CHARACTERIZATION OF RECRYSTALLIZATION TEXTURES IN Fe-3 % Si SHEETS BY EBSP : COMPARISON WITH X RAY DIFFRACTION

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I - INTRODUCTION

A first study relative to evolution of the local texture and of the nature of the grain boundaries during abnormal growth of Goss grains in Fe 3 % Si sheets, grade Hi-B ⁽¹⁻⁵⁾ has pointed out that at the primary recrystallized state, i.e. after decarburization, the Goss grains belong to small grain class, that their volume fraction determined by micro etch pit technique is very low and that apparently they are randomly distributed through the sheet thickness.

Using, X-ray diffraction , EBSP and SIMS, the aim of the present work was to specify mechanisms which lead to abnormal growth of Goss grains.

By X-ray diffraction, the gradient of the primary recrystallization texture was quantitatively characterized from incomplete pole figures ⁽⁶⁾ ; however with this technique, only the even part of the Orientation Distribution Function, ODF, is attainable.

By EBSP, the local texture, notably, spread of Goss grains through the sheet thickness, nature of grain boundaries which is a determining parameter for their migration, especially those between a Goss grain and its first neighbours were determined. With this technique it is finally possible to calculate the total texture function and so to measure the importance of the odd part of the ODF as well as the possible presence of "ghosts" ⁽⁷⁾.

Finally by SIMS, influence of the chemical parameter and more especially that of growth inhibitors Al N and Mn S upon abnormal growth was specified.

II - CHARACTERIZATION OF THE PRIMARY RECRYSTALLIZATION TEXTURE

During the final annealing of secondary recrystallization which is performed under hydrogen atmosphere, it was shown that Goss grains expand in subsurface, between the fourth and the fifth of sheet thickness. Some authors ⁽⁸⁾ have then suggested that "the Goss nuclei" were only located at the fifth of the thickness and that their origin would be related to the shear stress component during rolling ; moreover their size would be larger than that of the other grains of the matrix, in other words they would benefit from a size advantage which would allow them to grow in an exaggerated way. From a metallographic study, it was shown a total absence of large grains in subsurface : so research was done to determine if a preferential distribution of Goss grains was present through the thickness of the studied sheets.

Hence the primary recrystallization texture was determined in surface, at the fifth and in the middle of the sheets by X-ray diffraction and by EBSP, in this last case orientation of 1000 grains was characterised for each depth.

II.1 - X-ray diffraction study

Determination of the texture of our material was obtained from X-ray diffraction experiments in reflection up to $\chi = 80^\circ$ (χ = radial angle). The ODF calculation was performed from three incomplete pole figures {110}, {200} and {112} by a method described everywhere else by BAUDIN et al ⁽⁶⁾. In order to perform this calculation, the BUNGE ⁽⁹⁾ and ROE ⁽¹⁰⁻¹¹⁾ method which uses series expansions on spherical harmonic basis was chosen as well as the ROE notation for the EULER angles.

X-ray diffraction however presents a major disadvantage because it only allows to calculate the even part $\tilde{F}(g)$ of this texture function $F(g)$, the odd part $\tilde{\tilde{F}}(g)$ not being directly determinable ^{(7),(9),(12)}.

The figures 1 a,b,c allow to compare $\tilde{F}(g)$ calculated in the plane (ψ, θ) for $\varphi = 45^\circ$, respectively for the samples studied in surface, at the fifth and at the center.

Figure 1 : Sections at $\varphi = 45^\circ$ of the even part $\tilde{F}(g)$ of the ODF (X-ray diffraction) ; a) at the surface, b) at the fifth, c) at the center of the sheet at the primary recrystallized state.

For each case, texture showed two main preferential orientations :

- $\{111\} \langle 112 \rangle$ for which $\tilde{F}(g)$ increases from the center of the sheet to the surface ($\tilde{F}(g) \approx 8$ to 10).
- $\{100\} \langle 012 \rangle$ for which $\tilde{F}(g)$ decreases from the center to the surface ($\tilde{F}(g) \approx 5$ to 3).

Finally it is important to underline that with the X-ray diffraction technique, the Goss component $\{110\} \langle 001 \rangle$ was not detected.

II.2 - EBSD study

This technique, notably developed by DINGLEY ⁽¹³⁾ is especially interesting because it allows to determine the local texture, the spatial resolution being about 2000 Å. It permits the estimation of the number of Goss orientation among the grain population of the studied surface ; for each depth 1000 orientations were determined. Then it is worth noting that only one Goss grain, among the 3000 studied, was found in the middle of the sheet thickness and not at the fifth. So it is obvious that the volume fraction of the Goss grains is very low and that moreover they are randomly distributed through the sheet thickness, contrary to the current assumption.

The technique of individual orientation measurements points out another advantage because it allows one to calculate a total texture function $F(g)$.

Qualitative comparison of $\{111\}$ pole figures, on the surface, at the fifth and at the center of the sheets reveals that the spread of the $\{111\}$ poles about the normal to the rolling plane is less important on the surface than at the fifth and at the center. This observation is in agreement with the previous results obtained by X-ray diffraction.

