Proceedings of the 6th International Conference on Recrystallization and Grain Growth (ReX&GG 2016) *Edited by: Elizabeth A. Holm, Susan Farjami, Priyadarshan Manohar, Gregory S. Rohrer, Anthony D. Rollett, David Srolovitz, and Hasso Weiland*
TMS (The Minerals, Metals & Materials Society), 2016

NUCLEATION STAGE DURING IF STEEL RECRYSTALLIZATION AND INTERNAL MISORIENTATION PARAMETERS

A. Ayad[1,2], M. Ramoul[2], N. Rouag[2], A.D. Rollett[3] and F. Wagner[4]

[1]Département de pharmacie, Faculté de Médecine, Université Constantine 3, Nouvelle ville Ali Mendjeli, BP. 67A Constantine, Algeria.
[2]Laboratoire de microstructures et défauts dans les matériaux, Université Frères Mentouri Constantine 1, Alegria.
[3]Department of Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, PA 15213, USA.
[4]LEM3, (CNRS-UMR 7239), Université de Lorraine, Ile du Saulcy, 57045 Metz, France.

Abstract

Several misorientation parameters deduced or calculated from EBSD data can be used to follow the evolution of microstructure in deformation and recrystallization such as: BC (Band Contrast), GOS (Grain Orientation Spread), GND (Geometrically Necessary Dislocation density) and KAM (Kernel Average Misorientation). The KAM of a pixel is the mean value of misorientation between this pixel and its neighbors in a grid of (NxN) pixels in grain interior. In this work, we propose a modified KAM to characterize recrystallization in an IF steel, especially the nucleation stage. It is well known that nucleation starts in grains that have the highest stored energy of deformation, and then progresses in grains that have lower stored energy. The modified KAM value is quite sensitive to the state of the material. This presentation will discuss the relation between the modified KAM values, orientation gradients and the stored energy, as well as the possibility of using these values to identify nucleation sites.

Keywords: KAM, stored energy, nucleation, recrystallization, IF steel.

Introduction

Development of annealing texture in low carbon steels provides a high level of scientific interest. It has been widely studied in several papers [1-3]. Despite the effort dissipated, the subject still involves many unresolved questions regarding the complexity of microstructural inhomogeneity formed in the deformed state, estimation of stored energy, orientations of nuclei, nucleation rates for different orientations of deformed grains and different growth rates of nuclei into the deformed matrix. The progress that has been made in the Orientation Imaging Microscopy (OIM) has contributed significantly to the understanding of the recrystallization texture in low carbon and interstitial free (IF) steels. Currently, several misorientation parameters deduced from EBSD data are used to follow the evolution of microstructure in deformation and recrystallization such as: Grain Orientation Spread (GOS), Kernel Average Misorientation (KAM), Grain Average Misorientation (GAM) [4-8]. Geometrically Necessary Dislocation density (GND) can be also calculated from local misorientations [7, 8].
In this paper, we propose a modified KAM parameter in order to characterize recrystallization texture in an IF steel, especially the nucleation stage.

In general, a nucleus is a small area with few defects in a grain with much defects (i.e. with a high stored energy) ; the grain boundary pressure depends on the difference between the stored energies in the nucleus and in its vicinity. But to grow the nucleus needs also to have borders (boundaries) with a sufficient misorientation versus the neighboring areas which insures a sufficient mobility [9]. The relation between this internal misorientation parameter and the stored energy, as well as the possibility of using it to identify nucleation sites will be discussed.

Modification of the KAM

First, let us recall the definitions of certain internal misorientation parameters used in this work, in particular GOS and KAM. The GOS for a grain is the mean value of misorientations between all pixels of the grain and the average orientation of the grain. However, a KAM value for a pixel is obtained by averaging the misorientations between it and each of its first neighboring pixels in the same grain of this pixel. The modification to KAM that we propose in this paper is to not avoid calculation of misorientation between two pixels which do not belong to the same grain.(i.e. consideration of misorientations between pixels located on either side of the grain boundary).
The annealing texture of IF steels with moderately cold rolling reductions is controlled by the orientation of nuclei [10]. The dominant nucleation process is the formation of γ-fiber grains in highly deformed regions of γ-fiber orientations. This occurs near prior grain boundaries [11-14] and inside the deformed grains at deformation bands [15]. Regions near grain boundaries and triple points present a higher degree of orientation variation and higher dislocation density compared with the interiors of the grains [12, 16]. The increased dislocation densities and orientation variation in these grain boundary regions makes them potent sources of nuclei [17]. In addition, subgrains having a high stored energy and high angle grain boundaries are the most likely nucleation sites [9]. The modification of the KAM is proposed for better taking into account these changes in the vicinity of the grain boundaries and to adjust the stored energy in these potential nucleation sites.

Material and characterization techniques

Table I shows the chemical composition of the IF steel used in this work. This IF steel was cold rolled up to 75% thickness reduction (final thickness 0.7mm). For the recrystallization analysis, 75% cold rolled samples were annealed at 630°C for several durations. A previous studies shows that the primary recrystallization is nearly finished after 180 mn [18-19].

