GRAIN REFINEMENT OF LOW CARBON STEEL BY ECAP SEVERE PLASTIC DEFORMATION

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Abstract

The aim of this work is to evaluate the effect of initial structure modification and increased temperature on ultrafine grain structure formation of commercial low carbon steel AISI 1014. Low carbon steel was subjected to a severe plastic deformation technique know as Equal Angular Channel Pressing (ECAP) at different increased temperatures. The steel was subjected to ECAP with channel’s angle $\phi$ 90° at different temperature temperatures, in range of 150- 400°C. The number of passes at each temperature was N-3 and 4. The impact of different effective strain $\varepsilon_{\text{ef}}$ introduced upon microstructure changes was investigated using light, scanning (SEM) and transmission electron microscopy (TEM) methods. The microhardness results (HV) along the deformed bars supported the deformation straining effect. Repeating deformation procedure (Bc) the coarse and modified ferrite grains were refined and resulting structure consisted of the of fine grains having high angle boundaries. The features of grain refinement appeared in structure when effective strain $\varepsilon_{\text{ef}}$ reached value of 3 and 4. On the other side the heterogeneity in ultrafine grain structure formation was evident locally. Regardless the initial structure of steel and applied deformation conditions the applied effective strain was insufficient to ensure uniform deformation across and the length of steel bars. The size of newly born polygonal subgrains and new grains was in range of 300 - 600 nm. The tensile tests results confirmed strength increase of the steel. The formation of such predominant submicrocrystalline grain structure resulted in significant increase of the yield and tensile strength of the steel; however the restricted ductility after straining resulted.

Keywords: low carbon steel, severe plastic deformation, structure refining, strength and ductility.

INTRODUCTION

During the recent decade, bulk ultrafine grain structured materials produced by severe plastic deformation (SPD) have been investigated intensively. The production of fine grained materials by SPD, leads to a large number of investigations focusing on the substructure development and related mechanical properties. It has been already well known that SPD of metallic materials, involving processes such as equal angular pressing (ECAP), accumulative roll bonding (ARB), and high pressure torsion (HPT) and others methods, is capable of producing ultrafine grain (UFG) materials with submicrometer, even less to nanometer grain size [1, 2]. The fabrication of bulk materials with ultrafine grain sizes has attracted a great deal of attention over past two decades because of their enhanced properties [3 - 6]. The term ultrafine grain structure is referring to nanostructure with grain size less then 100 nm, and submicrocrystalline structure with grains between 100 to 1000 nm. In recent years it has become a worldwide effort to develop manufacturing process to obtain ultrafine grain structure in steels. However, materials with ultrafine grained structure, manufactured by the SPD process have the inherent limit for their practical use. Since the SPD accumulates extensive internal energy inside materials, considerable residual stress would still remain even after a large portion of internal energy is dissipated for grain refinement.
Currently, significant interest has shifted to the use of warm and/or even hot severe deformation in order to produce more stable UFG microstructure [7]. With cold ECAP, low and medium carbon steels can only be pressed by two or three passes with channel intersection of 90° before initiation of sample failure. The two to four passes realized with cold ECAP are insufficient and the achievable strain amount is insufficient to produce a fully refined grain structure [8]. To form stable ultrafine grain structure in metals and alloys, ECAP deformation should be carried out at increased temperature corresponding to the temperature of cold working [9].

The purpose of this work is to study the formation of submicrocrystalline structure in commercial low carbon steel AISI 1014 subjected to the large strain during warm deformation in dependence of varying temperature and strain level of ECAP performed. The influence of the temperature on the formation of ultrafine grain microstructure and in particular on the course of recovery process was studied.

**EXPERIMENTAL PROCEDURE**

**MATERIAL AND EXPERIMENTAL**

In this work, the commercial low carbon steel AISI14 was to use for experimental. The chemical composition of the steel is shown in Table 1. The experimental steel was received as rolled down plate. Prior ECAP pressing, a conventional austenitization of square shaped billets at temperature of 920°C for 1 hour was carried out, followed by air cooling. The initial microstructure of ferrite with scattered small cementite islands is presented in Fig. 1. From thermally treated billets the cylindrical specimens with initial diameter of 9 mm and length of 50 mm were cut off for the ECAP experiment. The ECAP pressing was performed for four different temperatures of 150, 200, 250 and 300°C. The of intersection angle of the two channels (φ) was equal to 90°. The ECAP die used for experimental was heated to the consecutive pressing temperature and held on the temperature for 30 minutes. The samples heated for 300 s prior pressing was done inside the pre-heated die until samples reached the pressing temperature. A 250 tons hydraulic press was employed and pressing rate of 16 mm/s was used. The temperature of the die was controlled within the range of ± 1°C. 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° always in the same direction. Three passes correspond to the total strain of ε ~ 3. This procedure is generally termed processing route Bc and it was selected because it leads most rapidly to formation of homogeneous microstructure of equiaxed grains separated by high angle grain boundaries. 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 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,

