for 1h at either 900°C, 1000°C or 1100°C.*

Figure III.5 summarises the evolution of the austenite grain size with the annealing temperature. For a given annealing temperature, steel Mn15 presents smaller austenite grains than steel Mn20. Moreover, a higher annealing temperature generally induces a larger grain size, except for sample Mn20 1100. However, the growth rate of the austenite grains appears higher in steel Mn20 than in steel Mn15.

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Mean austenite grain size [μm]

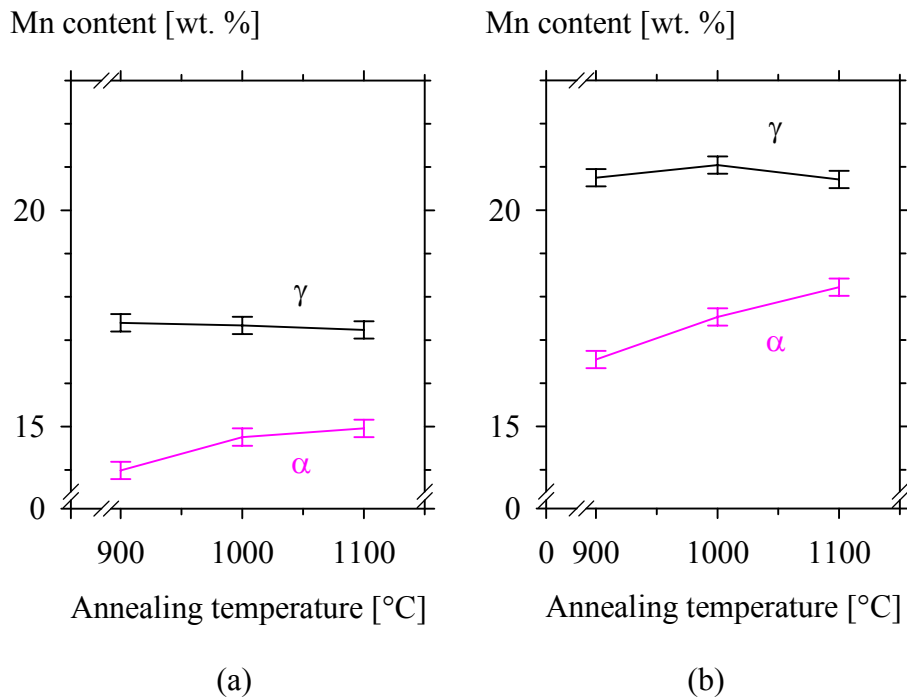


*Figure III.5 : Evolution of the mean austenite grain size
with annealing temperature for steels Mn15 and Mn20
annealed for 1h at 900°C, 1000°C or 1100°C.*

The annealing conditions influence the chemical composition of the phases. No sample-scale segregation of alloying elements can be observed. Furthermore, the level of silicon is constant for all the phases in all specimen. However, partitioning can be observed for manganese and aluminium. The influence of the annealing conditions on the manganese and aluminium content of ferrite and austenite in steels Mn15 and Mn20 are presented in Figures III.6 and III.7, respectively. Manganese segregates within the austenitic phase whereas aluminium tends to concentrate within the ferritic phase. Furthermore, a higher annealing temperature tends to slightly increase

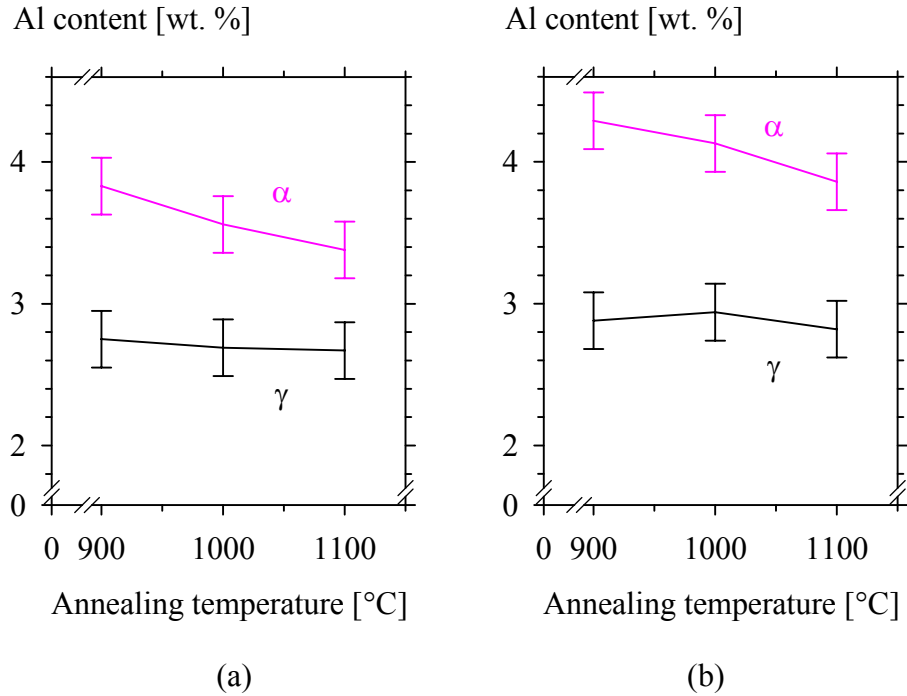
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the Mn content and decrease the Al content of the ferrite while they remain almost constant in austenite. The variation of chemical compositions is consistent with the variation of phase proportions shown in Figure III.4.



