The Influence of Nb and Ti on the Recrystallization of UHP Iron

P. Jouffrey, J. Driver

JOURNAL DE PHYSIQUE IV
Colloque C7, supplément au Journal de Physique III, Volume 5, novembre 1995

The Influence of Nb and Ti on the Recrystallization of UHP Iron

P. Jouffrey and J.H. Driver

Microstructures and Processing Laboratory, Ecole des Mines de Saint-Etienne, 42023 Saint-Etienne cedex 2, France

Abstract. The influence of Nb and Ti in solid solution on the recrystallization mechanisms and textures of cold rolled high purity iron has been studied by optical metallography, SEM, microtexture (EBSD) and macrotexture (X-ray) measurements.
Two UHP iron based alloys (Fe-0.08 wt% Nb and Fe-0.04 wt% Ti) together with two reference samples of UHP Fe and electrolytic iron, were treated to form an equiaxed grain structure then rolled 81 % to develop standard bcc rolling textures. The recrystallization mechanisms were then studied by partial recrystallization anneals in the temperature range 475 -800°C. The orientations of the new recrystallized grain were essentially a mixture of {111} and {100} parallel to the sheet planes. Carbon, Ti and to a lesser extent, Nb favour the nucleation and growth of {111} planes grains along prior grain boundaries.

1. Introduction

Over the last ten years, there has been a significant increase in the use of interstitial free (IF) steels for sheet metal applications requiring improved deep drawability. They possess good strength due to their fine grain size, high ductility by virtue of the low carbon contents ($\leq$ 500 pm) and particularly good anisotropy (Lankford) coefficients, r $\geq$ 2. These attractive properties are obtained by a combination of close compositional control and thermomechanical processing, in particular cold rolling and annealing. The fine grain size is obtained by trace addition of Nb and/or Ti which combine with the small amount of carbon to form fine carbide precipitate dispersions for grain boundary pinning. Usually the Nb or Ti contents are in excess of those required for stoechiometric carbide formation so that some metallic alloying element remains in solid solution. During annealing after cold rolling, the aim is to develop a recrystallization texture which gives high r values, ideally {111} components parallel to the sheet plane without any specific crystal lographic direction along RD. It is now considered that Nb and/or Ti contents greater than 0.05 wt % favour the formation of the {111} components, but it is not known to what extent the Nb/Ti elements act as carbide precipitates or in solid solution [1].
The aim of this on-going work is to characterize the role of Nb and Ti in solid solution on the recrystallization mechanisms and texture development in cold rolled iron. To avoid carbide precipitates an ultra purity iron from the EMSE has been used as the base metal. Binary alloys containing 0.04 wt% Nb or 0.08% wt% Ti after cold rolling were annealed to different degrees of recrystallization and the textures characterized, at the macroscopic level by Xray pole figures, and at the micron-scale by electron back scattered diffraction (EBSD). The results on the high binary purity alloys are compared with the behaviour of both UHP iron and electrolytic iron.

2. Experimental procedure

The Fe-Nb and Fe-Ti binary alloy compositions were chosen following the work of Houck, Hecler, and Elias [2] and Katoh et al. [3] concerning the respective influences of Nb and Ti in solid solution on the Lankford coefficients of IF steels. High r values, corresponding to the optimum recrystallization textures,

Article published online by EDP Sciences and available at http://dx.doi.org/10.1051/jp4:1995730

were attained for Nb contents ≥ 0.06% and Ti ≥ 0.03%. With a view to characterizing and comparing the recrystallization textures of Nb/Ti free (low r) and Nb/Ti containing alloys, the compositions of the latter were selected at 0.08 wt% Nb and 0.04 wt% Ti .
The binary alloys were prepared in the form of 200 g bars by induction melting ultra high purity iron with small quantities of high purity Nb and Ti under flowing argon. The alloy nomenclatures and compositions are given in Table 1.

