Materials Science Forum
ISSN: 1662-9752, Vols. 467-470, pp 1295-1300
doi:10.4028/www.scientific.net/MSF.467-470.1295

Online: 2004-10-15

Structural Evolution of IF Steel during Annealing after ECAP

Joke De Messemaeker, Bert Verlinden and Jan Van Humbeeck

Katholieke Universiteit Leuven, Department of Metallurgy and Materials Engineering, Kasteelpark Arenberg 44, B-3001 Heverlee, Belgium

Keywords: Ultrafine grained material, continuous recrystallization, equal channel angular pressing (ECAP), interstitial free (IF) steel.

Abstract. IF steel processed by equal channel angular pressing to an equivalent strain of 9.2 via route B_A was annealed for different times at 500°C and 600°C. At both temperatures the microstructural evolution shows continuous recrystallization followed by grain growth, in absence of primary recrystallization. At 600°C a slightly bimodal grain size distribution develops.

Introduction

This paper reports on a study of the thermal behavior of IF steel after severe plastic deformation to an equivalent strain of 9.2. Knowing this behavior is interesting both from a fundamental point of view as for possible applications of this ultrafine structured material, in case the deformation substructure can be converted into an ultra-fine grain structure during annealing [1]. The IF steel in this study was processed by equal channel angular pressing (ECAP) to a plastic strain of 9.2. Route B_A was chosen, as the microstructural development of IF steel during ECAP via this route was previously studied by the authors [2].

The as-deformed material was annealed for different times at 500°C and 600°C. Quantitative information regarding the microstructure after annealing was obtained by EBSD measurements. The minimum heat treatment required to obtain diffraction patterns of acceptable quality was 3h at 500°C.

Experimental

The hot-rolled Ti-stabilized IF steel used for this study had an equiaxed grain size of about 100µm. It was processed by ECAP in order to induce severe plastic deformation. A description of the ECAP process can be found in [3]. In ECAP the deformation occurs by simple shear parallel to the intersection plane of the two channels comprising the die, consequently termed the shear plane. The die used in this study has a channel intersection angle of 90°. Route B_A was used, meaning that the sample was rotated in between two passes over 90° alternatively clockwise and counterclockwise. This corresponds to a series of orthogonal changes of strain path [4], where the amount of strain in each continuous stage of the path is 1.15. In total 8 passes were applied, leading to a total equivalent strain of 9.2.

The useful volume of the ECAP samples was cut into smaller samples for annealing in a salt bath furnace at 500°C and 600°C respectively. Annealing times at 500°C were 3h, 24h, 72h and 128h, and at 600°C they were 5min, 15min, 2h, 15h and 100h.

TEM and EBSD measurements were used for microstructural observations and quantitative microstructural analysis respectively. All observations were made on the plane of plane strain of the last pass, i.e. the plane parallel to the symmetry plane of the ECAP die.

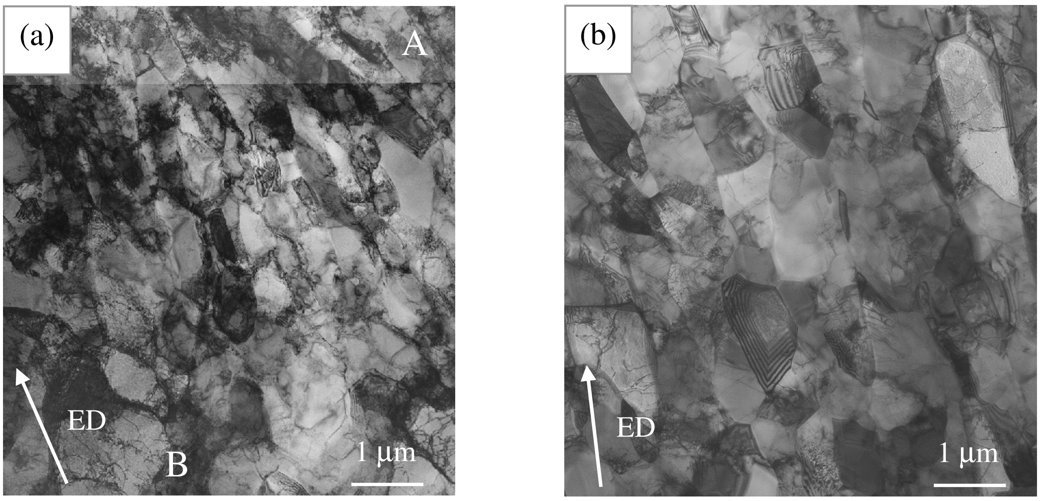

Figure 1: TEM micrographs of IF steel after 8 ECAP passes via route B_A (a) as-deformed and (b) annealed for 3h at 500°C. Micrograph (a) illustrates the microscopic heterogeneity between the substructure in a shear band (partly visible in area A) and beside it (area B). The viewing plane is the plane of plane strain of the last ECAP pass. ED is the extrusion direction of the last pass.