*Figure III.6 : Mn content of ferrite and austenite in steels
Mn15 (a) and Mn20 (b) annealed for 1h at 900°C, 1000°C or
1100°C, measured by WDX microprobe.*

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*Figure III.7 : Al content of ferrite and austenite in steels
Mn15 (a) and Mn20 (b) annealed for 1h at 900°C, 1000°C or
1100°C, measured by WDX microprobe.*

III.4 Discussion

III.4.1 Comparison between experimental results and calculated pseudo-binary phase diagrams

Figure III.8 presents two pseudo-binary Fe-Mn phase diagrams (with 3 wt.% Al and 3 wt.% Si) calculated either by Thermo-Calc using the COST+ database (a) or by MTData using the SUB_SGTE database (b). It can be noticed that both diagrams present a similar

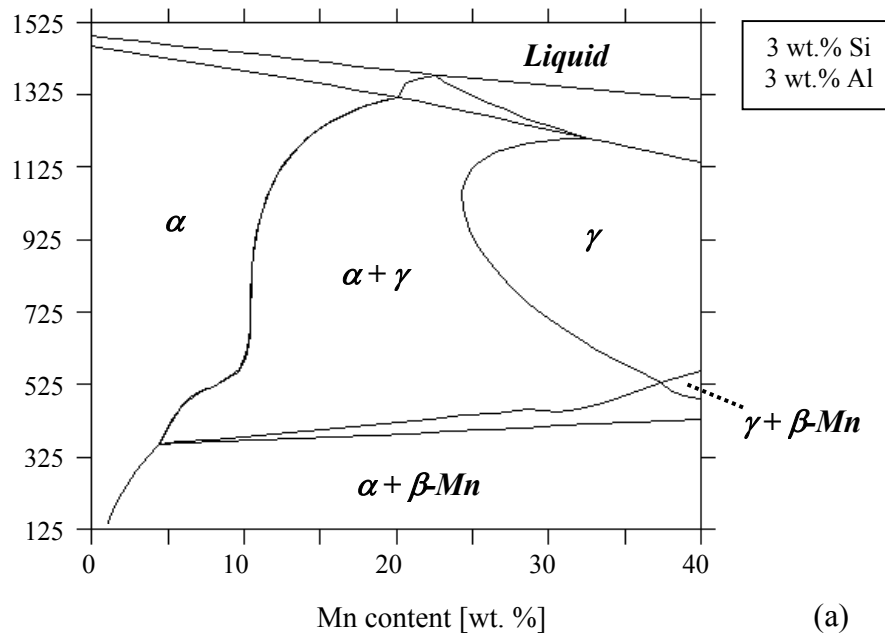
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general morphology. However, compared to the COST+ diagram, the austenitic domain is enlarged towards lower manganese content in the SUB_SGTE diagram, especially at high temperatures. On the other hand, the ferritic domain in the MTData diagram is reduced at high temperatures but enlarged at lower temperatures. In both diagrams, a β -Mn phase appears to be stable at low temperatures. However, this phase is not observed in our samples, which can probably be explained by the cooling rate too high to allow the formation of this phase. Figure III.8 (c) shows the pseudo-binary Fe-Mn phase diagrams (with 3 wt.% Al and 3 wt.% Si) calculated by MTData using the SUB_SGTE database, in which the β -Mn phase has been excluded. It can be seen that the austenite domain encloses now the former (β -Mn + γ) domain. Apart from that, the exclusion of the β -Mn phase brings about little other modifications in the stability domains of the phases and the (α + γ) domain is extended down to room temperature. Although strong differences exist between both diagrams, the phase diagrams are concordant with the fact that microstructure of steels Mn15 and Mn20 at room temperature is composed of various proportions of ferrite and austenite, and that the grade Mn20 will contain more austenite than the grade Mn15. Grässel and Frommeyer [Gräs98] have studied the microstructure of steel grades containing 3wt.% Al, 3wt.% Si and either 15wt.% Mn (similar to grade Mn15 in this work), 20wt.% Mn (similar to grade Mn20 in this work), 25wt.% Mn or 30 wt.% Mn. The samples were annealed for 2h at 1000°C then water quenched. They showed that a minimum level of about 25 wt.% Mn is necessary to obtain a fully austenitic grade. This observation seems to correspond better to the phase diagram calculated by