Table 1 : alloys compositions - *Measured by internal friction (INSA, Lyon)

Sample	C (ppm)	N (ppm)	Nb (wt%)	Ti (wt%)
UHP Fe	0,25 *	2 *	<0,01	<0,01
Elec Fe	<140	<10		
Fe - Nb	0,25 *	2 *	0,08	<0,01
Fe - Ti	0,25 *	2 *	<0,01	0,04

After melting the bars were hot forged to square sections of side ~ 11 mm and length ~ 95 mm. To obtain an initial state of equiaxed grains, the UHP metal bars were then annealed in the ferrite phase field (40 mns at 600°C). In contrast, the electrolytic iron was treated in the austenite domaine and rapidly cooled to give an acicular ferrite structure.
This recrystallized material (state I) was then cold rolled by 0,5 mm reductions to a final thickness reduction of 81% (ε = 1,7), corresponding to a typical cold rolling sequence of IF steels.
Specimens taken from the centre of the sheet were chemically cleaned then annealed under vacuum in sealed silica capsules for the recrystallization study. Annealing sequences were carred out for fixed times at 50°C temperature intervals or at a given temperature for different times.
The recrystallization kinetics were evaluated by hardness measurements (HV 10Kg). Macroscopic textures were determined by Xray pole figures using the Dosophatex 4 circle texture goniometer and appropriate software for ODF analysis. Microstructures and microtextures were characterized by SEM (JSM 6400) equipped with a Sintef EBSD system and semi-automatic Kikuchi line indexation. A high resolution TV scan rate BSE detector was used to obtain good quality images of the first stages of recrystallization.

3. Results and Discussion

3. 1 *Recrystallization temperatures*

Figure 1 illustrates the hardness values of the four grades after cold rolling, as a function of subsequent annealing temperature for a constant time (30 mn). It is evident that recrystallization in the binary alloys requires significantly higher annealing temperatures than the pure metal. Another feature is the break in the softening curve with temperature ; the low temperature softening is attributed to recovery effects while recrystallization dominates the high temperature behaviour. Finally, after recrystallization the micro-alloyed Nb and Ti alloys exhibit a slightly higher hardness due to Nb and Ti in solid solution. The hardness plots of figure 1 were used to select appropriate annealing temperatures for the recrystallization study.

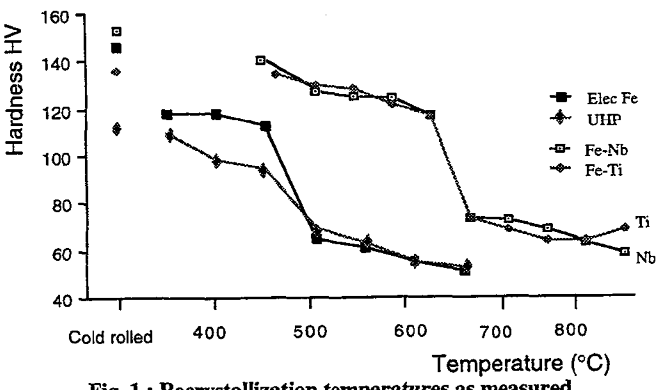


Fig. 1 : Recrystallization temperatures as measured by hardness tests after annealing 30 mn

3. 2 *Initial state prior to cold rolling*

After hot forging and annealing below Ac_1, the samples exhibit typical recrystallization textures (as measured by EBSD and X rays) characterized by <110> // to RD and a mixture of {111},{112}, {113} and {001} planes parallel to the sheet plane . A significantly weaker texture was found in the electrolytique iron after transformation from austenite .

3. 3 *Cold rolled and recovered states*

The cold rolled textures (Figure 2) are typical of bcc metal rolling textures, a partial α fibre with RD //<100> and the γ fibre components with ND // <111> [4] . There is a noticeable tendancy for the texture components of the micro alloyed "steels" to be less sharply defined than the high purity iron.
During recovery, the same texture components are retained but appear to be slightly reinforced (treatments of 30 mn at ~ 400°C for Fe and ~ 600°C for Fe-(Nb,Ti) . In the SEM the recovery process is characterized by the formation of a few bands of highly recovered subgrains. These bands, mostly of cube {100}<001> orientation, quickly develop polygonized structures of equiaxed subgrains whith relatively high local misorientations whichh make them easily identifiable in channelling contrast (BSE).

