EVOLUTION OF MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ULTRA-FINE-GRAINED INTERSTITIAL-FREE STEEL PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING

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Resume

Equal channel angular pressing (ECAP) is one of the severe plastic deformation techniques which is widely used for producing metals with ultra-fine-grained microstructures. In the present work the influence of number of pressing by route B_C on grain size, evolution of microstructure and mechanical properties of interstitial-free (IF) steel has been investigated by means of optical microscopy, electron back-scattering diffraction (EBSD) and tensile tests. It has been found, that the grain size decreases with increasing number of passes. Simultaneously tensile strength increases. The thermal stability of ECAP-processed microstructures has been also examined. It was found that the degradation of mechanical properties occurs only above 600 ˚C and 700 ˚C.

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1. Introduction

New technologies, especially in transport industry require developing of new materials. Particularly in the last decade an increasing interest in submicrocrystalline and nanocrystalline metallic materials is observed. The reason for that is an assumption of better mechanical properties based on Hall-Petch relationship in comparison with materials with coarser grain structure. One of the methods of preparation ultrafine-grained microstructures is application of severe plastic deformation (SPD) on materials. One of the most popular SPD methods is the Equal Channel Angular Pressing (ECAP) [1]. During the ECAP process, a billet of the material is pressed through a die consisting of two channels with identical cross sections, intersecting at an angle φ. The evolution of microstructure and final mechanical properties of the sample depend on number of passes, the angle of rotation of sample after the each pass and pressing temperature [2-6]. Generally, the ECAP process is suitable for achieving grain sizes in the range of hundreds nanometers [7-10]. Current research of ultra-fine-grained materials deals with problems of plastic properties at the microscopic scale, inhomogeneities in the structure in cross section of the workpieces and thermal stability of microstructure.

Interstitial-free (IF) steels with single phase ferritic microstructure constitute an important class of steels having carbon percentage as low as below 0.01. 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 these classes of steels by means of grain refinement mostly through SPD processes. This paper is focused on investigation of microstructural evolution and mechanical properties of ultrafine-grained IF steels processed by ECAP. Thermal stability of ECAP-processed microstructures is also examined.

2. Experimental methods

Measurements were performed on IF steel samples with a composition of 0.0026 wt.% C, 0.096 wt.% Mn, 0.045 wt.% Al and 0.041 wt.% Ti manufactured in Pohang Steel Company (POSCO, Korea). The processing of specimens was the following: after casting, the ingot was size-rolled to fabricate a plate of 5 mm thickness, homogenized for 1 h at 973 K, and then furnace-cooled. For ECAP processing, the billet was cut into workpieces with dimensions of 5 mm x 5 mm and length of 55 mm, which were annealed for 2 h at 973 K, furnace-cooled and surface-polished using 1200 grit SiC paper. Samples were pressed through the die with characteristic angles ϕ = 90˚ and ψ = 0˚ at room temperature, using route B_C. The ECAP-processing were conducted up to eight passes with a pressing speed of 2 $mm.min^{-1}$. For metallographic examination the samples were mounted in Epofix® resin and mechanically polished using standard methods. The specimens were etched in solution composed of 40 ml HNO_3 and 15 ml HF for 10 s. The Vickers microhardness 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. Both optical and transmission electron microscopy (TEM) were used to investigate the microstructure. TEM thin foils were prepared by the twin-jet polishing technique using a mixture of 6 % $HClO_4$ and methanol at -30 ˚C. In both techniques, a cross section (perpendicular to the extrusion direction - plane X) of the samples was investigated. Microstructure of the ECAP-processed samples was examined by EBSD on microscope Zeiss-LEO1530 equipped with a detector Nordlys II. This method allowed to evaluate the grain size of material by means of the linear intercept method (only high angle boundaries (>15°) were taken into calculations) and fraction of high-angle boundaries. Mechanical properties were tested in tension with a constant strain rate of 10^{-3} s^{-1} in the temperature range of 21 ˚C - 300 ˚C. These measurements were performed using an Instron 5882 testing machine and flat specimens with 12 mm gauge length and rectangular cross-section of 3.5 mm x 0.8 mm were used. The specimen tensile axis was parallel to the pressing direction. In order to reveal the deformation mechanisms, the microstructures of the deformed samples were examined by Quanta FEG scanning electron microscope. The preparation of the specimens for this investigation was similar to that of the metallography and longitudinal section of the samples, parallel to the pressing direction, was examined.