Description of the As-deformed State

The evolution of the microstructure of IF steel during processing by ECAP via route B_A has been discussed in a previous report [2]. The most important features of the microstructure after 8 passes will be briefly recapitulated.

The substructure generally consists of parallel extended planar dislocation sheets lying perpendicular to the plane of plane strain, at an angle of ~ 10° to the extrusion direction. Between the dislocation sheets less distinct transverse dislocation boundaries are formed. The mean linear intercept (MLI) perpendicular to the dislocation sheets is about 300 nm. A prominent feature of the microstructure are wide, loosely defined shear bands, several 100µm long, spaced a few 100 µm apart, and inclined at 35° to the extrusion direction. Within these shear bands the extended dislocation sheets are more closely spaced (area A in Fig. 1a), and adjacent to the shear bands, dislocations are arranged in a rounded, relatively large cell structure (area B in Fig. 1a). From EBSD

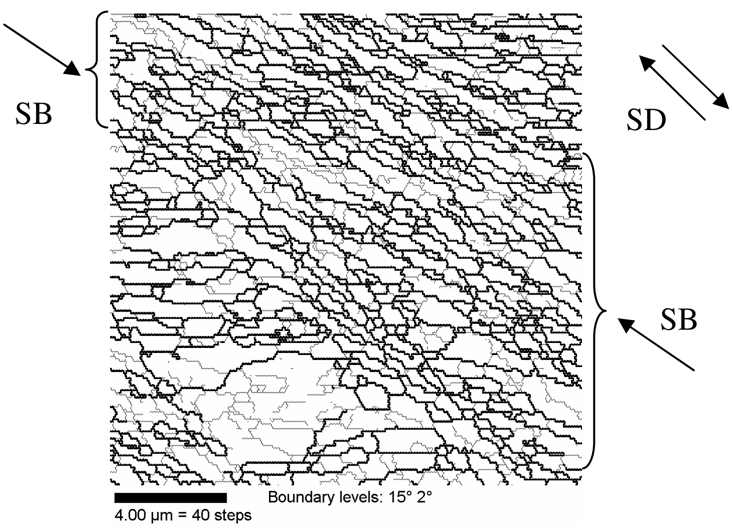

Figure 2: Boundary map with low angle boundaries (2°-15°, thin black line) and high angle boundaries (15° or higher, bold black line) for IF steel after 8 passes via route B_A and a subsequent anneal of 3h at 500°C. Note the structure differences inside the shear band (SB) and just beside it. SD is the shear direction of the last pass.

