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Effect of severe plastic deformation on microstructure and properties of duplex stainless steel

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Abstract. 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) is a technique used to process metallic materials with the aim of achieving enhanced mechanical properties through the creation of a structural state characterized by ultrafine grains on a sub-micrometer scale (ranging from 1 μm to less than 100 nm). Furthermore, if these ultrafine grain materials exhibit reasonable stability at elevated temperatures, they can demonstrate superplastic forming capabilities.

The structural changes induced by SPD, which lead to the development of sub microcrystalline states, primarily occur through the formation of dense dislocation walls known as dislocation sub-boundaries. As deformation increases, the misorientation of these sub-boundaries also rises, eventually reaching values typical of conventional high-angle grain boundaries, thereby resulting in the formation of a microcrystalline state [1-3].

Studying the structural modifications that occur during SPD of dual-phase alloys, such as duplex stainless steels (DSS) [4], is intriguing for several reasons. In these alloys, consisting of ferritic and austenitic phases, interphase boundaries can serve as markers for different mechanisms operating in the transformations of the two phases during SPD. This provides valuable insights into changes in phase shapes, their mutual arrangement, and the distinct plastic flow exhibited by the two phases during deformation. Furthermore, such studies can shed light on other potential transformations during and after SPD, including martensite formation from the austenite phase, precipitation of detrimental phases (such as sigma and chi), and the behavior of the alloy during severe extrusion processing [5-8].

This paper examines the SPD achieved through Equal Channel Angular Pressing (ECAP) in the 2507 SDSS grade. This specific grade was chosen due to the stability of its austenitic phase against

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martensitic transformation. The objective is to analyze the formation and evolution of the sub microcrystalline structure during ECAP processing in both the starting ferrite and austenite phases.

Materials and experimental procedures

The material tested shows a composition summarized in Table 1. It was delivered as a wrought rod of 20 mm in diameter in the solution-annealed condition at 1120°C for 1 hour and water quenched. The samples for the experiment had a diameter of 5mm and a length of 30 mm.

Table 1. Chemical composition of SDSS steel.

Cr	Mo	N	C	Ni	Mn	P	S	Si	Co
24.90	3.623	0.27	0.0463	6.55	0.530	0.0254	0.0113	0.624	0.079

The ECAP processes was performed on a hydraulic press DE-80 with 80 tons capacity, electronically controlled (speed of the plunger, pressure, travel length of the plunger). As a lubricant Molybdenum disulfide (MoS2) was used inside the channel.

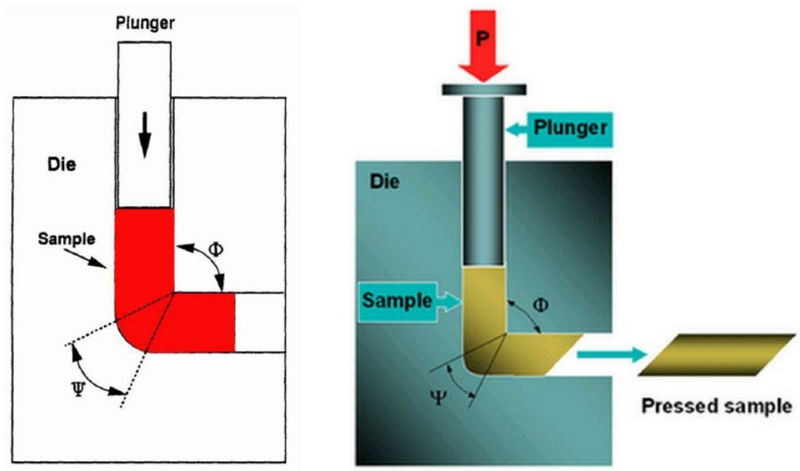

Fig. 1. ECAP worling

Samples for metallographic examination were obtained longitudinally and transversely, grinded with standard metallographic procedures (Silicon carbides paper up to 1200 grit size) and then polished with diamond suspension down to 1 micrometers. Etching was performed with 30wt% KOH solution under 2V for 15 sec. EBSD analysis was additionally performed but the samples had to undergo a further polishing step using colloidal silica (50 nm) for approximately 2 hours. EBSD was performed using and EBSD probe by Oxford Instrument in a field emission SEM FESEM JEOL J-71000F.