3. Result and discussion

Initial state of IF steel is characterized by a homogeneous microstructure, formed by equiaxed grains with the similar size. Crystallographic orientation map of the surface showed random orientation of the grains in the initial state. After the first pressing the grain size significantly decreases from the original 41 μm to 15 μm (Table 1).

Table 1

Evolution of grain size as a function of increasing number of ECAP passes

Sample	**0x ECAP**	**1x ECAP**	**2x ECAP**	**4x ECAP**	**8x ECAP**
d (μm)	41.7	15	8	0,4	0.36

At the same time the fraction of high-angle boundaries reduced about 17 %. The detailed TEM investigation of 1x ECAPed sample revealed a directionally aligned microstructure and development of subgrain formation. The observed region consists of bands of subgrains with sharp boundaries and dislocation-rich cells with fuzzy boundaries. The length of subgrains is 1-2 μm and their width 300-500 nm (Fig. 1).

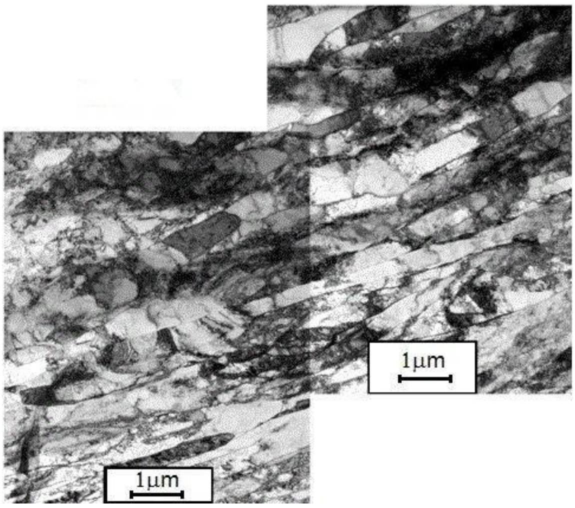

Fig. 1. TEM micrograph of the cross section of 1x ECAPed sample

Owing to this process the above mentioned reduction of high-angle boundaries fraction was observed. Further passing of the samples resulted in a refinement of the microstructure, but its rate is slower in comparison with the first pressing. After the fourth pass the microstructure was stabilized and grain size didn't change significantly with subsequent passes. The average grain size got settle at approximately 0.4 μm. The eight times ECAPed sample exhibit only slight increase in the fraction of high-angle boundaries (from 28 % to 31 %) in comparison to that after four passes. The ultra-fine-grained microstructure has expanded into a larger volume of material in accordance with the theoretical model of Kim and coworkers [11]. In agreement with this assumption the eight times ECAPed samples exhibit very fine and homogeneous microstructure, whose prevailing part is formed by new small equiaxed grains with a size of 200-500 nm containing only a few dislocations (Fig. 2). In order to investigate the thermal stability of the ECAP-processed microstructures and corresponding mechanical properties, the samples were annealed for 1 hour at 500 ˚C, 600 ˚C and 700 ˚C and quenched into the water.

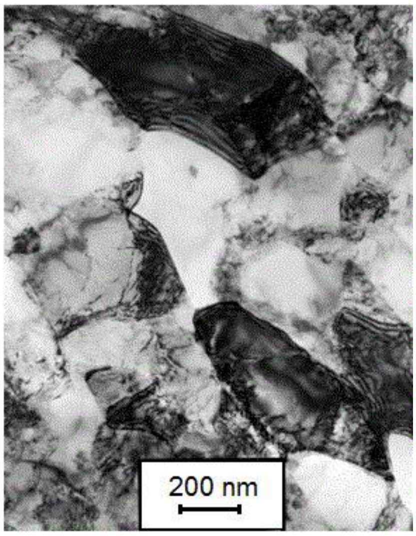

Fig. 2. TEM micrograph of the cross section of 8x ECAPed sample

Temperature and time of annealing were selected according to results published in [12]. Evolution of microhardness with increasing temperature of annealing is plotted in Fig. 3.

