Effect of Severe Plastic Deformation on microstructure and properties of duplex stainless steel


The excellent performance of duplex and super duplex stainless steels due to their high corrosion resistance and high mechanical strength is directly related to their austeno-ferritic microstructure. However, these steels may suffer the formation of secondary brittle phases when they reach temperatures between 600°C and 950°C causing catastrophic service failure of components. In order to understand the influence of the mechanical history of the steel, the equal channel angular pressing was applied on UNS S32750 bar samples. ECAP is processed by using a hydraulic press (DE-80). Microstructural characterization was carried out on the severe plastic deformed samples by means of OM, SEM and EBSD. Ultimate tensile strength has improved with a low decreasing of elongation after the first pass. The ECAP process resulted to produce a faster precipitation of secondary phases if compared with the non-ECAP samples.

KEYWORDS: DUPLEX STAINLESS STEELS, SEVERE PLASTIC DEFORMATION, ECAP, GRAIN REFINEMENT

INTRODUCTION

Severe plastic deformation (SPD), as large strain processing of metallic materials, is used and investigated mainly to obtain superior mechanical properties, achieved by a structural state with ultrafine grains on a sub micrometer scale (1 μm up to less than 100 nm). Moreover, such ultrafine grain materials, if reasonably stable at elevated temperature can exhibit superplastic forming capabilities.

The structural changes produced by the SPD and leading to the sub microcrystalline states, occur mainly by the formation of dislocations sub boundaries like dense dislocations walls. Increasing the deformation, the sub boundaries misorientations also increase to values typical for conventional high-angle grain boundaries, finally producing the microcrystalline state [1-3].

The study of structural modifications during SPD of a dual-phase alloy, like the duplex stainless steels (DSS) [4], is interesting by several aspects. In such dual-phase (ferritic-austenitic) alloys, interphase boundaries can be used like markers of different mechanism operating in the transformations of the two different phases during SPD, providing important information about changes in the phase shapes and its mutual arrangement, about different plastic flow of the two phases during deformation, etc. Moreover, such studies could give information about other possible transformations during and after SPD, like martensite formation from austenite phase, dangerous phase (sigma and chi) precipitation, about the behavior of the alloy during severe extrusion processing, etc. [5-8].

In this work, the SPD produced by ECAP in the 2507 SDSS has been examined. This grade was chosen by the stability of its austenitic phase against martensitic transformation, to analyze the sub microcrystalline structure formation and evolution during ECAP processing, in both the starting phases ferrite and austenite.

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MATERIALS AND EXPERIMENTAL PROCEDURE

The as received material was a wrought 2507 SDSS rod (20 mm), in the solution-annealed condition (1120°C, W.Q.), with the composition reported in Tab. 1. Samples for experiments have been machined to required dimensions of Ø5mm diameter and 30mm in length.

**Tab. 1 - Chemical composition of SDSS steel (in %wt).**

<table>
<thead>
<tr>
<th></th>
<th>Cr</th>
<th>Mo</th>
<th>N</th>
<th>C</th>
<th>Ni</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Co</th>
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<tr>
<td></td>
<td>24.90</td>
<td>3.623</td>
<td>0.27</td>
<td>0.0463</td>
<td>6.55</td>
<td>0.530</td>
<td>0.0254</td>
<td>0.0113</td>
<td>0.624</td>
<td>0.079</td>
</tr>
</tbody>
</table>

ECAP was processed by using a hydraulic press (DE-80), which has 80 tons capacity and equipped with an electronic system which controls the speed of plunger, pressure, travel length of plunger. Speed of plunger was displayed in an electronic display unit. Molybdenum disulfide (MoS2) was sprayed regularly inside the channel to reduce the friction. The scheme of the used process can be found in Fig. 1.

Samples for light and electron microscopy examinations were cut longitudinally and transversely, then prepared using standard metallographic procedures which includes grinding with silicon carbide (SiC) paper, then polished by using diamond suspension. Specimens were electrochemically etched in 30 wt% KOH solution with etching potential of 2V for 15 sec. For EBSD analysis specimens, in addition to previous procedure, it is necessary to include a final polishing stage using colloidal silica (0.05μm) for about 2 hours, to decrease the roughness in nanoscale. EBSD analysis have been obtained using a scanning electron microscope FESEM JEOL J-71000F with a coupled EBSD detector and the post processing software from Oxford Inst. The tensile test was carried out on short specimens made from ECAP processed billet. In order to analyze the influence of ECAP process on the precipitation kinetics one non-ECAP sample and one sample after ECAP were heat treated for 5 min at 830°C and analyzed by SEM in order to evaluate the type and number of secondary phases formed during the isothermal heat treatment.
RESULTS AND DISCUSSION

Hardness

To analyze the mechanical properties, micro hardness tests were performed using a Vickers micro hardness tester by applying a load of 1Kg over a period of 20s for both as received material and processed samples. The results are summarized in Fig.2.

