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Structure and mechanical behaviour of interstitial-free steel processed by equal-channel angular pressing

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ABSTRACT

The influence of the number of passes in equal channel angular pressing (ECAP) following route B_C on microstructure and mechanical properties of interstitial-free steel was investigated by means of tensile tests and X-ray texture and diffraction profile analysis. A significant improvement of the mechanical properties was found with increasing the number of ECAP passes. After 8 passes, beside the high strength considerable ductility was observed and at 300 °C the ductility was the same as for the initial sample but with a two-times larger strength. The high strength measured at room temperature was only slightly reduced during annealing at temperatures up to 500 °C.

1. Introduction

Over the past few years, tailoring microstructures with ultra-fine grain sizes in bulk materials has attracted significant interest from the scientific community. This is due to the fact that grain size strengthening is one of the few mechanisms that lead to improvement in the strength of materials, retaining an apprecia-ble level of ductility and flow properties. Ultrafine grain sizes in bulk materials can be achieved by severe plastic deformation (SPD), which involves extremely large imposed plastic strains without significant change of dimensions of the workpiece [1]. A number of innovative and nonconventional processing methods have emerged following this philosophy, e.g. high pressure torsion (HPT) [2], equal-channel angular pressing (ECAP) [3], multiaxial forging [4] and accumulative roll bonding [5].

Interstitial-free (IF) steels constitute an important class of steels having carbon content less than 0.01 wt.%. These steels are extensively used in automotive industries for making car bodies owing to the high formability that they possess. In recent years, efforts have been made to improve the strength of this class of steels by means of grain refinement mostly through SPD procedures.

In ECAP, a billet of the material is pressed through a die consisting of two channels with identical cross sections, intersecting at an angle φ. Numerous publications dealing with ECAP are devoted to face centered cubic (fcc) [6] and hexagonal close packed (hcp) materials [7]. However, reports on ECAP-processing of body centered cubic (bcc) materials, such as Fe [8] and ferritic or perlitic steel [9] are not as frequent. ECAP-processed IF steels, having a single-phase ferritic structure, only got into the focus of interest in the last few years [9]. The application of ECAP to IF steels improves mechanical properties [9], therefore understanding the underlying deformation mechanisms occurred during ECAP is of great importance. The knowledge of deformation behaviour of ECAP-processed samples in wide range of temperatures is very important for a successful implementation of this material to applications. Nevertheless, the number of papers treating the temperature dependence of mechanical properties has remained limited. In this paper, the microstructures of IF steel samples processed by ECAP for different number of passes and their mechanical behaviours at various temperatures are investigated.

2. Experimental procedure

The material used in this investigation was an IF steel with a composition of 0.0026 wt.% C, 0.096 wt.% Mn, 0.045 wt.% Al and 0.041 wt.% Ti. The processing of specimens prior to ECAP was the following: after casting, the ingot was size-rolled to fabricate a Plate 12 mm in thickness, homogenized for 1 h at 700 °C, and then furnace-cooled. For ECAP processing, the billet was cut into 12 mm × 12 mm × 60 mm workpieces, which were annealed for 2 h at 700 °C, furnace-cooled and surface-polished using 1200 grit SiC paper. The ECAP-processing was carried out at room temperature up to eight passes following route B_C, i.e. the billet was rotated by 90° clockwise about the longitudinal axis between the consecutive passes. The ECAP was performed with a pressing rate of 2 mm/min in a die with a channel intersection angle of $\varphi = 90°$.

For metallographic examination the samples were mounted in Epofix® resin and mechanically polished successively using 240-, 1200- and 2400-grit SiC papers.