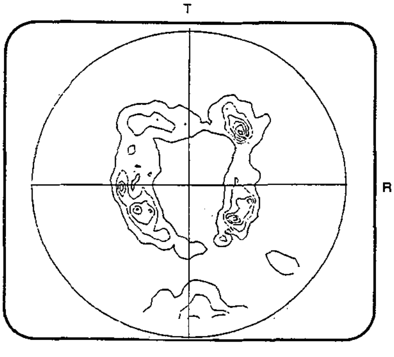


Fig. 2 : {110} Xray pole figure of cold rolled UHP iron

3. 4 *Partially recrystallized states*

Recrystallization proceeds in stages which can be descibed as (i) isolated grain nucleation, (ii) nucleation along bands, (iii) growth and impingement. The first isolated nuclei are developed essentially along grain boundaries. Intergranular nucleation accounts for only about 10% of the observed nuclei, always within grain orientations belonging to the γ fibre. Figure 3 shows a typical example of grain boundary nucleation in UHP Fe at 425°C creating a {111} <110> nuclei between two {111} <112> grains. Detailed EBSD microtexture analysis of about 50 nuclei in each of the four iron compositions reveals that the nuclei almost alway have {111}<uvw> or {100} <uvw> orientation as summarized in Table 2.

Table 2 : Isolated nuclei and banded nuclei orientation classed in approximate pourcentages of {111} and {001} components.

	Nuclei orientation (%)		**Band nuclei orientat. (%)**	
Sample	{111}<uvw>	{100}<uvw>	{111}<uvw>	{100}<uvw>
Elec Fe	85	15	70	30
UHP Fe	30	70	60	40
Fe - Nb	50	50	60	40
Fe - Ti	50	50	90	10

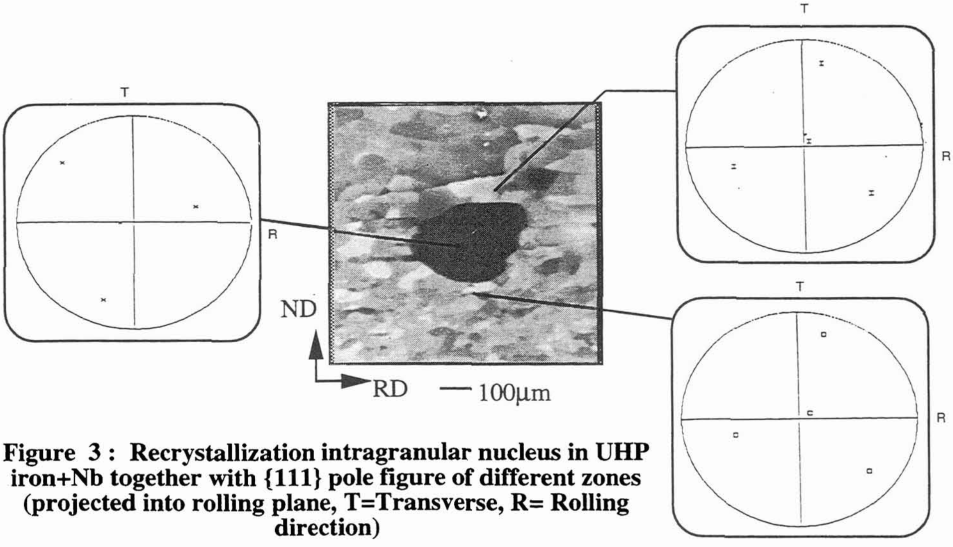


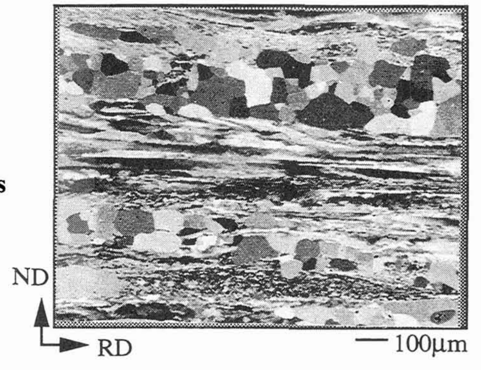
Figure 3 : Recrystallization intragranular nucleus in UHP iron+Nb together with {111} pole figure of different zones (projected into rolling plane, T=Transverse, R= Rolling direction)

The pronounced tendancy for {111} nucleation along grain boundaries in the electolytic iron is consistent with previous results of Hutchinson [5]. However, the UHP Fe completely reverses this trend since it strongly favours {100} grain nuclei. There is obviously a marked influence of solid solution elements on the nuclei orientation, substitutional elements such as Nb and Ti increase the amount of {111} compared with UHP Fe but not so much as in electrolytic iron.

