Ultrafine grain structure development in low carbon steel processed by equal channel angular pressing at increased temperature

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Abstract

Commercial low carbon steel AISI 1010 was subjected to Equal Angular Channel Pressing (ECAP) at different temperatures. The paper describes the refinement of a quiet coarse-grained ferrite microstructure to submicrocrystalline range by large plastic strain. The steel was deformed in an ECAP die with a channel angle $\varphi = 90°$, at different temperatures in the range of 150–300 °C. The number of passes at each temperature was $N = 3$. Optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to study the formation of substructure and ultrafine grains in the deformed specimens. The TEM study reveals that even at the lowest deformation temperature the extensively elongated ferrite grains with dense dislocation network dominate in the structure. However, at the lowest deformation temperature of the ECAP process, the randomly scattered polygonized subgrains are already present in the deformed structure. The activation of dynamic recovery process, even at the lowest temperature of equal channel pressing, contributed to the formation of individual polygonized grains. As the temperature of ECAP processing is raised, the process of dynamic polygonization and recrystallization proceeds more effectively and the submicrocrystalline structure is formed by sectioning of elongated ferrite grains. The size of newly generated polygonized grains (subgrains and/or submicrocrystalline grains) is in the range of 300–500 μm. The results proved that the warm ECAP of the low-carbon steel under specific testing conditions leads to the formation of a predominantly submicron grain structure. The formation of such predominant submicrocrystalline structure resulted in significant increase in the yield stress and tensile strength of the steel.

Key words: low carbon steel, warm ECAP, structure evolution, dynamic recrystallization, mechanical properties

1. Introduction

Nanomaterials are receiving increasing attention in the technical community in the last years. The field of severe plastic deformation (SPD) processing of UFG/nanostructured materials has been reached significant progress. Nanomaterials are defined as solids with nanoscale (typically 1–100 nm) structures or substructures [1]. Two approaches have been developed to synthesize nanostructured materials [2]. The first one is a "bottom-up" approach; the second approach for producing nanostructural materials is a "top-down" approach. Most bottom-up approaches produce nanopowders, while the most successful top-down approach has been via SPD techniques, when submicrocrystalline materials with grain size in the range of 100–1000 nm are received [3, 4]. However, high-pressure torsion method has the capability of producing the finer grain sizes in bulk pure metals and alloys, down to 80 nm [5].

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The fabrication of bulk materials with ultrafine grain size has attracted a great deal of attention over the past two decades because of the materials' enhanced properties [6–9]. The term "ultrafine grain structure" is referring to nanostructure with grain size of less than 100 nm, and submicrocrystalline structure with grains between 100 and 1000 nm. In recent years it has become a worldwide effort to develop a manufacturing process to obtain ultrafine grain structures in steels. Currently, there are two main approaches for refining ferrite grains down to the ultrafine grain range in bulk steels. While the first group comprises advanced thermomechanical processes, the approach of the second group employs various severe plastic deformation techniques to refine the structure.

The purpose of the advanced thermomechanical processes is to provide such processing conditions as to multiply ferrite nucleation sites and suppress grain growth to a great extent for overcoming through the limit of grain refinement of the conventional thermomechanical processes. In order to produce ultrafine ferrite grains of 1 μm or less, the following processes were found effective: strain-induced ferrite transformation [10], dynamic recrystallization of the austenite during hot deformation with subsequent $\gamma \rightarrow \alpha$ transformation [11], hot rolling in the intercritical region [12], and dynamic recrystallization of ferrite after heavy warm deformation [13]. The ultrafine grain steels exhibited 900 MPa strength without additional alloying and showed excellent properties like strength-elongation ratio, toughness and fatigue strength [14].