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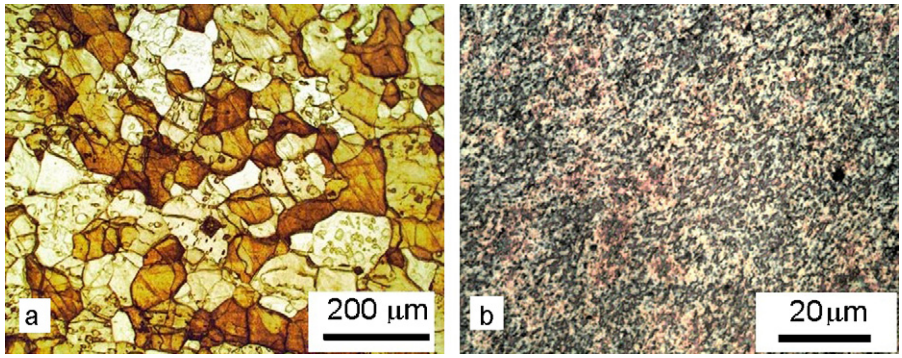

Fig. 1. Optical light microscope images of the microstructure of IF steel: (a) initial state and (b) after 8 ECAP passes via route B_c.

Subsequently, polishing by 3 μm and 1 μm diamond suspensions and a final etching in 2% Nital solution for 10 s were carried out.

The microstructure was studied both by X-ray line profile analysis and macroscopic texture measurements. The X-ray line profiles were measured by a high-resolution diffractometer with $CoK\alpha_1$ radiation (λ = 0.1789 nm) and evaluated by Convolutional Multiple Whole Profile (CMWP) fitting method [10,11]. In this procedure, the experimental pattern is fitted by the convolution of the instrumental pattern and the theoretical size and strain line profiles. Because of the ultrafine grained structure of the studied samples, the physical broadening of the profiles was much higher than the instrumental broadening; therefore, instrumental correction was not applied in the evaluation. The theoretical profile functions used in this fitting procedure are calculated on the basis of a model of the microstructure, where the crystallites have spherical shape and log-normal size distribution, and the lattice strains are assumed to be caused by dislocations. Macroscopic crystallographic textures of various conditions were determined by means of PHILIPS X'Pert PRO MRD device, using $Cu\text{-}K_\alpha$ radiation (λ = 0.15418 nm).Flat specimens with 12 mm gauge length and 1 mm in thickness were fabricated for tensile tests. These measurements were performed using an Instron 5882 testing machine at a strain rate of $10^{-3}\,s^{-1}$ in temperature range of 20–300 °C.

In order to investigate the thermal stability of the ECAP-processed microstructures, heat treatments at 500 °C for 20, 40 and 60 min were performed following the work of Niendorf et al. [12]. The Vickers microhardness of the annealed samples was measured using a LECO microhardness tester by applying a load of 100 g for 10 s and taking an average over 10 separate measurements.

3. Results and discussion

The ECAP-processing has a significant influence on the microstructure. The difference between the initial microstructure and that after 8 ECAP passes is presented in Fig. 1. As we reported in our previous work [13], the initial average grain size of 41 μm decreases to 0.36 μm after 8 passes. The initial microstructure does not exhibit any preferred orientation [13,14]. The results of texture measurements are presented for normal (ND) direction (for its definition see Ref. [15]). The background was subtracted from experimental data by means of Philips X'Pert Texture 1.0a software. Recalculated $\{1\,1\,0\}$ pole figure for the sample processed by 1 ECAP pass is presented in Fig. 2a. The maximums on the pole figure correspond to the J $\{1\,1\,0\}\left\langle 1\,1\,2\right\rangle$ components of simple shear ideal orientations [16] as it is also shown in the corresponding orientation distribution function (ODF) calculated for $\varphi_2 = 45°$ (Fig. 2e). The importance of J1/J2 components for ECAP-processed IF steels has been also reported by De Messemaeker et al. [15]. The second pass weakens this texture (cf. Fig. 2b). After the fourth pass, the (1 1 0) texture forms newly, nevertheless the maxima are tilted by 20° with respect to ED (Fig. 2c). Such tilt phenomenon has been reported by several authors in bcc structures [16–18] and could be explained by means of cloud-model of Tóth and coworkers [16,19]. After 8 passes the texture is fairly weak (Fig. 2d). This result deviates from the observation of De Messemaker et al. [15], who reported enhancement of J1/J2 components. This deviation can be attributed to the different sample preparation conditions (in [15] the samples were ECAP processed at 200 °C), but the exact explanation needs further experiments.