Mechanical testing was carried out on undersized specimen from ECAP processed samples. Moreover microhardness were performed with 1 Kg of weight for 20 s in a Vickers microhardness tester.

Results and discussion

Hardness:

As expected due to the extensive coldworking, hardness increased after each pass. The hardness was increased by 23% after the first pass, and only by 10% after the second pass (Fig. 2). This is because hardness will get saturated to a particular value after subsequent passes.

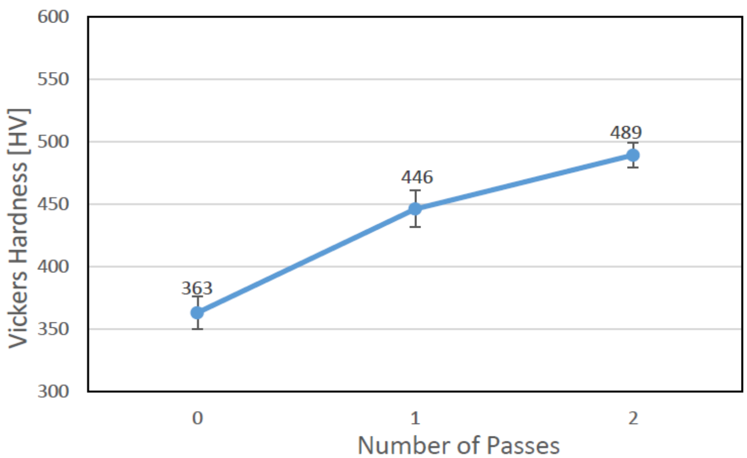

Fig. 2. Hardness improvement with respect to number of passes

Tensile test:

Compared to the coarse-graine counterpart, ECAP-processed SDSSs show significantly higher strength (Fig.3). A reduction in ductility is noted but generally is less compared to the conventional processing techniques such as drawing, rolling or extrusion. The ductility retention of ECAP processed specimens is greater compared to the cold rolled material [7].

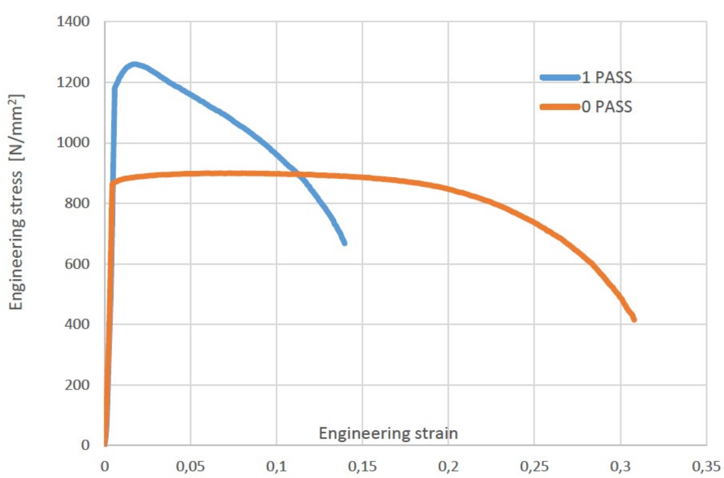

Fig. 3. Engineering stress-strain curve for 0 and 1 pass ECAPed samples

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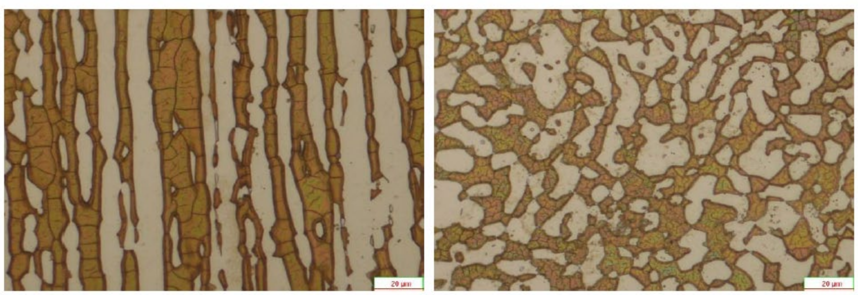
Table 2. Micro tensile test results

	0 Pass	1 Pass
Yield stress (YS) [N/mm^2]	864.62	1219.17
Ultimate tensile strength (UTS) [N/mm^2]	900.02	1310.06
Breaking stress [N/mm^2]	414.99	667.44
Percentage elongation (%)	43.37	28.47

Optical microscopy:
The phase balance of the as received material is almost equal (55±4% austenite and 45±3% ferrite). As can be seen from Fig.4 the microstructure shows elongated grains along the rolling direction, while the transverse section shows mainly equiaxed grains.