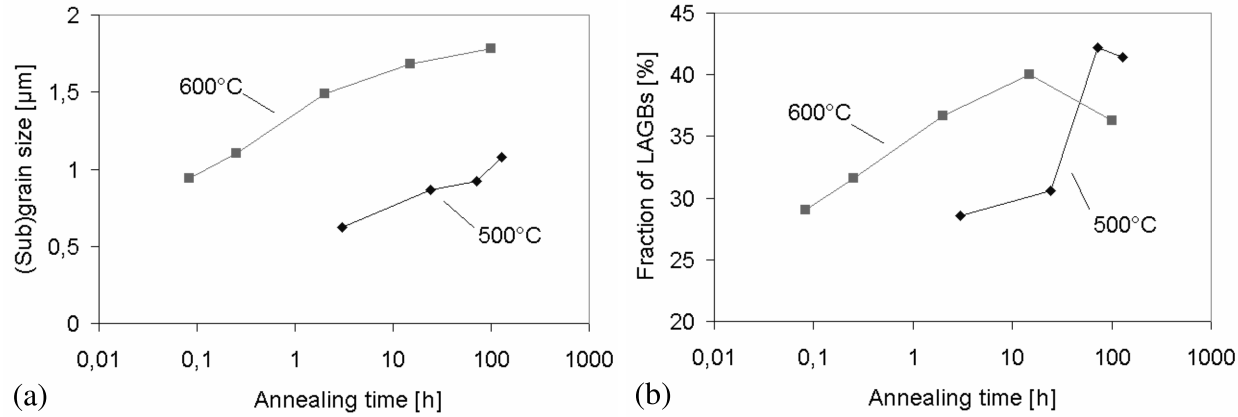

Figure 3: Evolution of the (sub)grain size (a) and the fraction of low angle boundaries (LAGBs) (b) with annealing time at 500°C and 600°C.

measurements on slightly recovered material, it was found that the fragmentation behavior of the grains during deformation depends on the initial grain orientation [2,5]. This difference, being very prominent after 1 pass, almost disappears after 8 passes, but some effect is still present. The structural heterogeneity caused by the local differences in degree of fragmentation and by the presence of the shear bands may play a role during the subsequent annealing behavior.

The deformation texture after 8 passes is weak, with a maximum intensity in the (110) pole figure of 2.5, and consists of 3 components where a <111> slip direction always tends to align with the shear direction of the last pass, and a {110}, {112} or {123} slip plane (in order of preference) tends to align with the shear plane of the last pass.

Evolution of Microstructure and Texture during Annealing

The transmission electron micrograph in Fig. 1b of the IF steel after 3h at 500°C shows a structure with well-defined boundaries tending to form equilibrium angles at the triple lines. The structure has coarsened with respect to the as-deformed state, but the morphological orientation - including the morphology of the shear bands - observed in the deformed structure is still apparent. Some dislocations are still present in the interior of the structure elements. From EBSD measurements an average subgrain size of 0.6 µm is calculated, and a fraction of high angle boundaries, carrying a misorientation of 15° or more, of 71%. A boundary map illustrating the preservation of the morphology of the as-deformed state is shown in Fig. 2.

The subsequent behavior at 500°C as well as the behavior at 600°C is characterized by continuous coarsening of the structure in absence of primary recrystallization. The evolution of the (sub)grain size and the fraction of low angle boundaries at both temperatures is shown in Fig. 3. Fig. 4 shows the evolution of the normalized grain size distribution at 600°C.

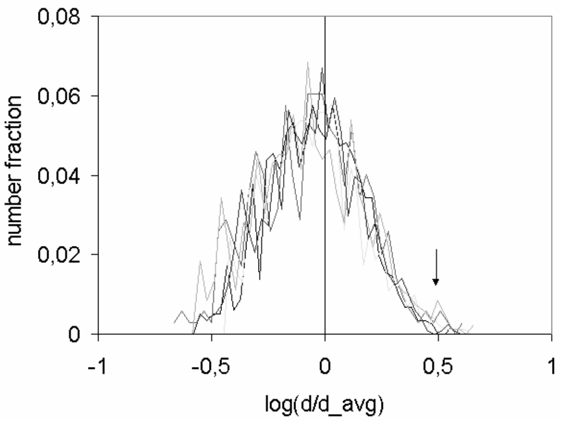

Figure 4: Normalized grain size distributions after different annealing times at 600°C. The arrow indicates a small peak at a grain size about three times the average. The height of the peak increases with increasing annealing time.

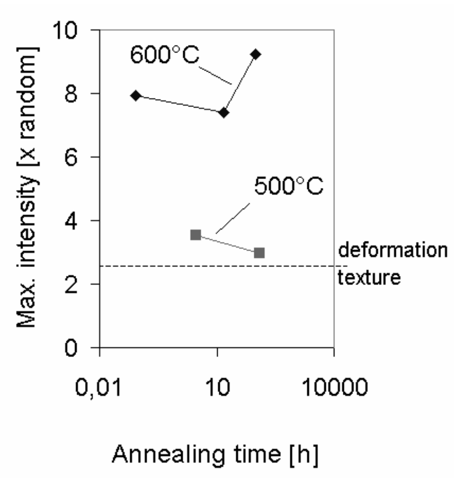

Figure 5: Texture evolution during annealing at 500°C and 600°C. The texture is characterized by the maximum intensity in the inverse pole figure for the shear direction of the last pass.