Since the dislocation structure plays a substantial role in mechanical properties, the microstructure of the ECAP-processed steel samples was studied by X-ray line profile analysis. As an example, the fitting of the X-ray diffraction pattern for the sample processed by 2 passes is shown in logarithmic intensity scale in Fig. 3. The open circles and the solid line represent the measured data and the fitted curves, respectively. The difference between the measured and fitted patterns is also plotted at the bottom of the figure. The area weighted mean crystallite size ($\langle x\rangle_{area}$) and the dislocation density (ρ) were determined from the fitting and listed in Table 1. These values are obtained by averaging the parameters determined on the cross and transverse sections of the ECAP-processed billets. The value of $\langle x\rangle_{area}$ is calculated as $\langle x\rangle_{area} = m\exp(2.5\sigma^2)$, where m and σ are the median and the log-normal variance of the size distribution density function obtained from the pattern fitting. The crystallite size (70–80 nm) determined by X-ray line profile analysis is lower than the grain size observed previously by electron microscopy (~400 nm) [13]. This phenomenon has been usually observed for plastically deformed bulk metals [20] and it can be attributed to the fact that the crystallite size determined from X-ray line profiles corresponds essentially to the mean size of cells/subgrains which is usually smaller than the conventional grain size measured in metals by electron microscopy methods [20]. The parameter q was also obtained from the fitting that characterizes the type of dislocations: edge or screw or mixed. In the case of Fe for pure edge and screw dislocations the values of q are 1.28 and 2.67, respectively. For a dislocation structure having mixed character the value of q is between these limiting cases. From the line profile analysis it can be concluded that the crystallite size saturated even after the first pass of ECAP, while the dislocation density increased up to 4 passes. This has been also observed for ECAP-processed Cu [21]. The character of dislocations is more screw as the q values revealed, which can be explained by the reduced mobility of screw dislocations compared to edge dislocations in bcc structures. This difficulty in motion of screw dislocations is due to the fact that the ground state dislocation core is dissociated into a non-planar configuration [22]. As a consequence, during ECAP-processing the edge dislocation segments can annihilate more easily than the screw ones thereby the remaining dislocations have more screw character.

Temperature of heat treatment was set to 500 °C in order to get data for the comparison with the results of Niendorf et al. [12], who, to the best of authors' knowledge, have presented the only study on thermal stability of ECAP processed IF steels. The dependence of Vickers microhardness on duration of heat treatment is presented in Fig. 4, including data from [12]. The hardness of the samples pressed via route B_c is higher than the values of the specimens processed for the same numbers of passes by route C and E (two extrusions along route C, then a 90° rotation, and then another 2× route C extrusions). The difference could originate from the latent