(a) (b)

Fig.4. Microstructure of studied DSS in (a) longitudinal section (b) transverse section

From Fig.5 it can be seen that deformations bands are present both after the first and the second pass of ECAP deformation. Those bands are common in multiphase metals deformed to high strain. This is due to the saturation of work hardening that causes local plastic instability. Ferrite and austenite have different plastic deformation behaviors due to their very different high stacking fault energy. Moreover, strain hardening is quite different in both ferrite and austenite which leads to the appearance of deformation bands. Optical microscope images (Fig. 5) clearly show that the spacing of deformation bands decreases with increasing deformation, indicating that grain subdivision continues to refine at high strain rate.

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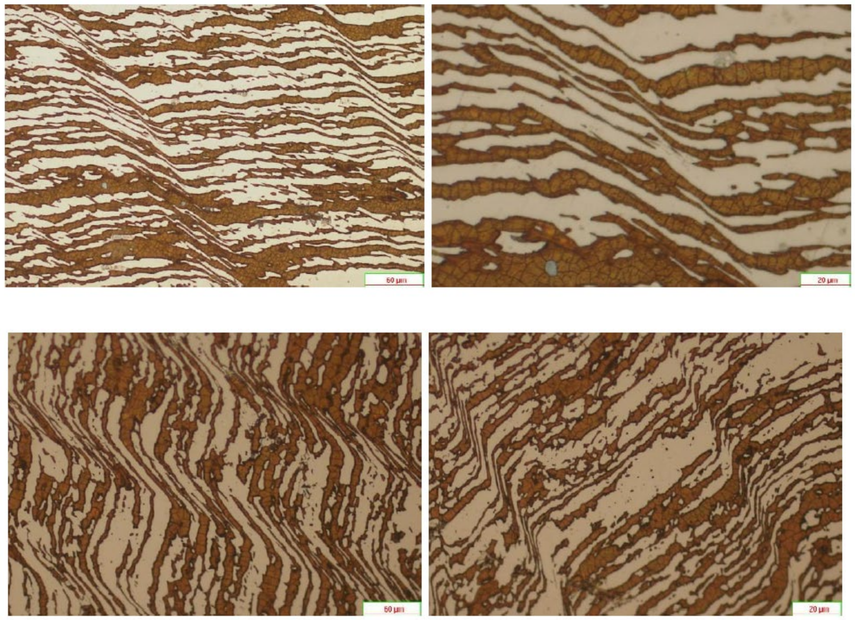

Fig. 5. Deformation bands observed after 1and 2 pass in optical microscope

EBSD analysis:
EBSD investigations is a very powerful tool in order to investigate the evolution of microstructure after each pass in ECAP processed materials. The EBSD maps have been performed along the longitudinal direction with respect the ECAP deformation (Fig.6).