It is also interesting to compare the pole figures recalculated from X-ray diffraction measurements and measured by EBSD, figure 2. An agreement is observed between these different figures which shows on one hand the quality of these measurement techniques, even if they are very different and on the other hand the good statistics obtained for 1000 individual orientations.

Figure 2 : {200} pole figures obtained by X-ray diffraction and by EBSD.

III - PRINCIPLE OF THE TEXTURE FUNCTION CALCULATION

The texture function $F(g)$ can be divided in a sum of a part directly determinable from pole figures, $\tilde{F}(g)$ and of a part not directly determinable $\tilde{\tilde{F}}(g)$, with

$$F(g) = \tilde{F}(g) + \tilde{\tilde{F}}(g) \quad (1)$$

For materials with centro-symmetrical grains, $\tilde{F}(g)$ corresponds to the spherical harmonic expansion with ℓ even, ℓ_e and $\tilde{\tilde{F}}(g)$ to spherical harmonic expansion with ℓ odd, ℓ_o .

$$\tilde{F}(g) = \sum_{\ell_e} \sum_m \sum_n f_{\ell mn} T_{mn}^{\ell} (g) \quad (2)$$

$$\tilde{F}(g) \approx \sum_{\ell_o} \sum_m \sum_n f_{\ell mn} T_{mn}^{\ell} (g) \quad (3)$$

where the $T_{mn}^{\ell} (g)$ are generalized spherical harmonics.

From N orientations g_i , the coefficients $f_{\ell mn}$ of these expansions can be calculated using the relation proposed by BUNGE ⁽⁹⁾ :

$$f_{\ell mn} = \frac{1}{N} \sum_{i=1}^N K T_{mn}^{\ell} (g_i) \quad (4)$$

with

$$K = \frac{\exp(-\ell^2 \phi_0^2 / 4) - \exp[-(\ell+1)^2 \phi_0^2 / 4]}{1 - \exp(-\phi_0^2 / 4)} \quad (5)$$

Expression of the K parameter proposed by WAGNER ⁽¹⁴⁾ allows to model each orientation by a gaussian of ϕ_0 parameter related to the total width at half height, b by the relation :

$$\phi_0 = b / 2 \sqrt{\ln 2} \quad (6)$$

Then the problem is to estimate b or ϕ_0 ; WAGNER proposed to approximate ϕ_0 with the help of the following relation :

$$\phi_0 \approx (2 \pi p/N)^{1/3} \quad (7)$$

where p is a fitting parameter characterizing the sharpness of the texture. If the texture is isotropic, p is equal to 1 whereas p tends towards zero for a

sharp texture. In general, in the case of medium textures, a value of 0.5 is chosen ^(14,15), that is $\phi_0 \approx 8.5^\circ$ for 1000 orientations.

So it is difficult to estimate in a precise way ϕ_0 and then the sharpness of the texture ; in our case and taking into account results obtained by X-ray diffraction, $p \approx 0.6$ that is $\phi_0 \approx 10^\circ$ was arbitrarily chosen.

In the case of sample cut off in surface, the figures 3 a,b,c respectively show, the texture functions, even $\tilde{F}(g)$, odd $\tilde{\tilde{F}}(g)$ and total $F(g)$ in the plane (ψ, θ) for $\phi = 45^\circ$. It is worth noting the similarity of the $\tilde{F}(g)$ functions calculated from X-ray diffraction measurements or from EBSP. This result points out the validity of the measurement statistics. However, it would be necessary according to the sharpness of the texture to optimize the number of measurements as WRIGHT and ADAMS ⁽¹⁵⁾ did for an aluminium sheet.

Figure 3 : Sections at $\phi = 45^\circ$ of a) $\tilde{F}(g)$, b) $\tilde{\tilde{F}}(g)$ et c) $F(g)$ of the surface by EBSP.

In a general way and whatever the studied sample, a decreasing evolution of the texture sharpness is observed from the surface to the center for the main orientation $\{111\} \langle 112 \rangle$. The weight of $\tilde{F}(g)$ corresponds approximatively to 15-20 % of $F(g)$.

The figures 4 a,b,c enable us to compare the total texture function $F(g)$ respectively calculated using samples cut off in surface, at the fifth and at the center. However few negative domains, not represented because negligible, remain, they are probably related to truncation errors ($\mathcal{L} = 22$).

Figure 4 : Sections at $\phi = 45^\circ$ of the total ODF $F(g)$, a) at the surface, b) at the fifth, c) at the center.

From these figures, it can be observed that the sharpness of the orientation $\{100\} \langle 012 \rangle$ increases from the surface to the center ; this result had already been observed on figure 1 which represents $\tilde{F}(g)$ calculated from pole figures measured by X-ray diffraction.

ROUAG ⁽¹⁾ in her thesis observed that the $\{111\} \langle 112 \rangle$ orientation at $\varphi = 40^\circ$ is divided for the sample at the sheet center. This result is not explained and it could be thought that it is the consequence on one hand of a bad measurement and on the other hand of the non determination of $\tilde{F}(g)$. But the figure 5 shows that it is not the case because this separation is found again for $\tilde{F}(g)$ calculated from X-ray diffraction and EBSP. It is also observed for $\tilde{F}(g)$ and then for $F(g)$.