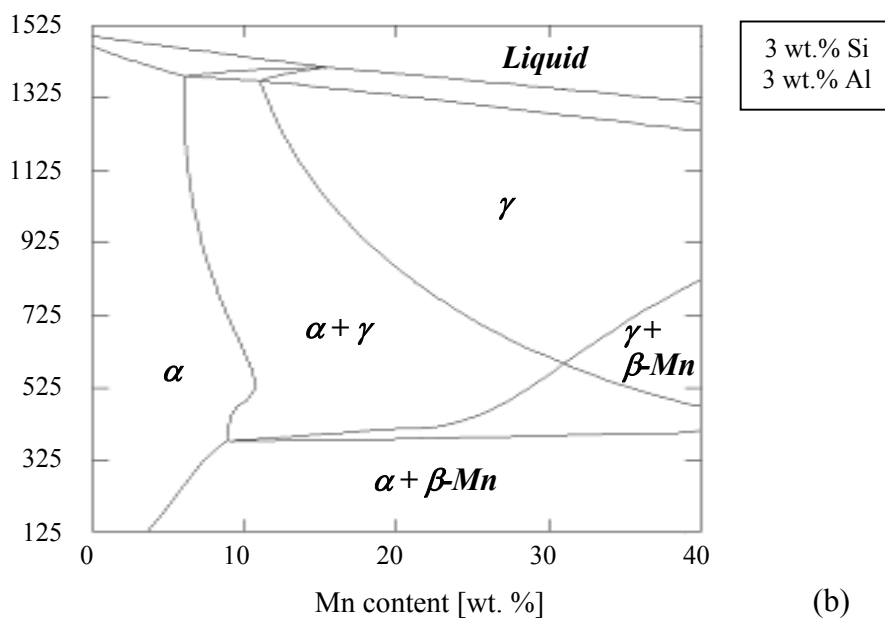
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Thermo-Calc, which will be used to compare with the observations performed on our samples.

Temperature [°C]



Temperature [°C]



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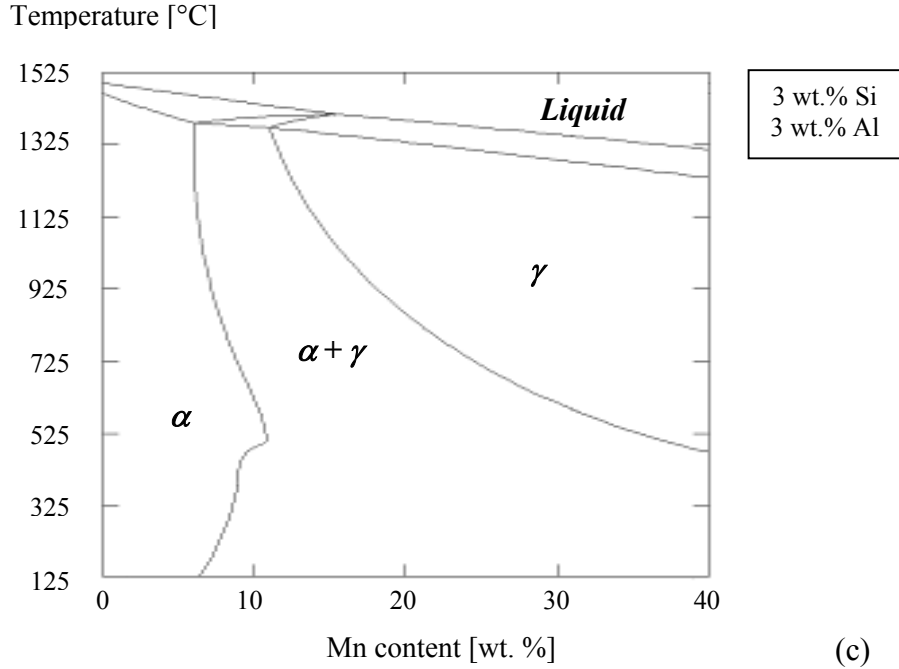
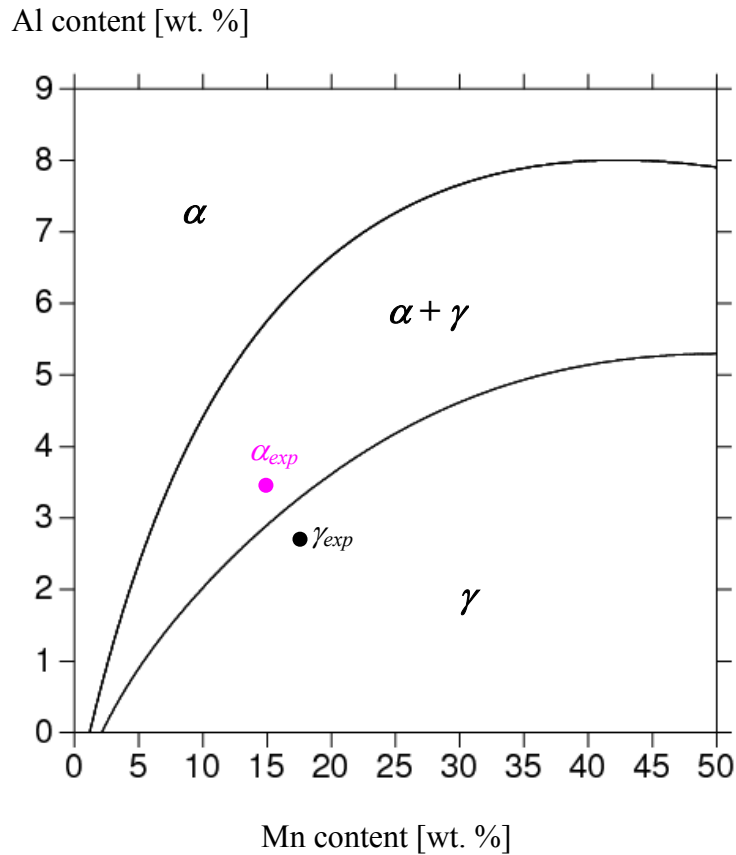