The second group comprises various severe plastic deformation techniques used to refine structure. To introduce large plastic strain into bulk material, different techniques have been used such as ECAP [6, 15], high-pressure torsion (HTP) [16], accumulative roll bonding (ARB) [17, 18], constrained groove pressing (CGP) [19], and others. It is especially the ECAP that generates interest among investigators since it is one of the advanced methods of severe plastic deformation used for metallic materials to produce massive billets with ultrafine-grained structure. Generally, materials fabricated these ways exhibit high strength but only limited ductility. Recently, different approaches have been considered in an attempt to overcome this limitation [8, 20–22]. A significant increase in plastic elongation resulted when structure with bimodal distribution of grain size was fabricated.

Most works are related to the ECAP of pure metals and rather plastic alloys. The use of ECAP for commercial steels has been rarely studied. There are studies dealing with the use of ECAP for low-carbon steels. With cold ECAP, low-carbon steels can only be pressed by two or three passes with channel intersection of 90° before initiation a failure of sample. The two to four passes realized currently with cold ECAP are insufficient and the achievable strain amount is insufficient to produce a completely refined grain structure [23]. To form stable ultrafine grain structure in metals and alloys, ECAP should be carried out at the temperature corresponding to the temperature of cold working [24].

The purpose of this work is to study the formation of submicrocrystalline structure in commercial low carbon steel AISI 1010 subjected to large strain during warm deformation in dependence on varying temperature of ECAP pressing. The influence of the temperature on the formation of ultrafine grain microstructure and in particular on the course of recovery process was studied by use of scanning and transmission electron microscopy.

2. Material and experimental

In this work, the commercial low-carbon steel AISI 1010 was used for experiments. The chemical composition of the steel is shown in Table 1. The experimental steel was received as a rolled-down plate. Prior to ECAP pressing, a conventional austenitization of square shaped billets at the temperature of 920 ℃ for 1 hour was carried out, followed by air cooling. From the treated plates the cylindrical billets with initial diameter of 9 mm and length of 50 mm were cut off for the ECAP experiment. The ECAP pressing was performed at four different temperatures of 150, 200, 250 and 300 ℃. The angle of intersection of the two channels (φ) was equal to 90°. The ECAP die used for the experiment was heated to pressing temperature and held for 30 minutes. The heating of sample for 300 s prior to pressing was done inside the pre-heated die until samples reached the pressing temperature. A 250-tonne hydraulic press was employed and pressing rate of 16 $\text{mm}\cdot\text{s}^{-1}$ was used. The temperature of the die was controlled within the range of ± 1 ℃. Each billet was pressed up to a total of three passes (N) through the die; the billet was rotated between the consecutive passes about its longitudinal axis by 90°

Table 1. Chemical composition of AISI 1010 steel in weight percent

Element	C	Mn	Si	P	S	Al	N	As	Cu
wt.pct	0.1	0.42	0.08	0.029	0.05	0.002	–	0.032	0.02

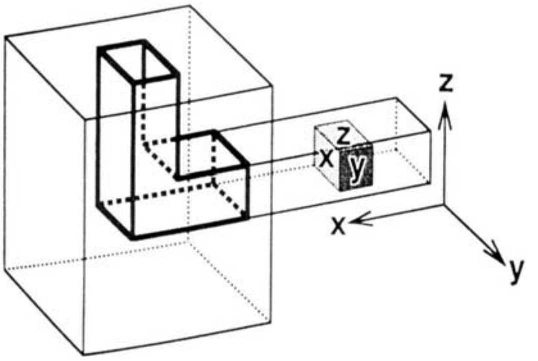

Fig. 1. Principle of ECAP processing.

always in the same direction. Three passes correspond to the total strain of $\varepsilon \sim 3$. This procedure is generally termed processing route B_c and it was selected because it leads most rapidly to formation of homogeneous microstructure of equiaxed grains separated by high-angle grain boundaries [25, 26]. It was not expected that stress generated in sample after each pass should be lowered due to static polygonization upon holding in die between passes.