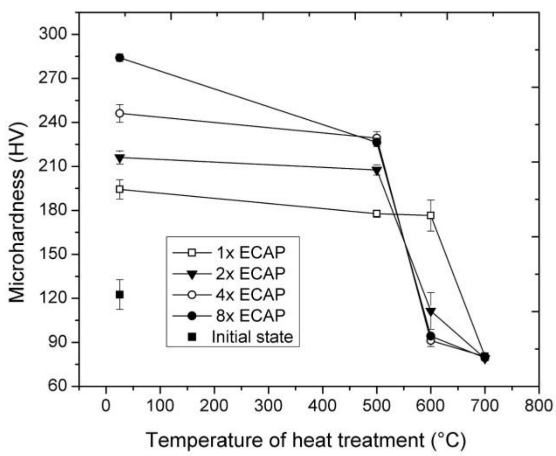

Fig. 3. Dependence of Vickers hardness on the temperature of annealing

The microhardness of the initial sample was also plotted for comparison. After annealing at 500 ˚C, the values of microhardness decreased by approximately 10 %

for all samples except eight times pressed sample. For this sample the value of microhardness decreased by 15 %. The microstructural observations didn't reveal any significant changes in the microstructure, so rather then recrystallization, the recovery of the microstructure takes place. Decrease of microhardness was caused by lower density of dislocations in the recovered material. In the case of eight times pressed sample, the observed moderate grain growth causes further decrease of the microhardness. At 600 ˚C a significant decrease in microhardness was already observed for samples with higher number of ECAP. Micrographs revealed the ongoing process of coarsening of the microstructure, which caused the decrease of the microhardness. The high stored strain energy was the driving force of grain growth in this case. Microhardness of all samples annealed at 700 ˚C decreased to the same value, but the resulting microstructures of the samples exhibit different grain sizes (Fig. 4).