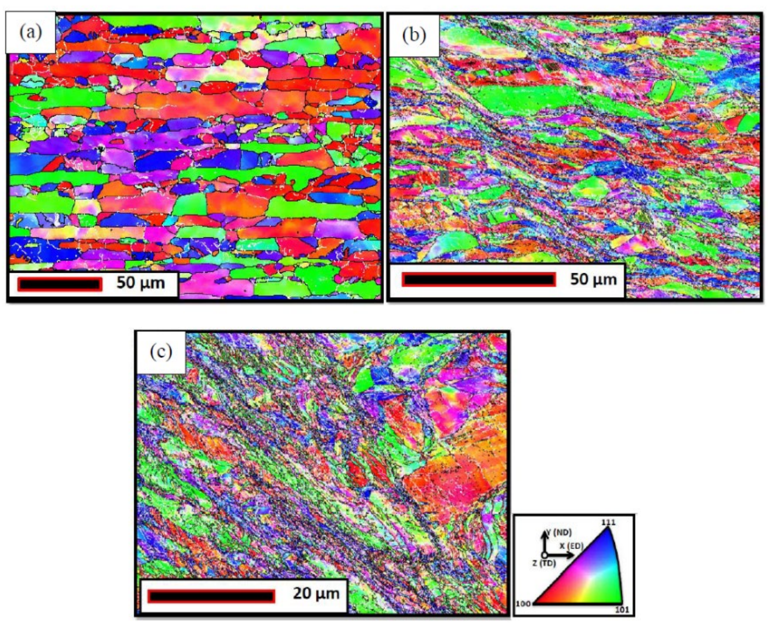

Fig. 6. IPF map with misorientations greater than 3°. Elongated as well as equi-axed grains are seen in the image. Also stereographic triangle, which indicates the orientatons of the grains in the IPF map. (a) 0 Pass (b) 1Pass (c) 2 Pass.

From the inverse pole figure map (FIG6.) it can be observed very extensive grain refinement near the deformation bands. After the two ECAP passes, the microstructure shows mainly equiaxed grains near the deformation bands and grains elongated near them.

The grain size just after the first pass is very small (0.9 µm). After the second pass the avarege grain size is reduced down to 0.4 µm.

Distribution of grain size in the non ECAPed material and after each pass is shown in Fig.7. The average grain size of the undeformed material is approximately 8.5 µm which get reduced to 0.9 µm after the first pass and to 0.4 µm after the second pass. Even though the as received material shows a very fine grain microstructure due to the effect of austenite and ferrite, the grain refinement of the ECAP processed is very strong.

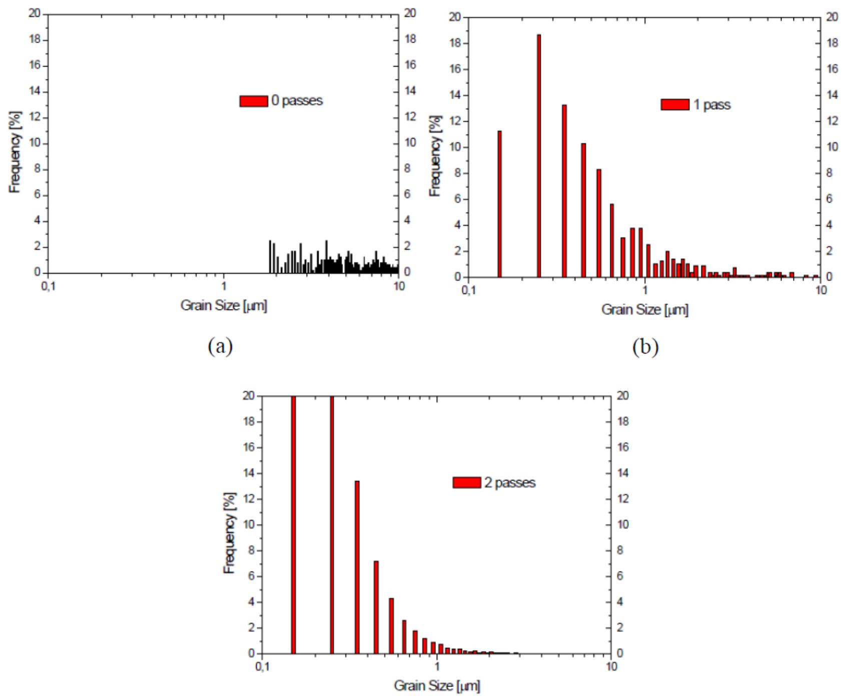

Fig. 7. Grain size distribution (a) 0 Pass (b) 1 Pass (c) 2 Pass

Grain refinement occurring during Equal Channel Angular Pressing (ECAP) can be attributed to the formation of deformation bands, which are an inevitable result of the process and have two potential origins. The first concept suggests that deformation bands originate due to the uncertainty involved in selecting the operative slip systems. In many cases, the applied strain can be accommodated by multiple sets of slip systems, resulting in different lattice rotations. Another origin is associated with the non-uniform straining, where different regions of a grain experience varying levels of strain. If the work done within the bands is insufficient for homogeneous deformation and if the bands can be arranged in a way that their net strain matches the overall deformation, they contribute to grain refinement. These deformation bands are separated by Geometrically Necessary Boundaries (GNBs). Additionally, boundaries can also form through the statistical trapping of dislocations, leading to the creation of Incidental Dislocation Boundaries (IDBs). The misorientation of both types of boundaries increases as the strain progresses. Furthermore, as deformation increases, the spacing between both types of boundaries decreases, indicating that grain subdivision continues to refine at higher strain rates.