The microstructure of processed samples was examined by NIKON 200 optical microscope (OM), JEOL JSM 6380 (SEM), and JEM-2000 FX (TEM) microscopes. The samples were sliced normal to the longitudinal axis of ECAP pressed billets. The specimens for optical microscopy were mechanically polished to a 0.05 μm finish and etched using a 3% Nital solution. Micrographs were taken at a location in the distance of 1/3 of diameter from front edge of the sample. Sample selection and preparation for microstructural analysis in the SEM and TEM were the same as those for the optical metallography. The TEM samples were produced by mechanical polishing to the thickness of about 50 μm, followed by electropolishing in the mixture of perchloric acid (10%) and acetic acid (90%) at room temperature and at the voltage of 12 V. Observations in the OM and TEM were made on the transverse plane X which lies perpendicular to the longitudinal axis of the billet as shown in Fig. 1. The selected area diffraction was use to evaluate the structure development at different temperatures of the ECAP process.

Microhardness was measured using a Vickers microhardness tester with the load of 1000 g. The microhardness distribution over the cross-section of the sample was measured. Mechanical properties were determined using ZWICK universal testing machine equipped with a Multisens extensometer. Tensile specimens 50 mm in length were cut off from the ECAP billets with gauge length of 20 mm and 3 mm in diameter. Tensile tests at a constant crosshead speed of 0.016 mm$\cdot$s^{-1} and running until failure were carried out. The engineering stress-strain curves were constructed.

3. Experimental results and discussion

3.1. Microstructural observation after ECAP

The analysis of billet microstructure initial prior ECAP showed that equiaxed ferrite grain morphology is uniform across the billet after applied solutioning at 920 °C. The cementite particles are precipitated along grain boundaries. Figure 2 represents an OM micrograph of initial ferrite microstructure of the steel. The mean linear intercept size of larger and smaller ferrite grains were $\sim$ 100 μm and $\sim$ 10 μm, respectively.

The use of repetitive pressing through the ECAP die provides an opportunity to develop different microstructures by rotating the samples between consecutive passes. Deformation characteristics for the chosen processing route B_c have been analysed in detail in sections normal to longitudinal axis. Both OM and SEM micrographs of the as-pressed steel billet provide evidence of effective straining.

Representative optical micrographs, taken on the X plane, are shown in Fig. 3. The effect of strain non-uniformity across the plane X upon three-fold die pressing is apparent in structure forming regardless of the ECAP process. On this plane, there are still some areas with equiaxed grains present. That can be the evidence that they did not experience the heavy strain of $\sim$ 3 as could be expected in route B_c. This is

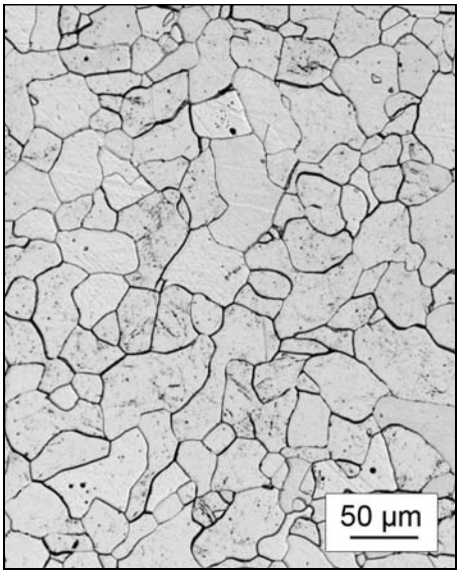

Fig. 2. Optical micrograph of nitial structure in billets after thermal treatment.