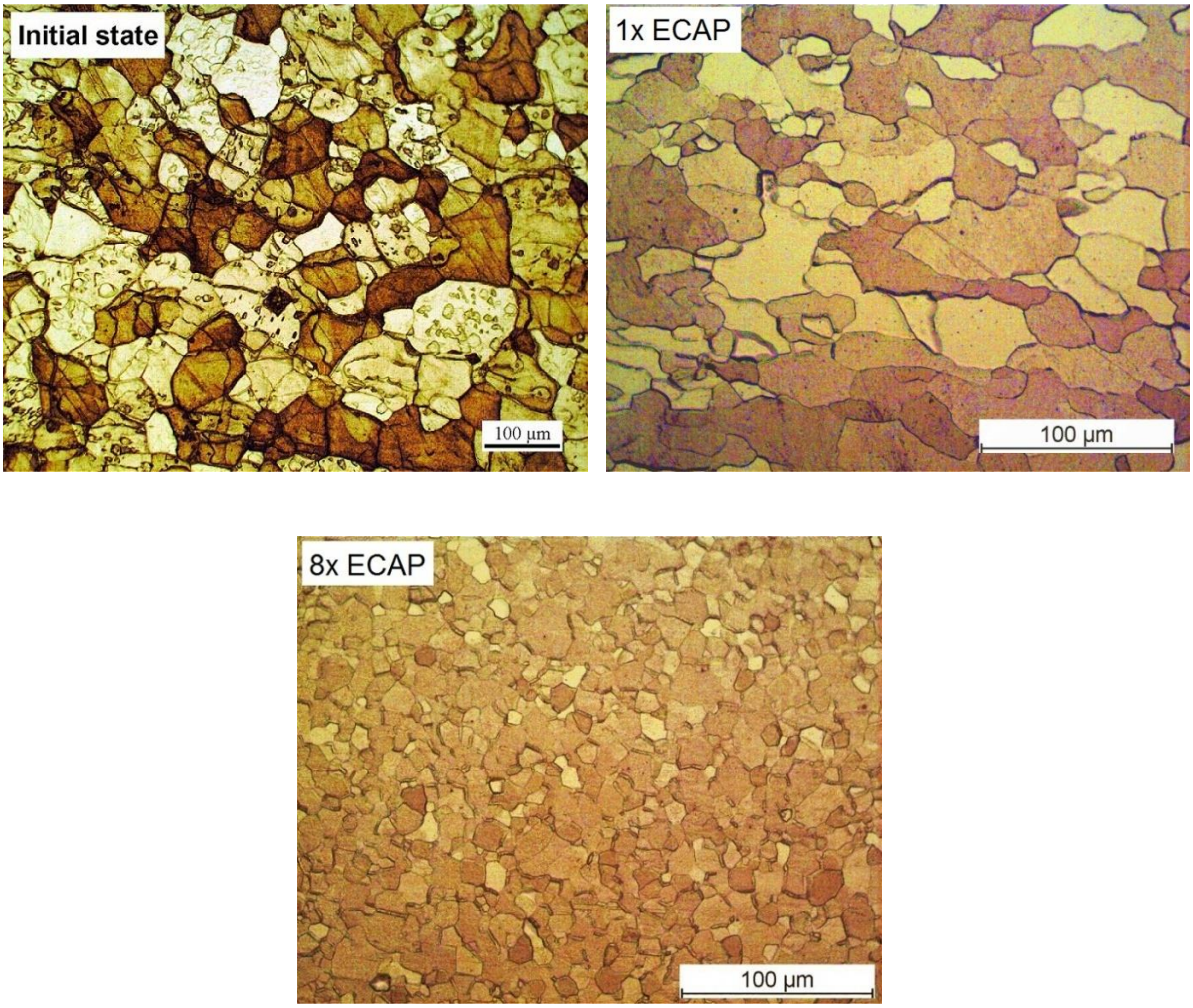

Fig. 4. Optical micrographs of initial state and samples annealed for 1 hour at 700 ˚C taken from the cross section

Even after annealing at 700 ˚C the microstructure of 8x ECAPed sample was not return to the initial coarse grained microstructure. The results of deformation tests are shown in Fig. 5. At room temperature, the value of both yield and tensile strength increased and the ductility decreased with the increasing number of passes as a consequence of the increasing dislocation density and the decreasing grain size. The main advantage of the ECAP process can be seen in Fig. 6, where the tensile curves of initial and 8x ECAPed state at 300 ˚C are depicted. It is obvious, that the eight times pressed sample exhibits two times higher strength at the same level of ductility. The observation by scanning electron microscope revealed that the microstructure of the eight times pressed samples after the tensile test at room temperature and at 300 ˚C differs in grain size (Fig. 7).

The grains are elongated and tilted from the tensile axis. For both temperatures, main deformation mechanism is dislocation slip. Ductility of the sample deformed at 300 ˚C was restored due to the dynamic recovery process and subsequently grain growth, which occurs for this sample already at 300 ˚C owing to the larger deformation energy stored in the sample. As a consequence of the recovery processes, decrease of dislocation density takes place followed by an increase of the dislocations' free path. It is noteworthy to mention, that the initial sample exhibits lower ductility at 300 ˚C, than that at room temperature. This fact can be attributed to the Portevin-Le Chatelier (PLC) effect. Dynamic strain ageing is responsible for serrations, which were observed on the true stress-true plastic strain curve. The ductility of initial sample at 300 ˚C was decreased due to the strong localization of deformation to the lower number of active slip bands [13].

4. Conclusion

In this work the influence of equal channel angular pressing (ECAP) on microstructure and mechanical properties of IF steel was studied. It was observed that ECAP has significant impact on the evolution of microstructure and mechanical properties. The increasing number of ECAP passes caused significant refinement of microstructure, augmentation of both dislocation density and fraction of high-angle boundaries. The original grain size of 41.7 μm gradually decreased to a value of 0.36 μm after eight passes and the tensile strength increased about 2 times. The thermal stability of the ECAP-processed microstructure was also examined. It has been found that effect of heat treatment at 500 ˚C is minimal and degradation of mechanical properties starts above 600 ˚C.

