Conclusion and Prospects

General Conclusion

Different aspects have been investigated in this work :

- the influence of the chemical composition and annealing conditions on the phases relative stability and the stacking fault energy of Fe-Mn-Si-Al steels ;
- different methods of grain refinement as well as the kinetics of grain growth in fully austenitic stainless steels ;
- the crystallographic mechanisms of the successive mechanically-induced phase transformations in both Fe-Mn-Si-Al and Fe-Ni-Cr grades ;
- the influence of these phase transformations on the mechanical properties.

The Fe-Mn-Si-Al steel grades containing 3wt.% of aluminium and silicon exhibit a ferrite – austenite microstructure for manganese contents of 15wt.% or 20wt.%. Al and, in a lesser extent, Si additions reduce the austenite stability while Mn increases it. Moreover, the increase of Al and Mn contents increases the stacking fault energy while Si decreases it. Therefore, the optimum of tensile elongation observed in Fe-Mn steels containing exactly 3wt.% Al and 3wt.% Si for all Mn contents is probably due to an optimum of phase transformations brought about by the different influences of these elements on the austenite. Segregations of Mn and Al were observed between the phases. 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.

Grain size refinement was achieved either by annealing after rolling at 20°C (to induce a cycle of phase transformations : austenite – α'-martensite – austenite), or after rolling at 200°C (to recrystallise the deformed austenite without inducing phase transformations). The method using a cycle of phase transformations proved to be the most effective due to the larger amount of new nuclei during retransformation compared to recrystallisation. Moreover, the kinetics of these phenomena are very different. Indeed, it was shown in partially transformed samples that the retransformation is almost complete before the recrystallisation starts and that the recrystallisation is not affected by the retransformation. Finally, the grain growth rate is so low that it can be neglected while recrystallisation or retransformation take place. The grain growth behaviour follows the Burke-Turnbull analysis and the grain growth exponent increases with the annealing temperature (*i.e.* the growth rate decreases). This could be due to the difference of original grain size (before the grain growth starts) as the grain growth rate is higher in a finer microstructure.

Based on the literature [Lee01a, Lee01b, Spen04, Gey05] and on observations in Ni-Cr stainless steel 301LN and Fe-Mn-Si-Al grade Mn20, a crystallographic mechanism of phase transformation involving two steps can be determined :

1. The austenite transforms into ε-martensite, which forms as bands along {1 1 1} austenitic planes. In steel Mn20, the orientation of ε bands correspond to the most favourably oriented planes for gliding of dislocations relatively to the externally applied stress, confirming the formation mechanism of ε-martensite by piling up stacking faults in the austenite.
2. The α'-martensite nucleates on the bands of ε martensite, generally at the junction of two bands. In Fe-Mn-Si-Al steels, the α' grains grow strictly within the ε phase while in Fe-Ni-Cr steels, the α' grains grow within the ε bands but also directly in the austenite grains.

A variant selection phenomenon can be observed during the formation of both ε- and α'-martensite. The relative weight of the ε variants depends on their orientation with respect to the externally applied stress. Moreover, in stainless steels, the α' variant selection is controlled by the crystallography of the transformations and the relative orientation of the transformation strain to the local stress field. In Fe-Mn-Si-Al steels, a very strong α' variant selection takes place and the α' orientation is dictated by crystallographic considerations only. In some rare occasions, a secondary austenite variant is formed at the junction of two ε bands in Mn steels. To accommodate the interface misorientation, the secondary austenite variant or a part of the neighbouring ε bands transforms into secondary ε-martensite, which presents a twin relationship with the band of primary ε-martensite.

During tensile straining, the proportion of ε phase increases, reaches a maximum then decreases in both Fe-Ni-Cr and Fe-Mn-Si-Al steel grades. In stainless steels, the α'-martensite proportion increases continuously with deformation. However, because of the phase transformations mechanisms, the formation of α'-martensite is prevented in Fe-Mn-Si-Al steels up to the point where about 10-12% of the austenite has transformed into ε-martensite. Then, the proportion of α'-martensite increases continuously with strain. In both materials, the work-hardening rate is directly related to the formation of α'-martensite while the proportion of ε-martensite does not seem to have any influence.