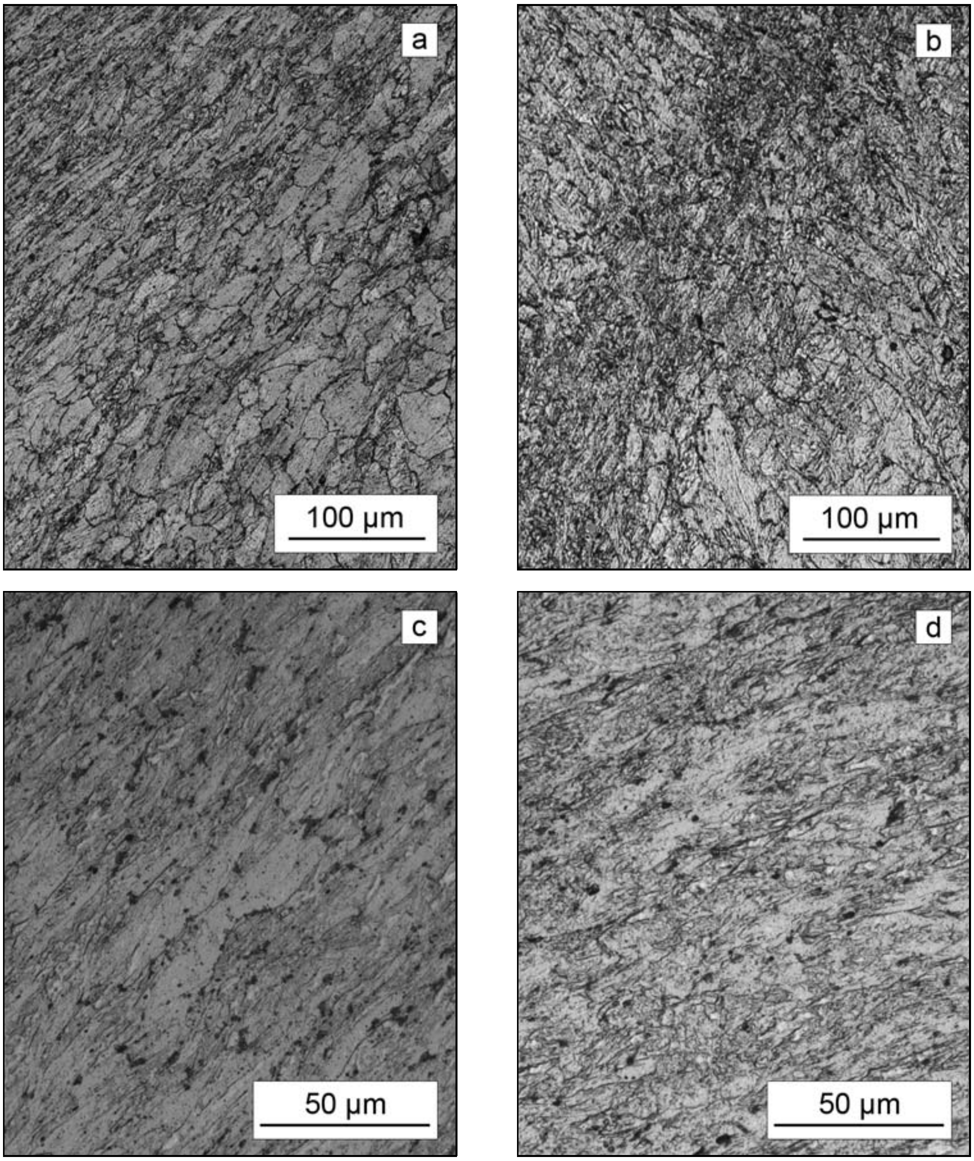

Fig. 3. Optical micrographs taken on the plane perpendicular to billet axis after three ECAP passes at temperature: a) 200 ℃; b) 200 ℃; c) 150 ℃; d) 250 ℃.

contrary to results observed in low-carbon steels and presented in [25, 26].

To examine the effect of deformation conditions on structure development in low-carbon steel during warm ECAP, SEM observation was used as well. The tendency to flow localization for two selected temperatures of 150 and 300 ℃, which are related to the lower and higher temperature of ECAP, can be noticed in Fig. 4. On polished and etched surfaces, the banded morphology of severely elongated grains is clearly visible, Fig. 4a. Very fine lamellar structure of elongated grains in one direction or irregularly bent lamellar structure (Fig. 4b) is observable on the surface. This fact, however, confirms that the sample experienced heavy deformation over the three passes. There is no doubt that in sample microvolumes the deviations due to non-homogeneous strain are present, which can affect the local structure formation and structure uniformity.