Figure III.8 : Pseudo-binary Fe-Mn phase diagrams (with 3wt.% Al and 3wt.% Si) calculated either (a) by Thermo-Calc using the COST + database or (b) and (c) by MTData using the SUB_SGTE database. The last phase diagram was calculated with the β -Mn phase excluded.

Figure III.9 presents the pseudo-binary phase diagram of Fe-Mn-Al alloy with 2.8 wt.% Si at 1000°C calculated by ThermoCalc using the TCFE2 database. The experimental chemical compositions of the phases measured in Mn15 1000 (α_{exp} and γ_{exp}) are represented on the diagram. Due to the segregation of the alloying elements within the phases, a complete comparison with the equilibrium situation is not

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immediate. However, the chemical compositions appear already to differ from the equilibrium situation.

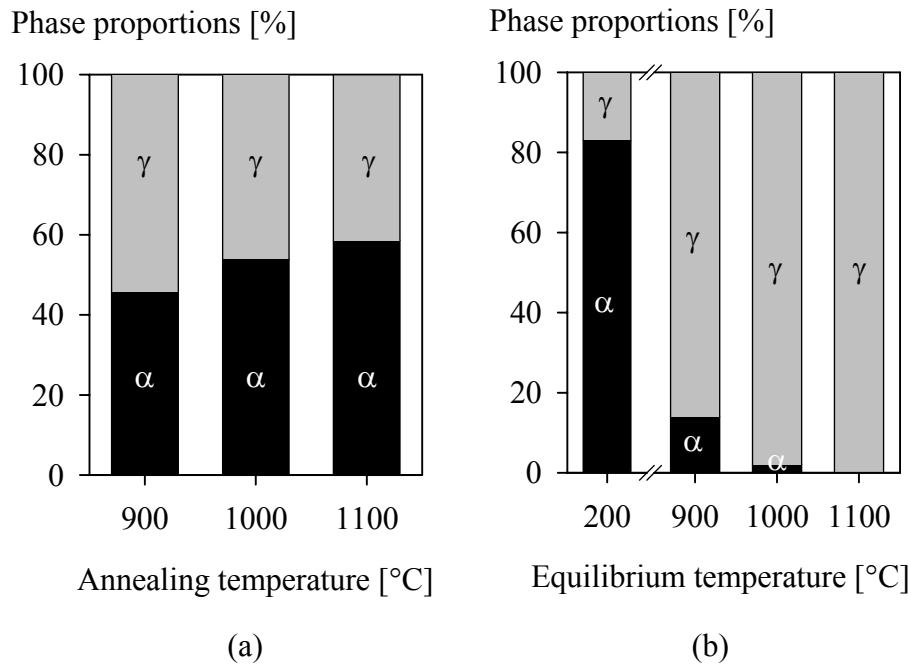


*Figure III.9 : Pseudo-binary phase diagram of Fe-Mn-Al alloy
with 2.8wt%Si at 1000°C calculated by ThermoCalc
using the TCFE2 database.*

Figure III.10 compares the proportions of phases present in steels Mn15 annealed for 1h at 900°C, 1000°C or 1100°C (from Figure III.4) with equilibrium proportions calculated with ThermoCalc using the TCFE2 database at 200°C, 900°C, 1000°C or 1100°C (the MTData

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predictions do not differ significantly). As far as steel Mn20 is concerned, a fully austenitic steel is predicted at the equilibrium. This Figure shows that, at the equilibrium, the proportion of ferrite decreases with increasing temperature, while the observations seem to indicate the opposite tendency. Indeed, the proportion of ferrite increases in steel Mn15 for increasing annealing temperatures. The equilibrium calculated at 200°C does not take into account the formation of β -Mn phase and therefore does not represent accurately the situation at low temperatures. However, it tends to confirm that a large proportion of ferrite should be found at room temperature.