Fig.8 shows the corresponding grain boundary maps, where 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 observed after the initial pass reveals a prevalence of low-angle boundaries, primarily in the austenite phase. Upon the second pass, the microstructure exhibits low angle misorientation boundaries in both the ferrite (BCC) and austenite (FCC) phases. Additionally, as the number of passes in ECAP is increased, a gradual transition occurs from an array predominantly composed of low-angle grain boundaries (LAGBs) to a predominantly high-angle configuration.

In the examined Dual-Phase Stainless Steel (DSS), the application of ECAP initially generates grain boundary misorientations with an abundance of low-angle boundaries. This pattern of misorientation distribution is consistently observed after ECAP due to the significant number of excess dislocations introduced into the billet during each pass through the die. However, there is an observed trend of increasing average boundaries misorientation with an increase in the number of passes. This presents an opportunity to utilize ECAP or other severe plastic deformation techniques to obtain fine-grained materials with varying but controlled boundary misorientation distributions.

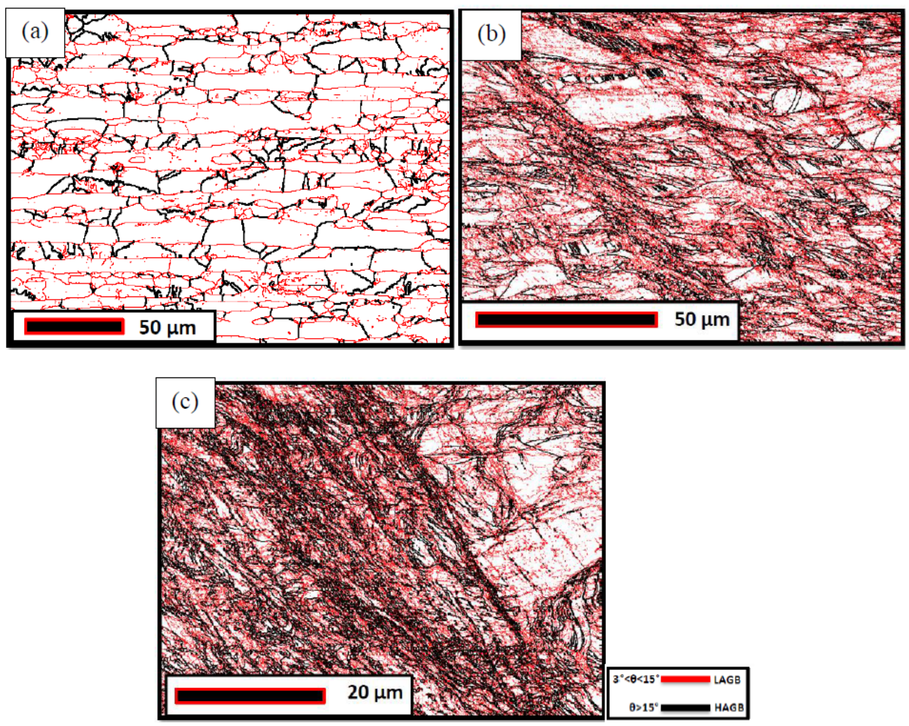

Fig.8. Boundary map which shows low angle grain boundaries and high angle grain boundaries (a) 0 Pass (b) 1 Pass (c) 2 Pass

After undergoing ECAP, the analysis of grain boundary misorientations indicates a predominance of low-angle boundaries. The consistent occurrence of a high proportion of low-angle boundaries in the misorientation distributions after ECAP is a direct result of the substantial number of excess dislocations introduced into the billet during each individual pass through the ECAP die. However, there is an observable trend of the average boundary misorientation increasing as the number of passes is increased. This presents a valuable opportunity to employ ECAP processing in the creation of materials with distinct yet controlled distributions of boundary misorientations.

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 to 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.

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