As pointed out previously, a multi-pass ECAP produces remarkably uniform microstructure if the number of passes is higher than three and if the angle of intersection of channels is $\varphi = 90°$. However, in this experiment non-uniformity in strain distribution was observed by OM and confirmed by SEM analysis. The microstructure of samples subjected to warm ECAP of 3 passes was further investigated on their normal planes (Fig. 1, X plane) by TEM. This analysis provided the substantial evidence that at the time of structure formation not only structure the fragmentation process modified the new ultra-fine grain structure but also the in-situ recovery processes contrib-

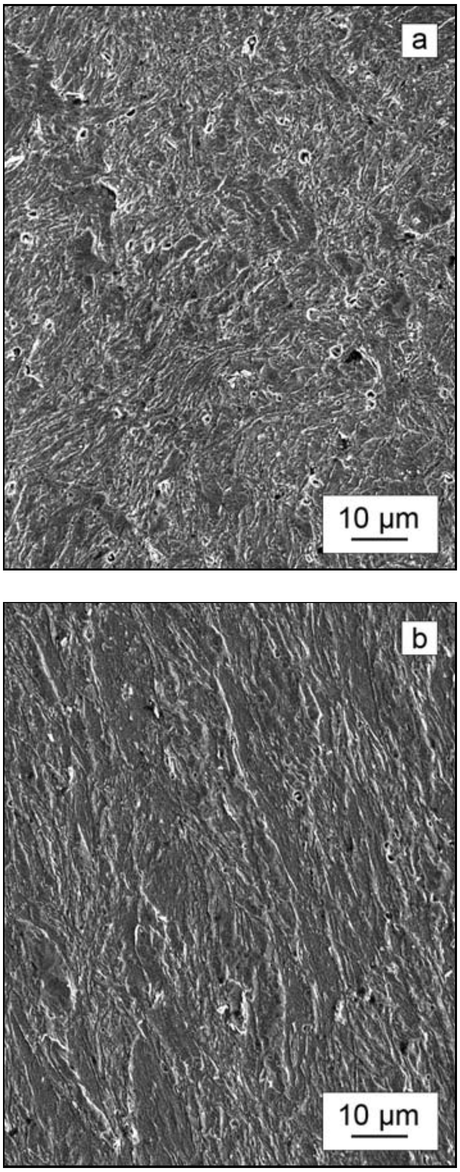

Fig. 4. SEM micrograph of the low carbon steel subjected to three ECAP passes: a) $T_{\mathrm{ECAP}} = 150\,^{\circ}\mathrm{C}$; b) $T_{\mathrm{ECAP}} = 300\,^{\circ}\mathrm{C}$.

uted a great deal to development of ultra-fine grain structure. The TEM provided an opportunity to analyse the changes in the structure, taking place during ECAP pressing on submicron level.

Figure 5 shows the corresponding TEM image of the deformed ferrite upon ECAP pressing at temperature of 150 ℃. For the most part, the microstructure consists mainly of parallel bands of elongated grains. The non-uniform grain size and morphology are presented in Fig. 6. The effect of elevated temperature on the onset of recovery has not been observed. High dislocation density and dislocation cells inside elongated grains are apparent.

The structure characteristics were observed not to have changed substantially upon ECAP pressing at the temperature of 200 ℃. However, the substructure characteristics depend on the local position. In some elongated ferrite grains, dislocation activities can be related to progress in polygonization and preliminary nucleation of new subgrains, Fig. 7. The fringe contrast along grain boundaries of elongated subgrains and small grain nuclei is a strong evidence of continuous recovery, probably dynamic recovery, in time of ECAP pressing and/or onset of dynamic recrystallization. Difraction pattern from a selected area of 1 μm indic-