The orientations of the recrystallized nuclei have also been compared with those of the surrounding grains but, as yet, we have not been able to establish well-defined orientation relationships (in agreement with similar work on IF steels, by M. Kiaei, B. Bacroix and J. H. Schmitt [6].

At a slightly later stage of recrystallization, the isolated intergranular nuclei are succeded by long bands of small recrystallization grains along distances of ~ 1mm (the approximate length of the as-rolled grains), implying easy nucleation along certain grain boundaries and micro-growth into specific grains. Figure 4 depicts an example of two recrystallized bands, one containing {111} <uvw> grains and the other a mixture of {111} and {100}. Within the recrystallized bands it is possible to find non-recrystallized areas of the original grains ; they usually have the same type of orientation as the recrystallized grains. There again, there is no clear trend for the orientation relations with the adjoining deformed grains. However as shown in Table 3 there is a general increase in the density of the {111} grains compared with the first stages of nucleation, particularly in the case of the titanium containing alloy.

Figure 4 : an example of two recrystallized bands in Fe-Ti



Towards the final stage of recrystallization (~ 90%) the remaining non-recrystallized grains can be considered to represent the orientations in which nucleation and/or growth is particularly difficult. A systematic study of the orientations of the last grains to recrystallize shows that apart from a few isolated {111} grains, the vast majority are α fibre components particularly {100} <uvw>. Recalling that the first grains to recover possess the cube orientation it would appear that are also the last to recrystallize, in agreement with the results of Lindh, Hutchinson and Bate [7].

3. 5 *Fully recrystallized material*

After both the isochronal and isothermal treatments to complete recrystallization, the final microstructures are relatively equiaxed grains (Figure 4) with the textures given schematically in Table 3 in terms of {111} and {100} sheet components.

Table 3 : Texture classifications after complete recrystallization

	Isochronal (30 mms)			**Isothermal (6 - 10h)**		
	°C	**{111}**	**{100}**	**°C**	**{111}**	**{100}**
Electro Fe	575	80	20	500	70	30
UHP Fe	550	50	50	475	50	50
Fe - Nb	800	60	40	700	60	40
Fe - Ti	750	70	30	650	70	30

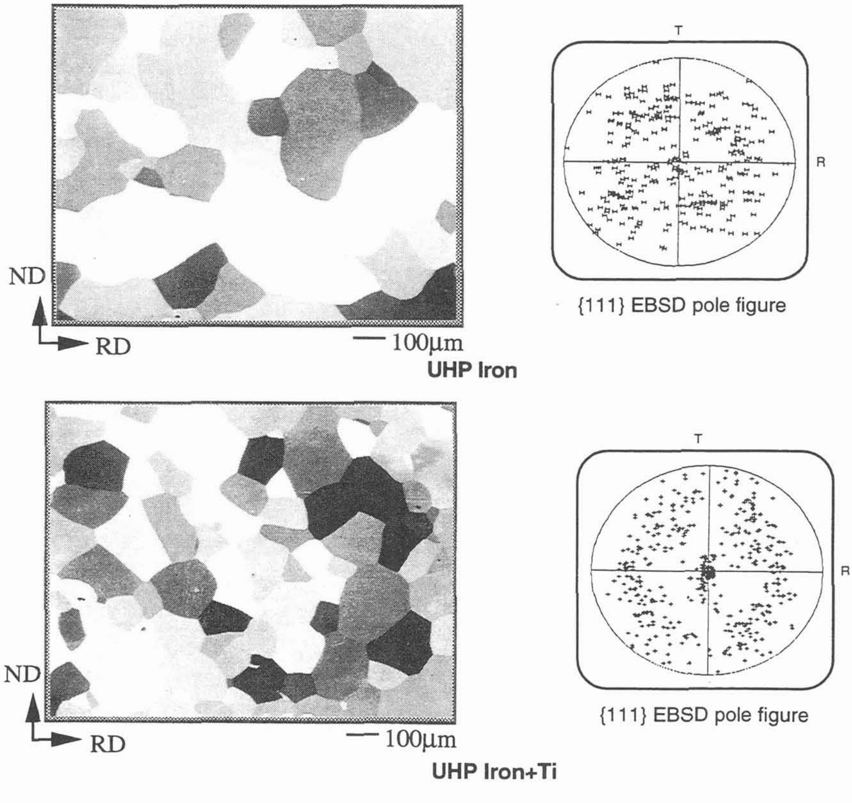


Figure 5 : The final microstructure and {111} pole figures after complete recrystallization