*Figure III.10 : Phase proportions in steel Mn15
(a) annealed for 1h at either 900°C, 1000°C or 1100°C ;
(b) at the equilibrium at 200°C, 900°C, 1000°C or 1100°C
(calculated by ThermoCalc using the TCFE2 database).*

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Table III.2 presents the equilibrium phase compositions calculated by ThermoCalc using the TCFE2 database (the MTData predictions do not differ significantly). At the equilibrium, it can be seen that the levels of Mn are lower and Al are higher, compared to the phase chemical compositions presented in Figures III.6 and III.7. Moreover, the Si also seems to segregate between ferrite and austenite although it could not be observed experimentally. Finally, it must be noticed that the Mn content of austenite at 200°C is very high, because the β -Mn phase was ignored.

Ferrite			
Temperature	Mn [wt. %]	Al [wt. %]	Si [wt. %]
200°C	8.29	3.23	3.28
900 °C	11.76	4.94	2.28
1000 °C	11.55	5.12	2.19
1100 °C	-	-	-

Austenite			
Temperature	Mn [wt. %]	Al [wt. %]	Si [wt. %]
200°C	51.89	2.47	0.56
900 °C	16.69	2.8	2.88
1000 °C	16.08	3.06	2.81
1100 °C	15.99	3.08	2.8

*Table III.2 : Phase compositions of ferrite and austenite
in steel Mn15 at the equilibrium at 200°C, 900°C, 1000°C or
1100°C (calculated by ThermoCalc using the TCFE2 database).*

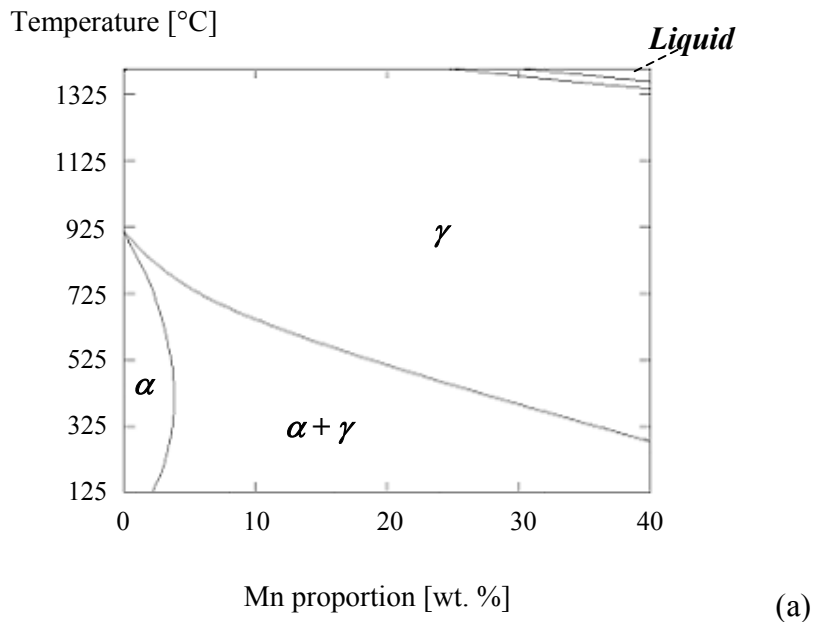
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The comparisons between the measurements and the equilibrium predictions show that the microstructure was affected by the cooling. Figure III.6 and III.7 show that the chemical compositions actually observed are quite far from the calculated equilibrium, either at the annealing temperature or at 200°C. It has been shown that the variation of phase composition as a function of annealing temperature is more important in ferrite than in austenite, where the chemical composition remains almost constant. As the equilibrium is not reached, the difference between austenite and ferrite could be partially explained by a larger diffusivity of alloying elements in ferrite (about two orders of magnitude larger in ferrite than in austenite [MnLR96]), as well as by the smaller proportion of ferrite, especially in steel Mn20. This evolution of the chemical composition in ferrite is compensated by a corresponding evolution of the phase proportions, which leads to a rise of ferrite proportion for higher annealing temperatures in steel Mn15. The same phenomena take place in steel Mn20, but their effect is less visible due to the very low ferrite content in this grade.