During annealing the deformation texture is preserved and even increases in intensity. Its evolution is presented in Fig. 5 by means of the evolution of the maximum intensity in the inverse pole figure for the shear direction of the last pass, showing only one peak at the <111> pole.

The evolution of the mechanical behavior during annealing at 500°C is shown in Fig. 6.

Discussion

Continuous Recrystallization. From the comparison of the 2 micrographs in Fig. 1 it is clear that after 3h at 500°C recovery has occurred by the development of subgrain boundaries from the dislocation substructure, and by some subgrain growth. The structure has also become more equiaxed with respect to the as-deformed state. Because of the high fraction of high angle boundaries, developed during severe deformation, a *grain* structure is developed rather than a subgrain structure. Therefore, according to the terminology of Doherty et al. [6], the observed process of recovery and structural coarsening is designated as *continuous recrystallization* (CRX).

The CRX at 500°C takes place well below the recrystallization temperature range of IF steel. However, at 600°C discontinuous (or primary) recrystallization (DCRX) could be anticipated, especially in view of the imposed degree of deformation and the applied annealing times [7]. Yet it does not occur, thus enabling the observed CRX. It is worthwhile questioning *why* DCRX does not occur. We believe that it is impeded by the dynamic recovery occurring during severe plastic deformation. Due to the high dislocation density generated, and due to the homogenizing effect of the high applied strain (evening out fragmentation differences and thus local stored energy differences between original grains), dynamic recovery occurs quite evenly throughout the substructure. This eliminates, upon subsequent annealing, the possibility of some specially recovered subgrains to form nuclei for DCRX and to grow into the surrounding substructure.

The loss of dislocation density and the coarsening after 3h at 500°C result in a decrease in yield stress of about 100 MPa (Fig. 6). Simultaneously, the hardening ability of the material is restored. With additional annealing to 72h the hardening rate increases even more, indicating that after 3h the recovery processes are not complete.

Grain Growth. When the CRX is complete, i.e. when the dislocation density in the (sub)grain interior has reached an equilibrium value and the former dislocation boundaries have become equilibrium grain boundaries, the grain assembly continues to grow, now solely driven by the decrease in total grain boundary energy. It is obvious that this grain growth (GG) occurs for the longer annealing times at 600°C (Fig. 3a), but it is difficult to pinpoint the transition between the 2 processes. The driving force for GG is low, which explains the slow growth at 500°C and the

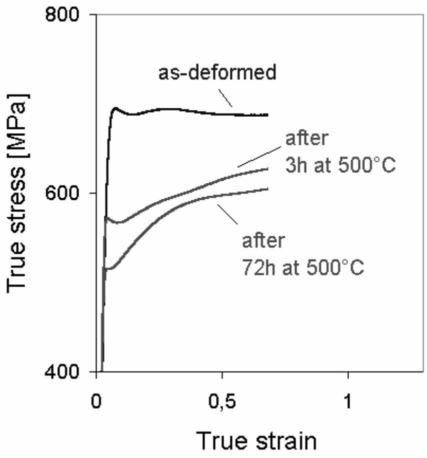

Figure 6: Stress-strain curves for compression tests on IF steel after ECAP (to a strain of 9.2) and after subsequent annealing at 500°C for 3h and 72h. The compression direction was parallel to the extrusion direction of the last pass.

relatively small grain size of about 2µm after 100h at 600°C. This in turn means that the fine-grained structure is relatively stable at the investigated temperatures. Both at 500°C and 600°C the low angle boundary fraction globally increases with increasing annealing time and thus with increasing grain size (Fig. 3b).

Development of Bimodal Grain Size Distribution. During GG at 600°C a slightly bimodal grain structure develops, illustrated by the boundary map of the IF steel after 15h at 600°C in Fig. 7. As shown by the evolution of the normalized grain size distributions in Fig. 4, this slight bimodality (indicated with an arrow) is not present from the first annealing times investigated, but develops during grain growth. The orientations of the large grains do not deviate from the overall texture, and as yet no plausible explanation for the phenomenon could be proposed. Possibly the origin of the deviating growth velocity of the larger grains lies in the inhomogeneities on the microstructural scale in the as-deformed state.

The occurrence of bimodality after annealing of severely deformed structures has previously been reported for Al [8], but no sufficient explanation was offered.