**Table 1. Chemical composition of AISI 1014 steel in weight percent.**

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>N</th>
<th>As</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt.pct</td>
<td>0.1</td>
<td>0.42</td>
<td>0.08</td>
<td>0.029</td>
<td>0.05</td>
<td>0.002</td>
<td>-</td>
<td>0.032</td>
<td>0.02</td>
</tr>
</tbody>
</table>

**Fig. 1.** Principle of ECAP processing.
followed by electropolishing in the mixture of perchloric acid (10%) and acetic acid (90%) at room temperature and at the voltage of 12 V. Observation 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 applied to specify the structure development at different temperatures of the ECAP process. Microhardness was measured using a Vickers tester with the load of 100g. 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 3mm in diameter. Tensile tests at a constant crosshead speed of 0.016 mm.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 microstructure 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 Fig. 2 represents an OM micrograph of initial ferrite microstructure of the steel. The mean linear intercept size of larger and smaller ferrite grains were \(\sim 100 \, \mu m\) and \(\sim 10 \, \mu m\) 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 analyzed in detail in sections normal to longitudinal axis. Both OM and SEM micrographs of the as-pressed steel provide evidence of effective straining.

Representative optical micrographs for T- 150°C, 200°C, 250°C and 300°C taken on the X plane are shown in Fig. 3 (a,b,c,d). 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 contrary to results observed in low carbon steels and presented in [9, 10].

In order to examine the effect of deformation conditions on structure development in low carbon steel during warm ECAP and SEM observation was used as well. The tendency to flow localization for two selected temperatures of 150 and 300°C, 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 $\phi = 90^\circ$. However, non-uniformity in strain distribution was observed by OM. The microstructure of samples subjected to warm ECAP at 300°C 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 the structure but also the in-situ recovery processes contributed a great deal to development of ultra fine grain structure. The TEM provided an opportunity to analyze the changes in the structure taking place during ECAP pressing on submicron level.

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

Fig. 3 c.d. Optical micrographs taken on the plane perpendicular to billet axis after three ECAP passes, T = c) 250°C; d) 300°C.

Fig. 4 SEM micrograph of the low carbon steel subjected to N=3 ECAP passes: a) $T_{\text{ECAP}} = 150^\circ\text{C};$ b) $T_{\text{ECAP}} = 300^\circ\text{C}.$

Fig. 5. a) TEM micrograph of elongated subgrains of ferrite after ECAP at 150°C; b) Different size of fragmented ferrite subgrains produced at ECAP of 150°C.
The structure characteristics were observed not to have changed substantially upon ECAP pressing at the temperature of 200°C. 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. Diffraction pattern from a selected area of 1μm indicates notable change in the angular spread of the spots.

As the temperature of warm ECAP is raised \( T_{ECAP} = 250°C \) the tendency for development of submicrocrystalline structure becomes stronger, which can be attributed to in situ dynamic polygonization and recrystallization. As a result, new subgrains form in clusters and the discernible dislocations inside of subgrains are forming 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°C 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.

### 3.2. Mechanical properties of steel after ECAP

In order to examine the effect of ECAP temperature the Vickers hardness HV1 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
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 $5\text{ mm/min}$ 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 deformation process was influenced by interior cracks present in specimens. On the other side it is noticeable that the reduction of area is, however, similar in others specimens, see 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 $680\text{ MPa}$ at $T_{\text{ECAP}} = 250^\circ\text{C}$. 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) is observable decrease for all temperatures, while length of uniform elongation shows only small extension. The increase of the UTS 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.

**Table 2. Microhardness of initial and ECAP samples**

<table>
<thead>
<tr>
<th>$T_{\text{ECAP}}$</th>
<th>annealed</th>
<th>150°C</th>
<th>200°C</th>
<th>250°C</th>
<th>300°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>HV1$_{\text{edge}}$</td>
<td>87</td>
<td>239.5</td>
<td>233</td>
<td>235</td>
<td>242</td>
</tr>
<tr>
<td>HV1$_{\text{center}}$</td>
<td>86.5</td>
<td>238</td>
<td>233</td>
<td>228</td>
<td>240</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>$T_{\text{ECAP}}$ [°C]</th>
<th>YS [MPa]</th>
<th>UTS [MPa]</th>
<th>A [%]</th>
<th>RA [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>150</td>
<td>562</td>
<td>824</td>
<td>11.5</td>
<td>38</td>
</tr>
<tr>
<td>200</td>
<td>814</td>
<td>819</td>
<td>9</td>
<td>36</td>
</tr>
<tr>
<td>250</td>
<td>680</td>
<td>779</td>
<td>13.5</td>
<td>39</td>
</tr>
<tr>
<td>300</td>
<td>662</td>
<td>761</td>
<td>13</td>
<td>35</td>
</tr>
<tr>
<td>initial state</td>
<td>252</td>
<td>307</td>
<td>38</td>
<td>58</td>
</tr>
</tbody>
</table>
4. CONCLUSIONS

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 Bc 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 optical microscopy.

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 temperature was apparent to support and accelerate the process of polygonization of deformed structure, which was observed at structure recovery at the lowest ECAP temperature of 150°C.

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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LITERATURE