It was observed that hardness increased after each pass. This increase in hardness can be attributed to the high increasing in dislocation density and grain refinement due to the high strain induced during ECAP processing. The hardness has improved nearly 23% after the first pass whereas only 10% after the second pass. There is a huge increment of hardness after the first pass and a smaller one after the second pass.

Fig. 2 - Hardness improvement with respect to number of passes

Tensile test

The stress-strain curves of the sample without ECAP and of the sample with one pass of ECAP are reported in Fig.3 and the extrapolated values of yield stress, UTS and elongation are summarized in Tab.2. It can be observed, as expected, a remarkable increase in the resistance but a reduction in the ductility after the ECAP process. ECAP-processed DSS have relatively low ductility but they usually demonstrate significantly higher strength than their coarse-grained counterparts. It is important to note that SPD processing leads to a reduction in the ductility which is generally less than in conventional deformation processing techniques such as rolling, drawing and extrusion. Processing by ECAP leads to a greater retention of ductility than cold-rolling [7].

Fig. 3 - Engineering stress-strain curve for 0 and 1 pass ECAPed samples
Stainless steel & duplex

Tab.2 - Micro tensile test results

<table>
<thead>
<tr>
<th></th>
<th>0 Pass</th>
<th>1 Pass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield stress (YS) [N/mm²]</td>
<td>864.62</td>
<td>1219.17</td>
</tr>
<tr>
<td>Ultimate tensile strength (UTS) [N/mm²]</td>
<td>900.02</td>
<td>1310.06</td>
</tr>
<tr>
<td>Fracture Strength [N/mm²]</td>
<td>414.99</td>
<td>667.44</td>
</tr>
<tr>
<td>Percentage elongation (%)</td>
<td>43.37</td>
<td>28.47</td>
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Microstructural analysis

The microstructure of as received material, reported in Fig.4, shows that it contains almost 55% of austenite and 45% of ferrite. The longitudinal section microstructure shows that the grains are elongated towards rolling direction.

Deformation bands are observed in first pass as well as in the second pass as shown in Fig. 5. Deformation bands are common in multiphase metals deformed to high strains. It is basically caused by local plastic instabilities when work-hardening becomes exhausted. But there is no perfect explanation about the microstructural mechanism. In DSS, ferrite and austenite have very different behaviors during plastic deformation. Ferrite presents dislocation cells, while austenite presents slip bands. Strain hardening also occur in different ways in both phases and those are probably the root causes for the deformation bands. Optical microscope images Fig. 5 clearly shows that the spacing of deformation bands decreases with increasing deformation, indicating that grain subdivision continues to refine at high strain rate.

**Fig. 4** - Microstructure of studied DSS in (a) longitudinal section (b) transverse section
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Fig. 5 - Deformation bands observed after 1 and 2 pass in optical microscope

The samples before and after the ECAP process were also observed with scanning electron microscope (SEM). The SEM analysis confirm, for the non ECAPed sample the information obtained with OM with the presence of 55% of austenite and 45% of ferrite. The micrographs obtained after 1 pass of ECAP in the longitudinal section are reported in Fig.6.

Fig. 6 - Microstructure of SDSS longitudinal section after one pass ECAP. a) Polygonization, austenite rotation b) large irregularities and c) small irregularities in the interphase boundaries are clearly shown.
As can be observed no precipitation of secondary phases can be detected. So ECAP process without heat treatment do not produce the precipitation of phases. SEM characterization of the ECAPed samples shows evidence of phase rotation that involves the change of the ferrite-austenite interphase boundaries character hence it can be assumed that the initial orientation relationship (K-S or N-W) suffers a lattice rotation too, modifying the interphase boundary coherency mainly if ferrite develops sub grains. Apart from lattice rotations taking place on a sub structural scale, macroscopic phase rotations are noticed too. Austenite can rotate as a whole within the ferrite matrix, which has simultaneously experienced intense local shearing. Moreover, due to shear strain, voids in the austenite phase are formed (and filled by ferrite penetration) causing the final longitudinal fragmentations of austenite.

EBSD analysis
The EBSD technique has found a wide field of applications in multiphase materials like duplex stainless steels, whereby using this method the nature of ferrite, austenite and any intermetallic phases can be identified. ECAP specimens were examined by EBSD in the longitudinal direction.

**Fig. 7** - IPF map with misorientations greater than 3°. Elongated as well as equiaxed grains are seen in the image. Also stereographic triangle, which indicates the orientations of the grains in the IPF map. (a) 0 Pass (b) Pass (c) 2 Pass.