Figure 5 : Sections at $\varphi = 40^\circ$ of a) $\tilde{F}(g)$, X-rays, b) $\tilde{F}(g)$, EBSP, c) $\tilde{F}(g)$, EBSP, d) $F(g)$, EBSP.

IV - INFLUENCE OF THE GRAIN BOUNDARY NATURE AND OF THE MATERIAL CHEMISTRY

During the final annealing of secondary recrystallization, it was shown ^(1,4) that the first steps of the abnormal growth are controlled in presence of inhibitors by the speciality of grain boundaries, indeed grains belonging to the

main component $\{111\} \langle 112 \rangle$ are consumed first by Goss grains whereas those of the secondary component $\{100\} \langle 012 \rangle$ disappear last.

This observation shows that there is a difference of mobility between the grain boundaries which separate on one hand the Goss grain from the $\{111\} \langle 112 \rangle$ grains and those which, on the other hand, separate the Goss grain from the $\{100\} \langle 012 \rangle$ ones and from the grains belonging to the random. Then it can be noticed that the misorientation between $\{110\} \langle 001 \rangle$ and $\{111\} \langle 112 \rangle$ corresponds to a coincidence boundary $4^\circ \Sigma 9$ whereas the misorientation between $\{110\} \langle 001 \rangle$ and $\{100\} \langle 012 \rangle$ corresponds to a general boundary. The CSL coincidence boundaries would be for the studied material, more mobile than the other boundaries at the beginning of the secondary recrystallization. This assumption is confirmed by the fact that the percentage of the CSL boundaries surrounding a Goss grain decreases from 15 to 5 %.

In the presence of Al N and Mn S, these CSL boundaries are more mobile than the general ones because they are less dragged by precipitates and possible segregations ; later growth is controlled by the size factor.

From these results, it appears that the presence of a favorable crystallographic neighbourhood $\{111\} \langle 112 \rangle$ in the present case increases the probability of having CSL boundaries in the Goss orientation. So it is necessary to look for the most appropriate matrix to the development of the desired orientation ; thus ROUAG ^(1,16) has simulated the domains of the Euler space favourable to the growth of such grains ; the texture of the material operates as a weight function introducing a presence probability of the CSL domains of this space. ROUAG ⁽¹⁾ has simulated these domains by embedding a Goss grain in the experimental primary matrix.

diagram at $\phi = 45^\circ$, figure 6 b, shows that the $\Sigma 9$ boundaries coincide with the maximum of the ODF, $\tilde{F}(g)$ of the main component $\{111\} \langle 112 \rangle$.

Figure 6 : a) CSL domains and maximums of $\tilde{F}(g)$ at $\phi = 0^\circ$,
b) CSL domains and maximums of $\tilde{F}(g)$ at $\phi = 45^\circ$, from ROUAG¹.

So it can be seen that this simulation is coherent with experimental results and that it allows us to define the good matrix which is necessary to the growth of a given orientation $\{hk\ell\} \langle uvw \rangle$. However this geometrical criterion

of speciality is not sufficient. It is necessary to take into account the role of the material chemistry and the energy of the boundary ; this last parameter was neglected in this study.

By SIMS, it has been pointed out, due to the hydrogen atmosphere, that the material is refined in subsurface during the final annealing ; in fact an impoverishment in nitrogen and in sulphur was observed. These elements migrate towards the surface under the action of the gradient of chemical potential related to the presence of the hydrogen atmosphere. Moreover at the industrial scale, the sheets are coated by a Mg O film which improves aluminium migration towards the surface.

The purification of the material being faster in subsurface than at the center, it becomes obvious that the Goss grains located in this zone grow more rapidly than those located at the center of the sheets. If the final annealing is performed under an Argon inert atmosphere, secondary recrystallization occurs, which is in agreement with the role played by the CSL boundaries, but the grain size is limited because of the presence of impurities which drag the grain boundaries. In this case a great part of the primary matrix remains after this annealing since the magnetic quality of these sheets is bad.

V - CONCLUSION

Thanks to the EBSP technique it has been possible to calculate :

- the total texture function $F(g)$,
- the even $\tilde{F}(g)$ and odd $\tilde{\tilde{F}}(g)$ parts of the true ODF, $F(g)$.

It has been shown that the odd part represents approximately 20 % of the total texture function ; finally the primary recrystallization texture not being very strong, no ghost has been detected.

It is worth noting for the even part a good coherence between the results of X-ray diffraction and those of EBSP.

EBSP allowed us to estimate the evolution of the percentage of CSL boundaries during the final annealing ; it showed that the volume fraction of Goss grains is very small (less than 10^{-3}) and that these grains are not located at the fifth of the sheet thickness.

As it has been shown the onset of the abnormal growth is controlled by the speciality of the boundaries, which means that "the Goss grain" has to be surrounded by the good primary matrix ; however it is obvious that this geometrical criterion is insufficient because the plane of the grain boundary has been neglected in this first attempt. It should be noted that the purification of the material is also an essential parameter to perfection of the Goss texture.

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