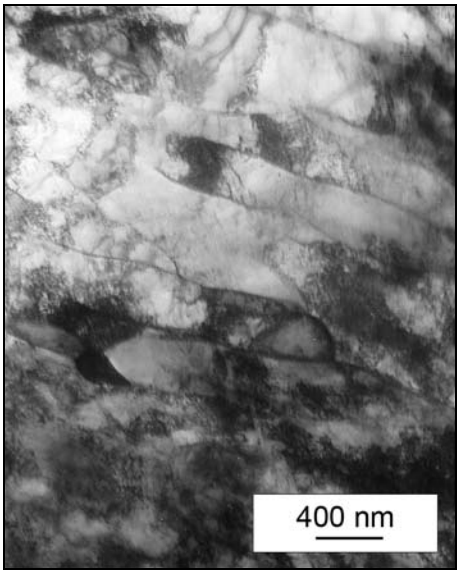

Fig. 5. TEM micrograph of elongated subgrains of ferrite after ECAP at 150 ℃.

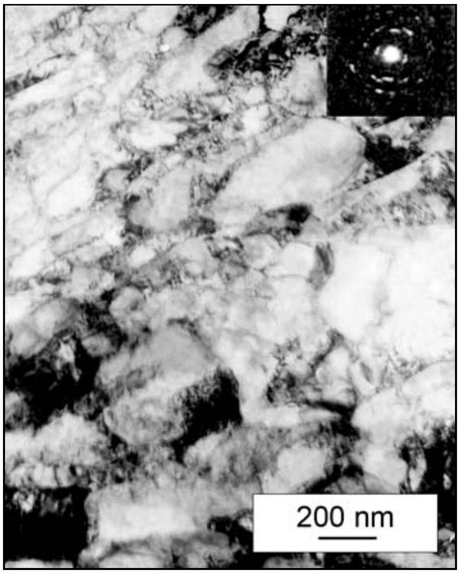

Fig. 6. Different size of fragmented ferrite subgrains produced at ECAP of 150 ℃.

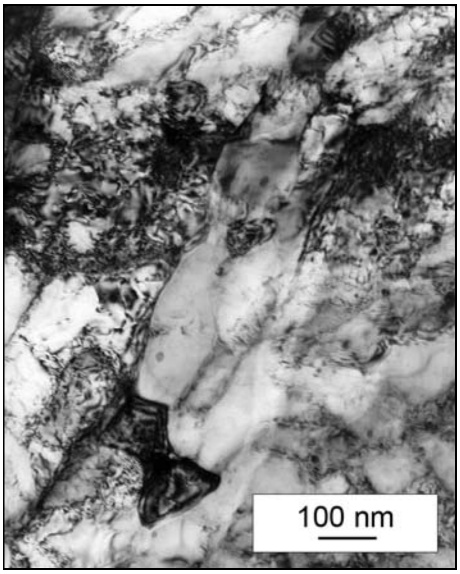

Fig. 7. TEM micrograph of new subgrains formation at ECAP temperature of 200 ℃.

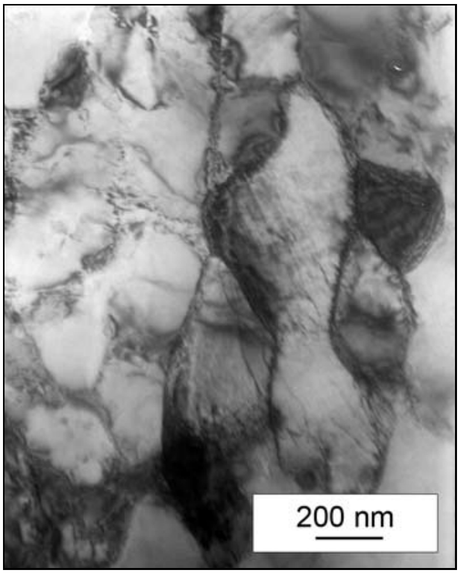

Fig. 8. TEM micrograph of new subgrains experienced dynamic recovery.