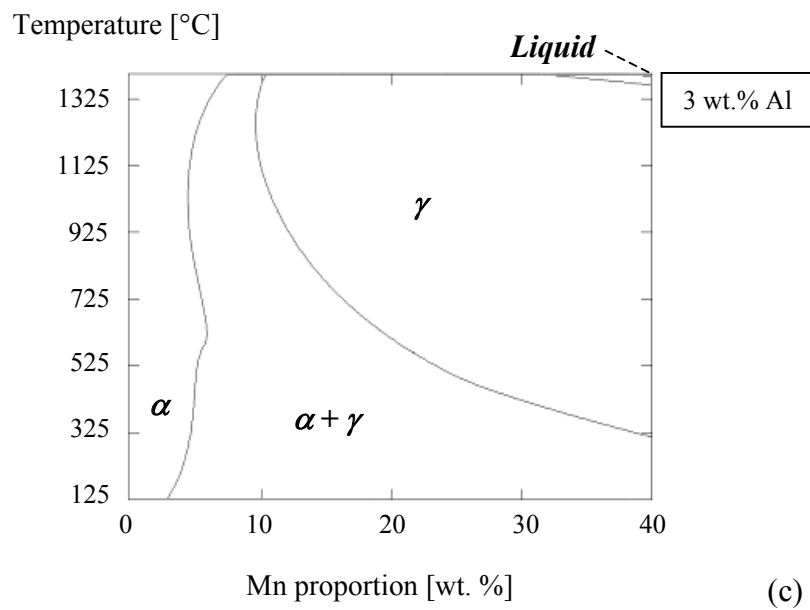
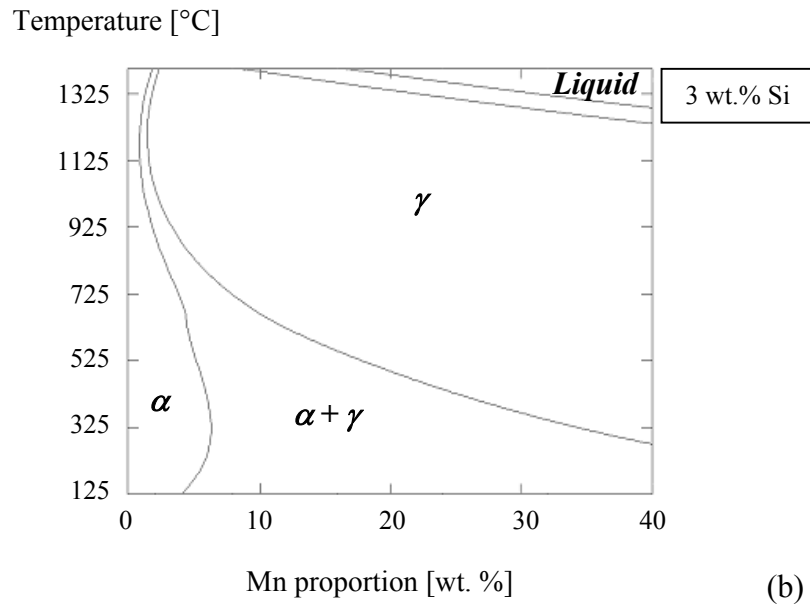
Finally, it must be noticed that a higher annealing temperature increases the austenitic grain size. However, the growth rate of the austenite grains is higher in grade Mn20 than in grade Mn15. This could be explained by the larger ferrite content in steel Mn15, which acts as a barrier against the austenite grain growth.

III.4.2 Influence of the chemical composition and the annealing conditions on the austenite stability

Figure III.11 shows (a) a Fe-Mn phase diagram and two pseudo-binary Fe-Mn phase diagrams with (b) 3 wt.% Si or (c) 3 wt.% Al, respectively. The β -Mn phase has been excluded from the possible phases. As it was mentioned in Chapter I, both Al and Si additions have an alpha-stabilising effect, although the influence of Si is weaker than Al. In the binary Fe-Mn diagram (Figure III.11 (a)), the austenite domain is very large and no ferrite is visible for temperatures over about 900°C. The pseudo binary phase diagrams (Figure III.11 (b) and (c)) show that the ferritic domain is enlarged by the addition of Si or especially Al. The difference is particularly marked at high temperatures.



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*Figure III.10 : Fe-Mn phase diagram (a) and
pseudo-binary Fe-Mn phase diagrams with
3wt.%Si (b) or 3wt.%Al (c) calculated by MTDData
using the SUB_SGTE database.*

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The M_s temperature for grades Mn15 and Mn20 can be estimated using the empiric formula developed for austenitic stainless steels and presented in Chapter I :

$$\begin{aligned} M_s \text{ (in } ^\circ\text{C)} = & 1302 - 42 \text{ (wt.\% Cr)} - 61 \text{ (wt.\% Ni)} \\ & - 33 \text{ (wt.\% Mn)} - 28 \text{ (wt.\% Si)} - \\ & 1667 \text{ (wt.\% C + wt.\% N)} \end{aligned} \quad (\text{III.1})$$

Using the experimental composition of the austenite, the M_s temperature for grades Mn15 and Mn20 are about 650°C and 525°C, respectively. However, these values are only indicative as the formula is used far outside its limit of validity and as the influence of Al content is neglected. The lower M_s temperature in steel Mn20 can be explained by the increase of alloying elements content, which is known to decrease M_s [Laco90]. Moreover, the increase of grain size also reduces the phase stability and increases M_s .