In high Mn grades, a higher Mn content decreases the yield strength as it increases the proportion of softer austenite. Moreover, it stabilises the austenite and increases the austenite stacking fault energy, which decreases the phase transformations. Furthermore, the modification of the phase proportions brought about by the higher Mn content also decreases the stress level in the austenite. This effect plays a capital role in the reduction of phase transformations. In stainless steels, an increase of the Ni content stabilises the austenite and an increase of the Ni and Cr contents rises the SFE. The combination of these effects tends to reduce the phase transformation rate and intensity in stainless steels with higher Ni- and Cr-levels. A temperature rise, either due to a higher testing temperature or strain rate, increases the SFE and stabilises the austenite, bringing about a decrease in the intensity and in the rate of phase transformations. Moreover, a smaller austenitic grain size also reduces the phase transformation. Finally, a higher stress triaxiality enhances the phase transformation in stainless steels as the applied stress tends to have a more favourable influence on the transformation strain. A lower Lode parameter has the same effect, as it corresponds to a more favourable shear component in the applied stress.

Prospects

The different aspects this thesis were thoroughly investigated using the time and means at our disposal. However, these results are not meant to be complete and future work would be required to complete this study.

The investigation of the influence of the chemical composition and annealing conditions on the Fe-Mn-Si-Al steels could be completed by testing other Al and Si content. Moreover, the effect on the austenite stability could be estimated by measuring the M_s temperature for different chemical compositions.

Two ways of completing the analysis of retransformation and recrystallization presented in Chapter IV should be investigated :

1. the first steps of retransformation should be studied with more attention, which implies to repeat the measurements with a lower annealing temperature or for shorter annealing times. The influence of the microstructure prior to annealing could be examined more thoroughly by the analysis of two complementary sets of samples, deformed at different temperatures so the proportion of martensite after rolling would be either 100% or 30 to 50%.
2. it has been shown that the nucleation step is a key parameter to determine the final microstructure. To look into the influence of the nucleation step, the mechanical treatment prior to annealing could be modified to reach the same phase proportions, but with different levels of deformation. Indeed, the density of defaults in the austenite and the martensite fraction influences the mechanisms of recrystallisation [Belya05]. In our case, it could change, for example, the density of preferred sites for nucleation. Another possibility would be to increase the heating rate for the annealing. This would make the nucleation step predominant compared to grain growth, and the driving force and the number of nuclei would be increased. Indeed,

it has been observed that for the samples annealed at high temperatures, the heating rate was not large enough to allow easy comparisons.

In order to complete the understanding of the phase transformation mechanisms, a systematic study of the orientation relationships and variant selections observed during deformation would be necessary. Indeed, the limited amount of grains examined does not allow to validate general laws. Moreover, a closer look at the nucleation mechanisms and at the interface relationships should be performed. TEM analysis or in-situ measurements at various stage of deformation could provide a fine understanding of the sequence of events. The influence of grain size could also be quantified if a systematic analysis would be performed in fully austenitic steels, taking into account the size and the orientation relatively to the applied stress for each studied grain.

If the crystallographic mechanisms of formation of secondary variants in Fe-Mn-Si-Al steels were satisfactorily explained by hard sphere models, the conditions which govern the formation of secondary variants instead of α'-martensite were not clearly elucidated. A systematic study on the orientation of the austenite grain relatively to the applied stress would be necessary to determine the necessary conditions for this type of phase transformations.

Several parameters affecting the phase transformations and the mechanical properties have been studied. However, the measurements were all issued from experiments performed in laboratory conditions. In the idea of an industrial use, it would be useful to see how the phase transformations and the mechanical properties would evolve in tests designed to match future applications : industrial annealing conditions, straining under various conditions of temperature, loading rate and triaxiality, etc. Indeed, it has been shown that the conditions of deformation have a tremendous influence on the phase transformations and on the mechanical properties. Therefore, this thesis may be used as a basis to understand the phenomena taking place in the materials, but not as a predicting tool for future applications.

References

[Gey05] N. Gey, B. Petit and M. Humbert, *Metal. and Mater. Trans. A*, 2005, Vol. 36A, p. 1957.

[Lee01a] E. H. Lee, M. H. Yoo, T. S. Byun, J. D. Hunn, K Farrell and L. K. Mansur, *Acta Mat.*, 2001, Vol. 49, p. 3269.

[Lee01b] E. H. Lee, T. S. Byun, J. D. Hunn, M. H. Yoo, K Farrell and L. K. Mansur, *Acta Mat.*, 2001, Vol. 49, p. 3277.

[Spen04] K. Spencer, **The work hardening of austenitic stainless steel, applied to the fabrication of high-strength conductors**, PhD Thesis, Mc Master University, 2004.