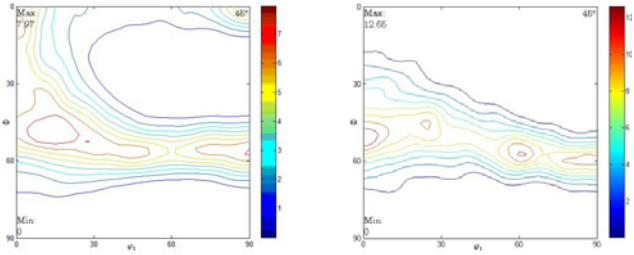
Table I. Chemical composition of the steel under study.

Element	C	Mn	P	S	N	Si	Cu	Ni	Cr	Al	Ti	Fe
%wt 10^{-3}	8	196	4	10	3.1	4	7	18	14	41	97	bal.

The specimens were ground mechanically with SiC grit paper followed by mechanical polishing using 3 and 1 μm diamond suspension, respectively. For EBSD analysis, samples were further electropolished. EBSD analyses were carried out in a JEOL 6500F field emission gun scanning electron microscope (FEG-SEM). EBSD mappings were visualized and post-processed using the HKL Channel 5 and MTEX software [20].

Evolution of texture during recrystallization

The ODFs of the 75% cold rolled and at the end of primary recrystallization states, have been calculated from the individual orientations of the EBSD maps. ODFs calculations have been made assuming cubic-orthorhombic symmetries. The main features appear in the φ_2=45° section which are shown in Figure 1.
It is observed that the texture after cold rolling consists of the α-fiber and γ-fiber orientations with an almost uniform intensity along these two fibers (Figure1a). The maximum intensity is located in γ-fiber with a large spread. It should be noted the presence of a secondary maximum on the {001}<110> component of the α-fiber.
For the recrystallized sample (630°C, 180 mn), the texture consists mainly of a strong γ-fiber with a relatively large spread around it, especially in the α-fiber direction (i.e. for φ_1=0°). Inside this γ fiber, the density is approximately evenly distributed with a slight peak around the orientation {111}<110> (Figure 1b).



(a) (b)

Figure 1. φ2 = 45° sections of the ODF for: (a) 75% cold rolled state ; (b) Fully recrystallized state (630°C, 180 mn).

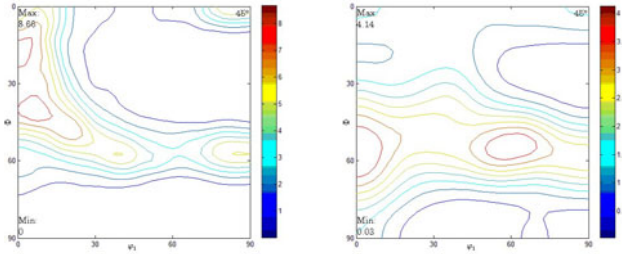
Correlation between annealing texture and stored energy

Recrystallization consists of two stages, nucleation and growth, and two main theories of the texture development are considered [2, 9, 21, 22]:

- Oriented nucleation: the final texture of the material depends primarily on the orientation of the nuclei, and that the nuclei cannot have all orientations but depend on the deformed matrices.
- Oriented (selective) growth: nuclei with a certain misorientation relation to the surrounding matrix will grow faster and control the final texture.

In order to understand the texture evolution during recrystallization in the 75% cold rolled IF steel and an eventual role of oriented nucleation, the microstructure and texture were analyzed in early stage of recrystallization (630°C, 5 mn). By using a severe criterion GOS ≤ 0.5° to partition the recrystallized grains (nuclei), the recrystallized volume fraction is ~ 4%.
The global texture and the nucleation texture (only orientation of nuclei) of this sample are shown in Figure 2. When comparing texture at early stage of recrystallization (Figure 2a) with the deformed state (Figure 1a), it is evident that they display similar morphologies, although the

intensities are different. However, texture of nuclei shows a strong γ-fiber (Figure 2b), it is qualitatively similar to that of the recrystallized state (Figure 1b). Therefore texture development in this case can be considered to be nucleation controlled (oriented nucleation).

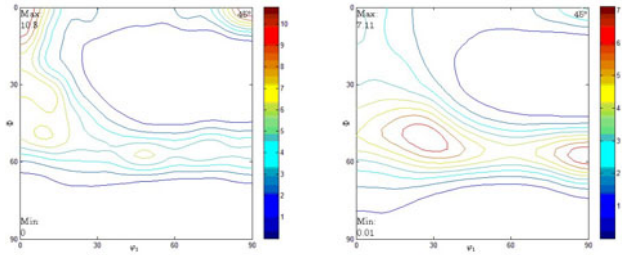


(a) (b)

Figure 2. φ2 = 45° sections of the ODF for the early recrystallization stage (630°C, 5 mn) showing: (a) the global sample ; (b) texture of nuclei (GOS≤0.5°).

Most theories of oriented nucleation in steels are based on the high stored energy block concept, where subgrains with the highest stored energy correspond to the ones which recrystallize first during annealing [21]. Furthermore, it is widely accepted that high stored energy nucleation generally leads to {111} oriented nuclei, therefore, the γ-fiber grains are favored for nucleation [2, 12, 21]. Confirmation for this point is seen in the clear similarity between the early texture of recrystallized grains and their final texture.