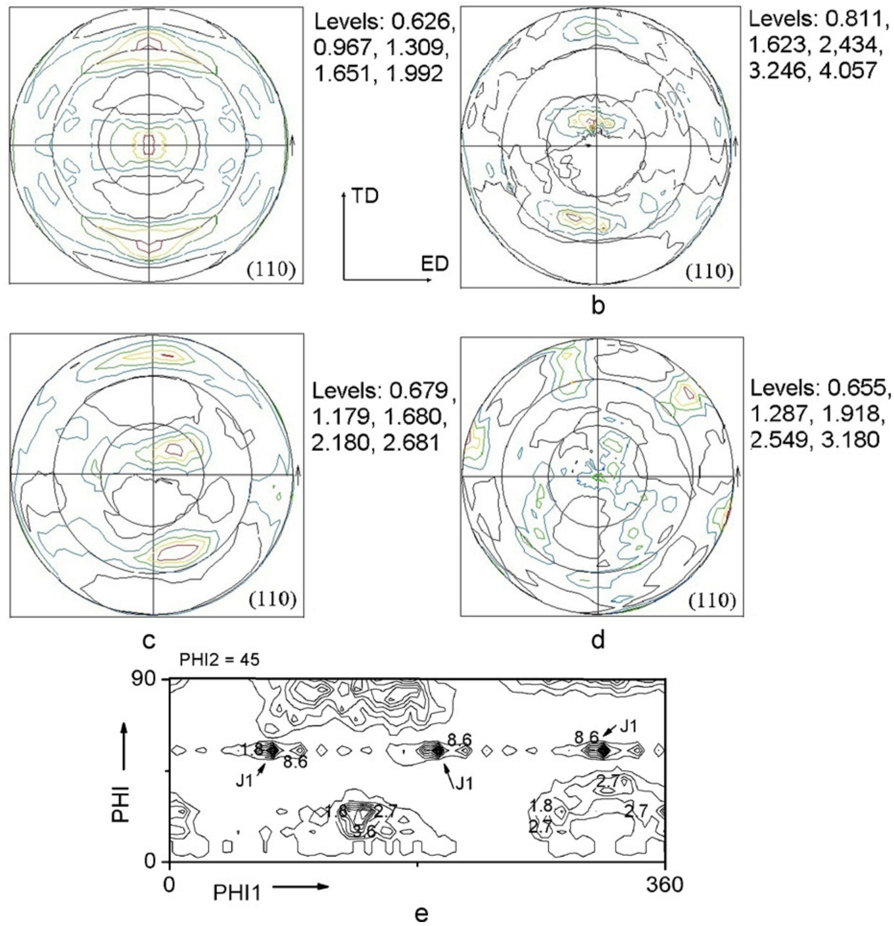

Fig. 2. {1 1 0} pole figure of the ECAP processed IF-steel: (a) after 1 pass, (b) after 2 passes, (c) after 4 passes, (d) after 8 passes, all via route B_c. (e) Experimental ODF for $\varphi_2 = 45°$ after 1 pass.

hardening [23], appearing for route B_C, discussed in detail in our previous work [13]. After 20 min of annealing, the values of microhardness decrease by approximately 10–20% and there is no further decrease for longer annealing times. The detailed microstructure analysis did not find any evidence of grain growth therefore the hardness reduction was most probably caused by recovery processes [24].

Table 1

The crystallite size $<x>_{area}$, the dislocation density (ρ) and the parameter q describing the edge/screw character of dislocations obtained by X-ray line profile analysis as a function of number of ECAP passes.

Sample	$<x>_{area}$ [nm]	ρ [10^{14} m^{-2}]	q
0 ECAP	<1 μm	<0.1	–
1 ECAP	72 ± 10	6.2 ± 0.6	2.3 ± 0.1
2 ECAP	80 ± 10	8.3 ± 0.7	2.4 ± 0.1
4 ECAP	80 ± 8	10.3 ± 1.0	2.4 ± 0.1
8 ECAP	66 ± 8	10.3 ± 1.1	2.5 ± 0.1

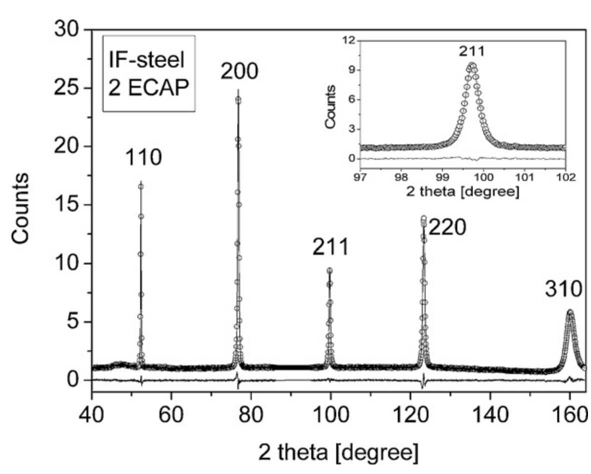

Fig. 3. X-ray diffractogram fitted by the CMWP method for the sample processed by 2 ECAP passes. The open circles and the solid line represent the measured data and the fitted curves, respectively. A magnified part of the diffractogram is shown in the inset.