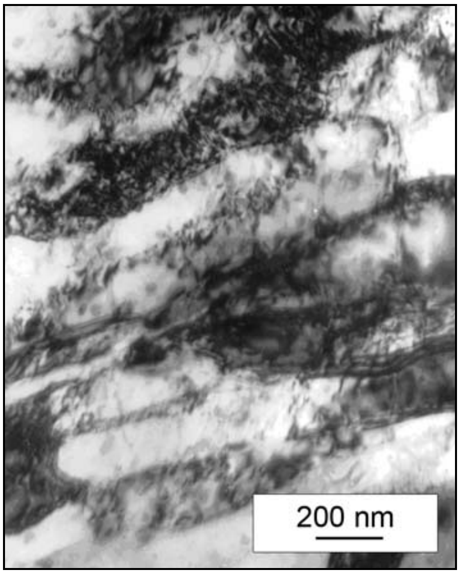

Fig. 9. TEM micrograph of newly born subcrystalline structure.

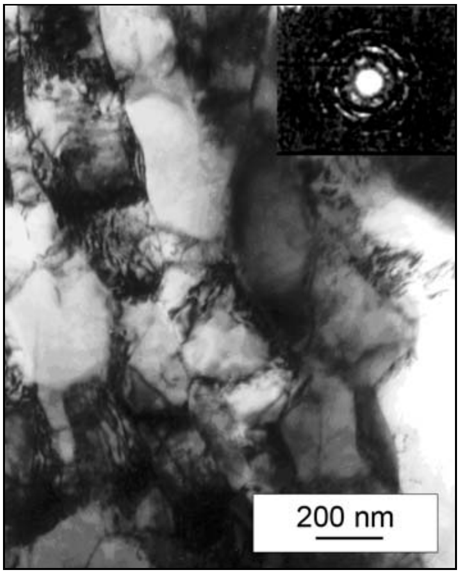

Fig. 10. TEM micrograph of well defined polygonal subcrystalline grains.

ates notable change in the angular spread of the spots.

As the temperature of warm ECAP is raised ($T_{\mathrm{ECAP}} = 250$ ℃), the tendency for development of submicrocrystalline structure becomes stronger, which can be attributed to in-situ dynamic polygonization and recrystallization process. As a result, new subgrains form in clusters and the discernible dislocations inside subgrains are building the dislocation networking, Fig. 8. These subgrains can act as nuclei at formation of submicrocrystalline structure. The more grown and already equiaxed grains with less dislocations can be seen in Fig. 9. This time, the ECAP was performed at the temperature of 300 ℃ and the triple effect of working temperature, introduced strain and latent heat generated by severe deformation acts as effective driving force for dynamic recrystallization process, which in local areas supported the formation of polygonal recrystallized submicron grains, Fig. 10. The presence of net pattern in SAED suggests the presence of a reasonable portion of boundaries having high angles of misorientation.

Table 2. Microhardness of initial and ECAP samples

ECAP$_{\text{tempr.}}$	Annealed	150 °C	200 °C	250 °C	300 °C
HV 1$_{\text{edge}}$	87	239.5	233.6	235.7	242
HV 1$_{\text{center}}$	86.5	238	233	228	240

Table 3. Mechanical properties variation in dependence of the ECAP temperature

T_{ECAP} (°C)	YS (MPa)	UTS (MPa)	A (%)	RA (%)
150 (1)	562.4	824.3	11.4	37.8
200* (2)	814.1	818.9	8.8	36.1
250 (3)	680.3	779.7	13.6	38.8
300 (4)	662.3	760.9	13.0	35.0
Initial state	252.0	307.0	38.2	–

*all data influenced by structure defect; ( ) curve

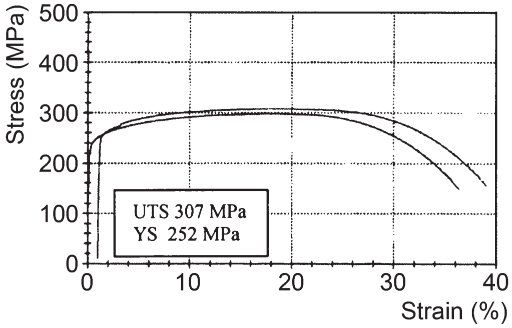

Fig. 11. Engineering stress-strain curves of solutioned steel.