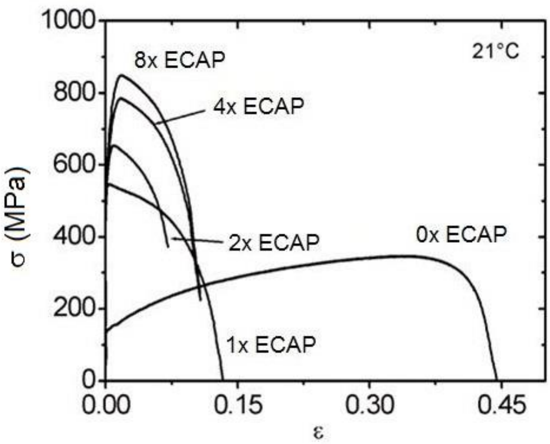

Fig. 5. True stress-true plastic strain curves at 21 ˚C obtained for various numbers of ECAP passes

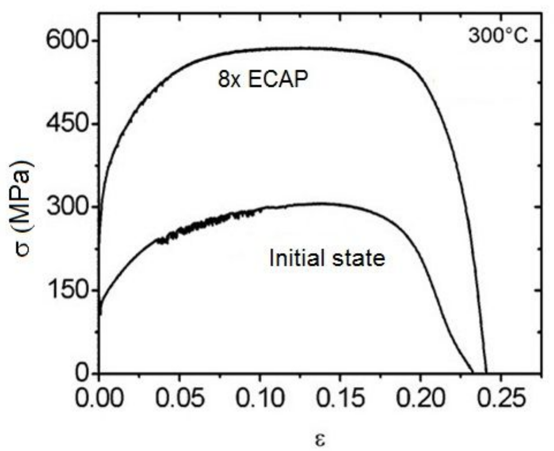

Fig. 6. Comparison of true stress-true plastic strain of initial state and 8x ECAPed samples, respectively, at 300 ˚C

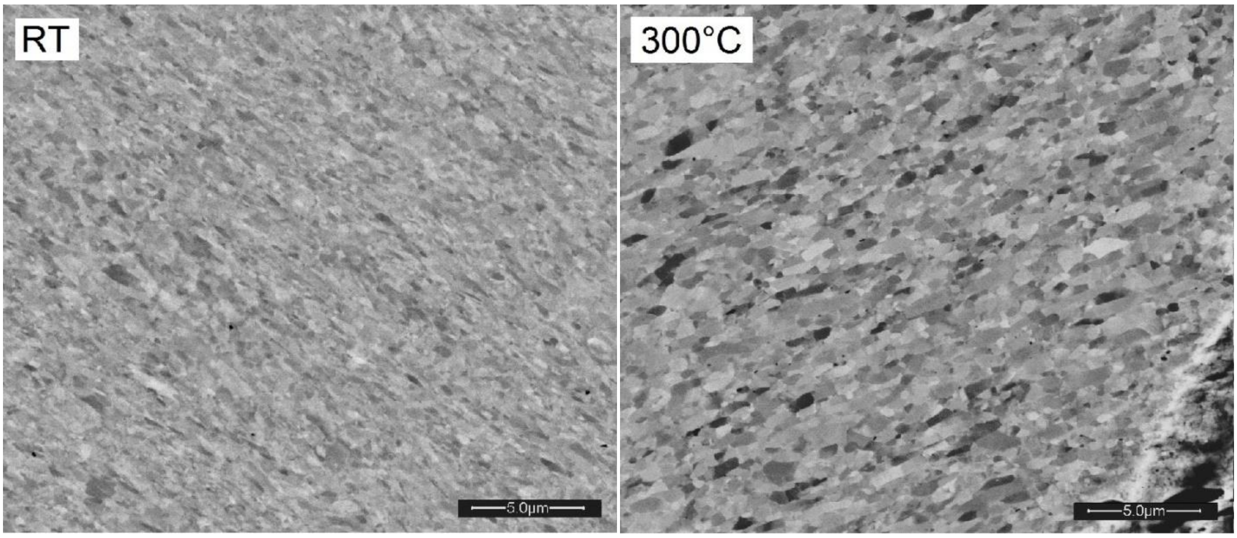

Fig. 7. SEM micrographs of the longitudinal section of 8x ECAPed sample deformed at room temperature and at 300 °C

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