It is clear that the relative strengths of the {111} and {100} components are virtually independant of the type of treatment, whether short times at relatively high temperatures or long periods at temperatures about 100°C lower. The important result is the strong influence of the solid solution alloying elements, both interstitial (C in electrolytic iron) and substitutional (Nb and Ti) in increasing the final proportions of the {111} sheet plane component compared with UHP Fe. The role of carbon seems to be in conflict with many results on IF steels (see for example Henry et al. [8] and the review by Hutchinson [9], which indicate that the {111} component, and the consequent high r values, is increased at low carbon contents. However, these previous studies did not examin carbon contents less than 10 ppm as in the present work on UHP Fe. Taking the present results on UHP Fe at < 1 ppm C together with previous studies at low carbon steels (≤100 ppm C) we conclude that C in the range 1 - 100 ppm has a strong influence on the recrystallization nucleation mechanisms and the final texture. The other point to emerge is that both Nb and Ti in solid solution also favour the nucleation of {111} oriented grains although Ti appears to be more effective. Since nucleation occurs principally along the grain boundaries, it is clearly the behaviour of these solid solution elements in the boundary regions which controls the texture evolution and therefore the drawability of these IF steels.

4. Conclusions

The recrystallization mechanisms of high purity iron (UHP Fe and electrolytic iron) and two UHP Fe based alloys (Fe - Nb, Fe - Ti) have been investigated after cold rolling and annealing by detailed micro texture analysis. The principal results can be summarized as follows :

- the rolling and recrystallization textures of these HP materials are quite similar to those of industrial IF steels ; partial α fibre and complete γ fibre in the as - rolled state and retained γ fibres particularly {111}, together with some {100} sheet plane components after recrystallization.
- Nb and Ti in solid solution increase the recrystallization temperature of UHP Fe by 200 and 150°C respectively,
- the first nuclei, usually along grain boundaries, have {111} plane orientations in electrolytic iron but both {111} and {100} in UHP Fe with or without Nb or Ti additions.
- the nucleation and growth of the {111} component is favoured by C, Ti and Nb additions, in order of decreasing importance, compared with UHP iron.
- the cube oriented grains appear to recover rapidly but are often the last to recrystallize.

5 Reference

1- H. Inagaki *"Fundamental Aspect of Texture Formation in low Carbon Steel"* ISIJ, **Vol 34**, pp 313 - 371 (1994)
2 - R. E. Houck, A. J. Hecler, and J. A. Elias, *"Texture in Deep Drawing Colombium - Treated Interstitial-Free Steel"*, Met. Trans. **Vol. 6A** N° 10, pp 1683-1692, (1975).
3 - H. Katoh, H. Takechi, N. Takahashi, and M. Abe, *"Cold Rolled Steel Sheets Produced by Continuous Annealing"* Ref. 4, pp 37-60.
4 - D. Schlafer and H. J. Bunge, *Texture 1*, 157 (1974).
5 - W. B. Hutchinson *"Recrystallization Textures in Iron Resulting from Nucleation at Grain Boundaries"* Acta. Metall. **Vol 37** N° 4 pp 1047-1056, (1989).
6 - "Experimental studie of nucleation in irons" - M. Kiaei, B. Bacroix and J. H. Schmitt 37em Colloque INSTN (1995)
7 - E. Lindh, B. Hutchinson and P.Bate *"Texture evolution during recrystallization of ultra low carbon steel"* Material Science Forum, **Vol 157 - 162**, pp 1997 - 1002 (1994)
8 - J. Henry, J. Copreaux, N Lorenzelli, et A. Barbu - *37ème Colloque de Métallurgie de l'INSTN* Saclay (21-23 Juin 1994).
9 - B. Hutchinson *"Practical aspect of texture control in low carbon steels"*, Material Science Forum **Vol 157 - 162**, pp 1917 - 1928 (1994).