IPF color maps in Fig. 7 clearly shows that there is huge grain refinement near the deformation band. After 1 and 2 pass microstructure contains equiaxed grains at deformation bands and elongated grains next to the deformation band.

There is an enormous change in average grain size after first pass. It is about 0.9μm. After the second pass the average grain size is about 0.4μm. This shows that there is a grain refinement at each and every pass. Grain size of 0 pass is about 8.5 microns as can be seen in Fig.8 which is quite large in comparison to the ECAPed samples. Nevertheless a grain size of 8.5 microns it’s quite small in a conventional metallic alloy and it is due to the fact that the presence of one phase hinder the growth of the other and vice versa.
Grain refinement during ECAP may be attributed to the deformation banding that inevitably occurs during the process and has two possible origins. First: deformation bands originate due to the ambiguity associated with the selection of the operative slip systems. In many cases, the imposed strain can be accommodated by more than one set of slip systems which lead to different lattice rotations. Second: inhomogeneous straining i.e. different regions of a grain may experience different strains if the work done within the bands is less than that required for homogeneous deformation and if the bands can be arranged so that the net strain matches the overall deformation.

These deformation bands are separated by geometrically necessary boundaries (GNBs). Boundaries are also formed by statistical trapping of dislocations, forming the so-called incidental dislocation boundaries (IDBs). The misorientation of both types of boundaries increases with strain. Furthermore, the spacing of both type of boundaries decreases with increasing deformation, indicating that grain subdivision continues to refine at high strain rate.

Fig. 9 shows the corresponding grain boundary maps in which the high angle grain boundaries (HAGBs) with a misorientation angle above 15° are shown as black lines and boundaries 3° and 15° are shown as red lines.

The microstructure after the first pass shows that low angle boundaries are prevailing, mainly in the austenite. After second pass the microstructure shows low angle misorientation boundaries both in ferrite (BCC) and austenite (FCC). Moreover, as the number of passes in ECAP is increased, there occurs a gradual transition from array of predominantly low-angle grain boundaries (LAGBs) to a predominantly high-angle configuration.

Also in the examined DSS ECAP produces at first grain boundaries misorientations with an excess of low-angle boundaries. This is a recurrent feature of the misorientations distribution after ECAP, as natural consequence of the large numbers of excess dislocations introduced into the billet on each separate passage through the die. However, the tendency for the average boundaries misorientation increase with increasing the number of passes. This could provide an opportunity to make use of ECAP, or other severe plastic deformation facilities, to...
obtain fine grain materials having different, but controlled, boundary misorientation distribution.

Effect of ECAP on the precipitation of secondary phases

The samples with and without ECAP were heat treated at 830°C for 5 minutes in order to induce the precipitation of secondary phases. The SEM micrographs of the samples after the heat treatment can be observed in Fig. 10. The precipitation of the secondary chi and sigma phases occurred after the ECAPed and non ECAPed samples. As expected, the highest precipitation has been detected in the ECAPed samples. This is because precipitation started at austenite-ferrite boundaries, following a longitudinal pattern among ferrite and austenite interphase boundaries. The boundaries between ferrite and austenite stringers formed during shear deformation are preferential sites for secondary phases nucleation. An increase in the number of grain boundaries can be observed after the ECAP process, as was previously described, so the precipitation occurred at shorter dwell time at the same ageing temperature than for non ECAPed specimen.

![Fig. 9](image1)

Fig. 9 - Boundary map which shows low angle grain boundaries and high angle grain boundaries (a) 0 Pass (b) 1 Pass (c) 2 Pass

![Fig. 10](image2)

Fig. 10 - SEM analysis of the sample with and without ECAP after heat treatment at 830°C for 5 minutes
CONCLUSIONS
The main results of the mechanical and microstructural characterization of the DSS steel processed by ECAP can be summarized:
1. Significant increment in hardness after the first pass about 23%. After the second pass the hardness has improved to 489HV, as compared to 363HV for its coarser part.
2. Ultimate tensile strength has improved by 45% after first pass. Percentage elongation decreased from 43% to 28% after the first pass. Engineering stress - strain curves showed increment in strength, reduction in ductility.
3. There is a significant grain refinement, the average grain size after the 2 pass is about 0.4μm, as compared to 8.5μm for its coarser part of 0 pass.
4. EBSD analysis and different maps showed the grain refinement especially near the deformation bands. Spacing of these deformation bands decreases with increase in deformation, indicating that grain subdivision continues to refine at high strain rate. IPF maps of ECAPed samples contains equiaxed grains at deformation bands and elongated grains next to the deformation bands.
5. The kinetic of secondary phases precipitation is faster in thermally treated ECAPed samples than in non ECAPed ones. This precipitation occurred at shorter dwell time at the same ageing temperature than for non ECAPed specimen.

REFERENCES