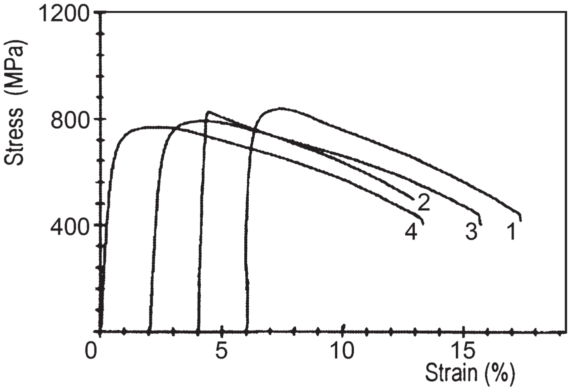

Fig. 12. Stress-strain curves of ECAPed samples.

3.2. Mechanical properties of steel after ECAP

In order to examine the effect of ECAP temperature the Vickers hardness HV 1 was measured prior to and after ECAP on the plane perpendicular to the pressing direction (X area). 1 kg load applied for 10 s was used for the measurement. The hardness values were taken as the average of a minimum of 3 measurements. The records are stated in Table 2. The hardness variation with increasing temperature of ECAP is seen, and a little unexpected is the value at the highest temperature of ECAP 300 °C, where the most advanced effect of dynamic recrystallization on submicrocrystalline structure formation was observed.

The results of tensile testing at room temperature using an initial crosshead rate of 5 mm/min for samples are shown in Fig. 11, for fully annealed condition, and in Fig. 12 for ECAP specimens. In case of the fully annealed condition, there is an extensive period of strain hardening and a high elongation to failure. The deformation behaviour of ECAP specimens is very similar in all specimens where tensile strength is decreasing as ECAP temperature increases. However, a little different behaviour is observed in the specimen 3, which does not exhibit any work hardening following yielding. After reaching a maximum strength at a small strain a continuous drop in stress-strain curve occurs. The amount of uniform deformation is, therefore, very small and the stress-strain curve is similar to that anticipated in a work hardening. (The analysis of fracture confirmed that the deformation process was influenced by interior crack present in specimen. On the other side it is noticeable that the reduction of area is, however, similar as in other samples, Table 3.) Generally, the obtained results confirm the considerable increase of tensile strength as compared to that of annealed steel. The yield stress is more than twice higher, reaching the maximum value of 680 MPa at $T_{\text{ECAP}} = 250$ °C. The region of strain hardening prior to the softening is visible and the amount of uniform elongation is increasing with increasing temperature of ECAP. However, for the strength properties (UTS and YS) decrease for all temperatures is observable, while the length of uniform elongation shows only small extension. The decrease of the UTS with increasing temperature can be attributed to the effective dynamic recrystallization process resulting in formation of submicrocrystalline microstructure where increased fraction of submicron grains in structure is apparent. The resulting volume fraction of newly recrystallized grains with dislocations network inside is still not yet prepared to recovered plastic ability in ECAPed specimens.

4. Conclusion

Microstructural evolution during warm ECAP pressing was studied in low carbon steel with ~ 0.1 wt.% C steel. The major results can be summarized as follows:

1. The ECAP processing route B_c was performed at four different elevated temperatures and the billets were pressed in three passes. Intensive and yet non-uniform strain in the billets, excluding the end regions, was observed by OM.
2. Formation of heavily deformed substructure was apparent in samples investigated by TEM analysis. In elongated ferrite grains the substructure consisting of dislocation cells and subgrains was found.
3. The ECAP conducted at elevated temperatures was apparent to support and accelerate the process of polygonization of deformed structure, which was observed at structure recovery at the ECAP temperature of 200 °C and higher.
4. At the highest ECAP temperature of 300 °C, the process of dynamic recrystallization effectively transformed the elongated structure of ferrite and contributed to formation of the stable submicrocrystalline structure, which resulted in strength properties decrease but partly recovered the plastic ability of ECAPed steel.

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