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

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The influence of the number of passes in equal channel angular pressing (ECAP) following route B\textsubscript{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\textdegree 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\textdegree C.

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1. Introduction

Over the past few years, tailoring microstructures with ultrafine 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 appreciable 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 \(\varphi\). 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\textdegree 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\textdegree 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\textsubscript{C}, i.e. the billet was rotated by 90\degree 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 Epoxy\textsuperscript{®} resin and mechanically polished successively using 240-, 1200- and 2400-grit SiC papers.

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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 (\(<\varphi_{\text{area}}\)) and the dislocation density (\(\rho\)) 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 \(<\varphi_{\text{area}}\) is calculated as \(<\varphi_{\text{area}} = m \exp(2.5\sigma^2)\), where \(m\) and \(\sigma\) 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
hardening [23], appearing for route Bc, 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].

The crystallite size $<x>_{\text{area}}$, the dislocation density ($\rho$) 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.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$&lt;x&gt;_{\text{area}}$ [nm]</th>
<th>$\rho$ $\times 10^{14}$ m$^{-2}$</th>
<th>$q$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 ECAP</td>
<td>1 $\mu$m</td>
<td>&lt;0.1</td>
<td>--</td>
</tr>
<tr>
<td>1 ECAP</td>
<td>72 $\pm$ 10</td>
<td>6.2 $\pm$ 0.6</td>
<td>2.3 $\pm$ 0.1</td>
</tr>
<tr>
<td>2 ECAP</td>
<td>80 $\pm$ 10</td>
<td>8.3 $\pm$ 0.7</td>
<td>2.4 $\pm$ 0.1</td>
</tr>
<tr>
<td>4 ECAP</td>
<td>80 $\pm$ 8</td>
<td>10.3 $\pm$ 1.0</td>
<td>2.4 $\pm$ 0.1</td>
</tr>
<tr>
<td>8 ECAP</td>
<td>66 $\pm$ 8</td>
<td>10.3 $\pm$ 1.1</td>
<td>2.5 $\pm$ 0.1</td>
</tr>
</tbody>
</table>

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 Bc. (e) Experimental ODF for $\varphi_2=45^\circ$ after 1 pass.

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].
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 exhibited 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 Bc 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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