III.4.3 Influence of the chemical composition and the annealing conditions on the stacking fault energy

The chemical composition of the alloys influence the stacking fault energy (SFE). Indeed, in iron alloys, increasing the Al or Mn content increases the SFE while increasing the Si content decreases the SFE [Chen93, Oh95, Pete97]. Using a thermodynamic model, Grässel *et al.* [Gräs97] calculated the SFE for a Fe – 25Mn – 3Si –

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3Al alloy as a function of the temperature. The thermodynamic model is expressed as :

$$SFE_{\gamma} = 2 \rho \Delta G^{\gamma \rightarrow \varepsilon} + 2 \sigma^{\gamma/\varepsilon} \quad (\text{III.1})$$

where ρ is the density of atoms in a closed packed plane in moles per unit area (here $2.53 \cdot 10^{-5}$ mol/m²), $\Delta G^{\gamma \rightarrow \varepsilon}$ is the free energy for the transformation of austenite into ε -martensite and $\sigma^{\gamma/\varepsilon}$ is the γ/ε interfacial energy. As this is not available for the studied alloys, the value of $\sigma^{\gamma/\varepsilon} = 10$ mJ/m² published for Cr-Ni austenitic stainless steel was used. Based on the regular solution model, the evolution of $\Delta G^{\gamma \rightarrow \varepsilon}$ as a function of the temperature was calculated as :

$$\begin{aligned} \Delta G^{\gamma \rightarrow \varepsilon} = & X_{Fe} \Delta G^{\gamma \rightarrow \varepsilon}_{Fe} + X_{Mn} \Delta G^{\gamma \rightarrow \varepsilon}_{Mn} + X_{Al} \\ & \Delta G^{\gamma \rightarrow \varepsilon}_{Al} + X_{Si} \Delta G^{\gamma \rightarrow \varepsilon}_{Si} + X_C \Delta G^{\gamma \rightarrow \varepsilon}_C \\ & + X_{Fe} X_{Mn} (X_{Fe} + X_{Mn})^{-1} \Delta Q^{\gamma \rightarrow \varepsilon}_{FeMn} + \\ & X_{Fe} X_{Al} (X_{Fe} + X_{Al})^{-1} \Delta Q^{\gamma \rightarrow \varepsilon}_{FeAl} + X_{Fe} \\ & X_{Si} (X_{Fe} + X_{Si})^{-1} \Delta Q^{\gamma \rightarrow \varepsilon}_{FeSi} + X_{Fe} X_C \\ & (X_{Fe} + X_C)^{-1} \Delta Q^{\gamma \rightarrow \varepsilon}_{FeC} + \dots \end{aligned} \quad (\text{III.2})$$

where X_i is the mole fraction of the element i and $\Delta Q^{\gamma \rightarrow \varepsilon}_{ij}$ corresponds the interaction parameter between the elements i and j . The thermodynamic data as function of the temperature T are summarized in Table III.3.

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Element i	$\Delta G^{\gamma \rightarrow \varepsilon}_i$ [J/mol] (T [K])
Fe	$-1828.4 + 4.686 T$
Mn	$3970 - 1.66667 T$
Si	$-1800 + T$
Al	$5481.04 - 1.799 T$
C	-24595.12

Elements i, j	$\Delta Q^{\gamma \rightarrow \varepsilon}_{ij}$ [J/mol]
FeMn	$-10836 + 22886 X_{Mn}$
FeSi	1780
FeAl	3323
FeC	42500

*Table III.3 : Thermodynamic data used for the calculation
(from [Gräs97])*

Figure III.12 presents the evolution of the austenite SFE as a function of the temperature in grades Mn15 and Mn20. The levels of Al, Si or Mn used in the model correspond to the average composition of the austenite in samples of steels Mn15 and Mn20, respectively. According to Grässel *et al.* [Gräs97], the selected thermodynamic data used for calculation are not precise enough at low temperatures, but it can nevertheless be seen that at room temperature, the SFE of grades Mn15 and Mn20 are about 9 mJ/m² and 15 mJ/m², respectively.

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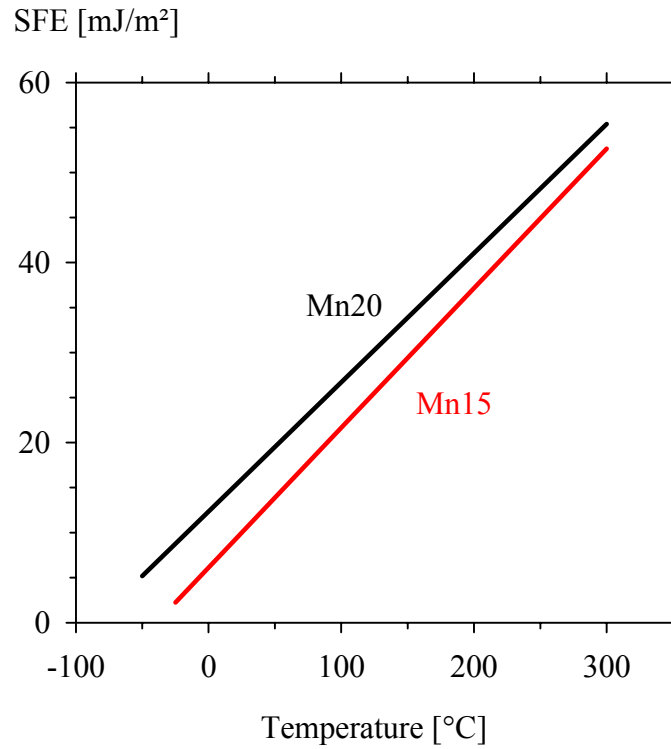