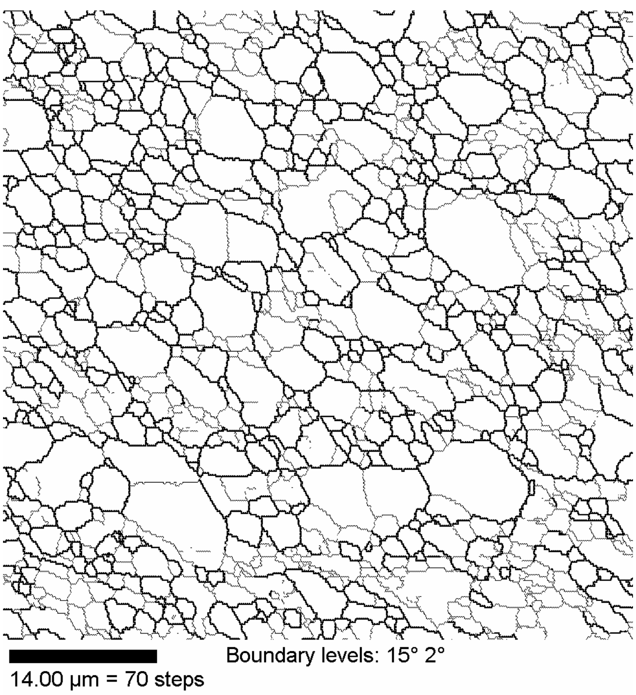

Figure 7: Boundary map with low angle boundaries (2°-15°, thin black line) and high angle boundaries (15° or higher, bold black line) for IF steel after 8 passes via route B_A and a subsequent anneal of 15h at 600°C.

Texture Evolution. The initial texture sharpens during annealing, especially at 600°C where the maximum intensity in the inverse pole figure of the shear direction more than triples with respect to the as-deformed state, to an average of 8 (Fig. 5). At 500°C the intensity increase is more modest, to an average of 3.3. Texture sharpening has been reported before for CRX [6] and GG [9]. It is expected to cause some orientation pinning during GG, notably at 600°C. As yet this could not be related to the observed bimodality.

Conclusion

It was found that during annealing of severely deformed IF steel at 500°C and 600°C continuous recrystallization occurred, followed by grain growth. The latter leads to a slight grain size bimodality at 600°C, which may arise from inhomogeneities in the substructure of the as-deformed state.

The annealing experiments will be continued at higher temperatures in order to find out whether primary recrystallization will occur, and in order to study the grain growth over a larger grain size range.

Acknowledgements

The authors would like to thank the Fonds voor Wetenschappelijk Onderzoek - Vlaanderen (FWO) for financial support through grant N° G.0208.02. The work was performed in the framework of project IAP P8/05 of the Belgian Science Policy. JDM acknowledges financial support through a scholarship of the Fonds voor Wetenschappelijk Onderzoek - Vlaanderen (FWO).

References

[1] D.G. Morris and M.A. Muñoz-Morris: Acta Materialia Vol. 50 (2002), p. 4047.

[2] J. De Messemaeker, B. Verlinden, J. Van Humbeeck and L. Froyen: in *Nanomaterials by Severe Plastic Deformation* (Wiley-VCH, Germany 2004), eds. M.J. Zehetbauer and R.Z. Valiev, p. 332.

[3] V.M. Segal: Materials Science and Engineering Vol. A197 (1995), p. 157.

[4] L. Dupuy and E.F. Rauch: Materials Science and Engineering Vol. A337 (2002), p. 241.

[5] M. Seefeldt and P. Van Houtte: in *Nanomaterials by Severe Plastic Deformation* (Wiley-VCH, Germany 2004), eds. M.J. Zehetbauer and R.Z. Valiev, p. 226.

[6] R.D. Doherty, D.A. Hughes, F.J. Humphreys, J.J. Jonas, D. Juul Jensen, M.E. Kassner, W.E. King, T.R. McNelley, H.J. McQueen and A.D. Rollett: Materials Science and Engineering Vol. A238 (1997), p. 219.

[7] K. Verbeken, L. Kestens and J.J. Jonas: Scripta Materialia Vol. 48 (2003), p. 1457.

[8] C.Y. Yu, P.L. Sun, P.W. Kao and C.P. Chang: Materials Science and Engineering Vol. A366 (2004), p. 310.

[9] F.J. Humphreys and M. Hatherly: *Recrystallization and Related Annealing Phenomena* (Pergamon, Great Britain 1995).