When considering the textures of pixels having the lowest values of the modified KAM (representing 25% of the total number of pixels) and pixels with the highest values of the modified KAM (representing also 25% of the total number of pixels) of the 75% cold rolled sample (Figure 3), it appears that the orientations of high KAM pixels are more concentrated in the γ-fiber and the maximum intensity of the ODF corresponds to {111}<112> component (Figure 3b), whereas the orientations of lowest KAM pixels are near homogenously distributed around γ-fiber and more concentrated on α-fiber showing reinforcement on {001}<110> component (Figure 3a). This probably leads to the texture evolution seen previously in the experimental study, due to nucleation of orientations belonging to γ-fiber which based on the high stored energy mechanism. Indeed, it has been shown that nuclei are oriented γ-fiber and the deformed area with γ-fiber orientations is consumed first then the deformed α-fiber which tends to persist to the end of recrystallization [21]. The texture is usually a strong γ-fiber, after complete recrystallization. Thus, the modified KAM can be used to describe semi-quantitatively the distribution of the stored energy in a deformed matrix and to detect nuclei which could grow during recrystallization.



(a) (b)

Figure 3. φ2 = 45° sections of the ODF for the 75% cold rolled state: (a) 25% of pixels with lowest modified-KAM values; (b) 25% of pixels with highest modified-KAM values.

Conclusion

In this paper, a misorientation parameter deduced from EBSD data is used as an semi quantitative indicator for the stored energy level in a 75% cold rolled IF steel. It consists of a modification of the KAM that does not exclude misorientation between neighbors located on either side of the grain boundary. The motivation of this modification is the consideration of the orientation gradient, and the orientation variation as well as the higher dislocation density in regions near grain boundaries compared with the interiors of the grains.

By comparing texture of the deformed state, especially that of pixels with high modified-KAM values and textures of early stage of nucleation and fully recrystallized states, it was found that nucleation consists mainly of γ-fiber orientations that possess a relatively high values of the modified KAM (i.e., high stored energy). Therefore, KAM is a good parameter to detect nuclei which could develop during recrystallization of IF steels.

References

1. I. Samajdar, B. Verlinden, L. Kestens and P. Van Houtte, *Acta mater.*, 47 (1) (1999), 55.
2. K. Verbeken, L. Kestens, J.J. Jonas, *Scripta Mater.*, 48 (2003), 1457.
3. B. Radhakrishnan, G.B. Sarma, *Mater. Sci. and Eng. A*, 494 (2008), 73.
4. A.J. Schwartz et al., *Electron Backscatter Diffraction in Materials Science*, (New York, Springer, 2009).
5. M.H. Alvi, S. Cheong, J.P. Suni, H. Weiland, A.D. Rollett, *Acta Mater.*, 56 (2008), 3098.
6. R.S. Yassar, J.C. Baird, M.F. Horstemeyer, *Mater. Sci. and Eng. A*, 517 (2009), 286.
7. M. Calcagnotto, D. Ponge, E. Demir, D. Raabe, *Mater. Sci. and Eng. A,* 527 (2010), 2738.
8. D.P. Field, L.T. Bradford , M.M. Nowell , T.M. Lillo, *Acta Mater.*,55 (2007), 4233.
9. F.J. Humphreys, M. Hatherly, *Recrystallization Related Annealing Phenomena*, (Oxford, Pergamon Press, 1995).
10. L. Kestens, J.J. Jonas, *ISIJ Int.*,37 (1997), 807.
11. M. Abe, Y. Kokabu, Y. Hayashi, S. Hayami, *Trans. Jpn. Inst. Met.*,23 (1982), 718.
12. H. Inagaki, *Trans. Jpn. Inst. Met.*, 28 (1987), 251.
13. W.B. Hutchinson, *Acta Metall.*, 37 (1989), 1047.

14. M.R. Barnett, L. Kestens, *ISIJ Int.*, 39 (1999), 923.
15. Y.Y. Tse, G.L. Liu, B.J. Duggan, *Scripta Mater.*, 42 (2000), 25.
16. V. Randle, N. Hansen, D. Juul Jensen, *Phil. Mag. A*, 73 (1996), 265.
17. M.D. Nave, M.R. Barnett, Mater. *Sci. and Eng. A*, 386 (2004), 244-253.
18. A. Ayad, N. Allain-Bonasso, N. Rouag, F. Wagner, *Mater. Sci. For.*,702-703 (2012), 269.
19. A. Ayad, F. Wagner, N. Rouag, A.D. Rollett, *Comp. Mater. Sci.*, 68 (2013) 189.
20. F. Bachmann, R. Hielscher, H. Schaeben, "Texture Analysis with MTEX-Free and Open Source Software Toolbox", *Solid State Phenom.*, 160 (2010), 63.
21. H. Magnusson, D. Juul Jensen and B. Hutchinsson, *Scripta mater.*, 44 (2001), 435.
22. P. Lin, G. Palumbo, J. Harase, K.T. Aust, *Acta Mater.*, 44(1996), 4677.