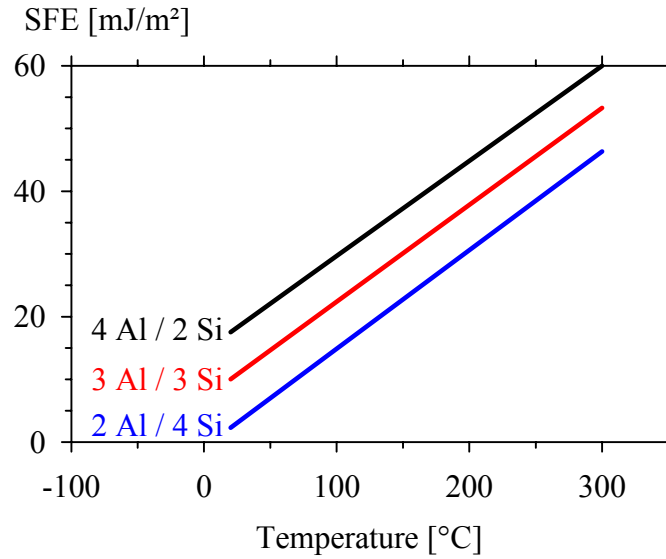
Figure III.12 : Evolution of austenite SFE with temperature in the austenite of steels Mn15 and Mn20 calculated by thermodynamic model from [Gräs97].

Finally, the austenitic grain size is also known to have an influence on the SFE. Indeed, as it was presented in Chapter I, increasing the grain size decreases the SFE. However, the influence is difficult to quantify in alloys such as steels Mn15 and Mn20 where both the proportions and chemical compositions of the phases are also modified by the annealing treatments.

III.4.4 Influence of the Al and Si content on the mechanical properties

As it was demonstrated in the previous sections, Al and Si additions modify the austenite stability and SFE, which affects the mechanically-induced phase transformations and as a consequence the mechanical properties. A study made by Grässel and Frommeyer [Gräs98] showed that for a given Fe-Mn alloy, the testing of different Al/Si ratios proved that the tensile strength increases with increasing Si and decreasing Al contents. However, the addition of 3wt.% Al and 3wt.% Si brings about an optimum in the total elongation under uniaxial tension. Figure III.13 shows the evolution of SFE with temperature in steel Mn15 calculated for three different wt.%Al / wt.%Si ratios (4/2, 3/3 or 2/4) using the thermodynamic model presented in the previous section. The increase of Al/Si ratio increases the SFE, which tends to prevent the phase transformations at room temperature. However, due to the stronger alpha-stabilising effect of the Al, increasing the Al/Si ratio also reduces the stability of the austenite, which favours the phase transformation. The combination of these contradictory effects could lead to an optimum of transformation and transformation rate, which probably explains the optimum of ductility observed in alloys containing 3wt.% Al and 3wt.% Si.

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*Figure III.13 : Evolution of austenite SFE with temperature in steel Mn15 with different levels of Al and Si additions, calculated by thermodynamic model from [Gräs97].
(all quantities are in wt.%)*

III.5 Conclusion

The influence of chemical composition and annealing temperature on the microstructure, the phases relative stability and the SFE of Fe-Mn-Si-Al alloys are investigated in this Chapter. The observations will be used as basis for the study of the Fe-Mn-Si-Al steel grades in the next Chapters.

It can be concluded that the steel grades containing 3wt.% Al and 3wt.% Si exhibit a ferrite-austenite microstructure for Mn contents of 15wt.% or 20wt.%. This is consistent with the literature [Gräs98]

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which proves that a minimum of about 25wt.% Mn is required to obtain fully austenitic alloys. The β -Mn phase predicted by the calculated phase diagrams is not found in our samples because of its low temperature of formation. On the one hand, Al and in a lesser extent Si additions reduce the austenite stability while Mn increases it. On the other hand, the increase of Al and Mn content increase the stacking fault energy while Si decreases it. Therefore, the optimum of tensile elongation observed in Fe-Mn steels containing 3wt.% Al and 3wt.% Si is probably due to an optimum of phase transformations brought about by the different influences of these elements on the austenite.

The imposed annealing conditions on steels Mn15 and Mn20 lead to segregation of manganese between the phases and in a lesser extent of aluminium. The equilibrium compositions predicted by the phase diagrams are not reached. The different annealing conditions induce variations of chemical composition in the ferrite while it remains almost constant in the austenite. These variations are accompanied by an evolution in the proportions of the constitutive phases, especially in steel Mn15, where a higher annealing temperature increases the proportion of ferrite. Finally, the growth rate of the austenite grains is higher in grade Mn20 than in grade Mn15, probably due to the smaller fraction of ferrite which acts as a barrier against the austenite grain growth.

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