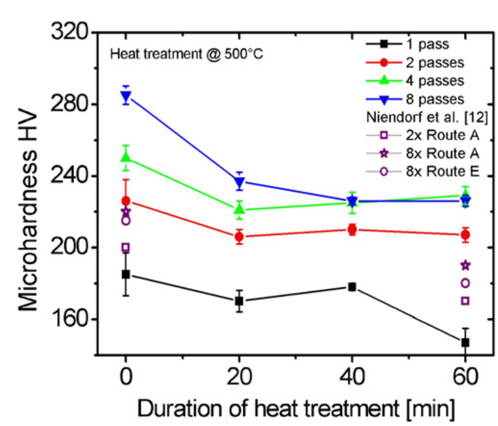

Fig. 4. Dependence of Vickers microhardness on annealing time (at 500 °C). Data for routes C and E are from Ref. [12].

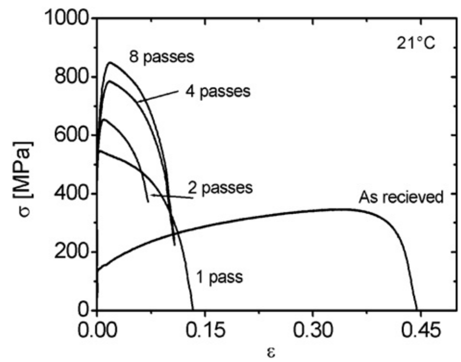

Fig. 5. True stress–plastic strain curves at 21 °C for various numbers of ECAP passes.

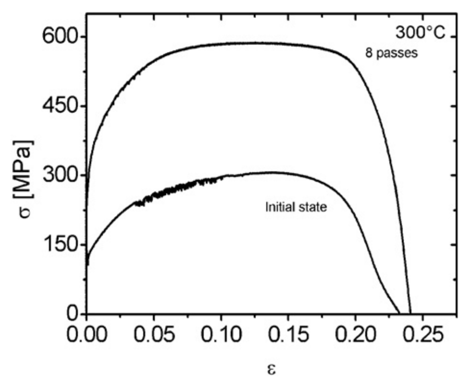

Fig. 6. True stress–plastic strain curves at 300 °C in the case of the initial state and for the sample processed by 8 ECAP passes.

The results of tensile testing at room temperature (21 °C) for various numbers of passes using an initial strain rate of 10^{-3} s^{-1} are shown in Fig. 5. Due to ECAP-processing the flow stress increased while the strain to failure decreased, which is a general finding for ECAP-processed materials [25,26]. The flow stress for all ECAP-processed samples reaches a maximum at a small strain and than decreases with increasing strain. Such behaviour is very similar to cold rolled samples [27] and indicates only a small amount of uniform elongation. The ductility lost due to ECAP-processing for 2 passes was partially regained after 4 and 8 passes. This may be a consequence of the change of grain boundary character. A previous study [28] on ECAP-processed Al6082 alloy has shown that the fraction of high angle grain boundaries increased even after the saturation of the parameters of the microstructure (e.g. dislocation density). As a result of this evolution, the strength further increased and the initial texture was diminished after 8 passes as well as the grain orientation distribution became to be close to random case.

In Fig. 6 the flow curves obtained at 300 °C for both the initial state and the sample processed by 8 passes are displayed. The jerky flow appearing on flow curves for both states can be attributed to the Portevin-Le Chatelier (PLC) effect as it was observed, e.g. by Pink [29]. The flow curves in Fig. 6 revealed that the ECAP-processing significantly improved the mechanical behaviour at 300 °C since the sample processed by 8 passes exhibits nearly the same ductility as in the initial case while its strength is approximately 2 times higher.

4. Conclusions

IF steel samples processed at room temperature by ECAP up to a total of 8 passes via route B_C were investigated. Significant microstructure refinement occurred as a consequence of the ECAP process. After the first pass a strong {1 1 0} texture forms, which vanishes after the eighth pass. The dislocation density saturates after the fourth pass and the dislocations have more screw character. The strength of the samples increases monotonously with increasing the number of ECAP passes while the ductility lost after 1 pass was partially regained at higher number of passes. The sample processed by 8 passes shows very high strength together with a considerable ductility. The high strength of the ECAP-processed samples is maintained even at 500 °C. The benefit of ECAP-processing is evident for the specimen subjected to 8 passes since this sample shows nearly the same ductility but two times higher strength than the initial sample at 300 °C.

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