Short Communication

Inducing heterogeneity in an austenitic stainless steel by equal channel angular sheet extrusion (ECASE)

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A B S T R A C T
An austenitic stainless steel was processed at room temperature by equal channel angular sheet extrusion (ECASE). The microstructure evolution was heterogeneous across the sheet thickness with a higher martensitic transformation in the zones closer to the edges than in the sheet core due to the deformation-induced martensite (DIM) phenomena. The material heterogeneity induced by one ECASE pass gave a good strength–ductility relationship.

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1. Introduction

Austenitic stainless steels (ASSs) are recognized as non-magnetic materials and they can be used in a wide range of temperatures from cryogenic temperatures to the red-hot temperatures of furnaces and jet engines [1]. ASSs present interesting microstructural and mechanical properties especially after the application of some heat treatments and deformation processes. Besides excellent corrosion resistance and good weldability, ASSs can reach yield stresses from 200 MPa in the coarse-grained (CG) state to 2000 MPa after cold

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working [2,3]. The great increment in the yield stress can be mainly attributed to some deformation-induced martensite (DIM) effect, where two types of martensite can be formed: HCP ε-martensite and BCC α′ martensite [2,4,5].

Materials processed by severe plastic deformation (SPD) techniques present interesting behavior not only because of the very low amount of porosity of these materials but also for their mechanical properties. However, as many studies have already showed [6–9] the large increment in the material strength due to the plastic deformation uses to be accompanied by ductility reduction. Another issue related with the production of ultra-fine grained (UFG) materials is the mass productivity of high-quality materials when manufactured by SPD techniques, since the processing demands are highly increased by the dimensions of the processed material.

For solving this problem some new SPD techniques have been proposed. Equal channel angular rolling (ECAR) [10,11] and equal channel angular pressing (ECAP) modified techniques (ECADS or ECASE) [12] are some of these equal channel angular pressing (ECAP) modified techniques. These processes allow the production of larger ultrafine and heterogeneous microstructure sheets [11,13,14]. According to Zisman et al. [12] the process deformation is a function of both the simple shear per pass (γ) coupled with the strain related with the elongation (εy) and thickness reduction (εz) components as Eq. (1) shows.

\[ ε = \frac{\sqrt{2}}{3} \left[ (ε_y - ε_z)^2 + (ε_z)^2 + \frac{3}{2} γ^2 \right]^{\frac{1}{2}} = \frac{γ}{\sqrt{3}} \left[ 1 + 2 \left( \frac{ε_z}{γ} \right)^2 \right] \]

Today, most of the research related with the SPD techniques does not concentrate in very high material deformation. New investigations are focusing in the early deformations stages like the effect of one ECAP pass over the mechanical properties of ARMCO iron [15] or the modification of conventional methods such as the study of Park et al. [16] in an interstitial free steel deformed by differential speed rolling. For that reason, in this study one ECASE pass will be used to develop an optimal microstructure heterogeneity form the strength–ductility perspective.

Some new researchers [17,18] demonstrated that a heterogeneous microstructure can be a good option to produce materials with good strength and ductility, simultaneously. Ma et al. [18] established that in a heterogeneous structure both soft and hard regions can exist together. Where the soft regions deform plastically more than hard ones giving rise to gradients of plastic deformation. Heterogeneous nanostructures can offer a high capacity of storing more geometrically necessary dislocations, thereby enhancing the strain hardening and consequently providing a good combination of strength and ductility [19–21].

The aim of this work is the study of the microstructure and mechanical properties of the ASS 304L processed by one ECASE pass showing that this process can be a good alternative to produce heterogeneous microstructures at the early deformation stage of the ECASE process, either spatially heterogeneous or as phase mixtures, with a good strength–ductility relationship.

2. Experimental procedure

An ASS 304L (≤0.03C; 18–20Cr; 69.9–74%Fe; ≤2Mn; 8–12Ni; ≤0.045P; ≤1Si; ≤0.03S (in wt%)) was received in the form of 20 mm width and 5 mm thickness sheets. ECASE was carried out at room temperature using a die with an inner angle of φ = 150° (see Fig. 1a). Before the ECASE process the material was heat treated at 850 °C for 2 h and further water quenched.

The microstructure of the samples was characterized by electron backscattered diffraction (EBSD). For this purpose, specimens were cut from the transversal direction (TD) plane of the ECASE samples and mechanically polished from 2500 grit SiC paper until 0.02 μm in colloidal silica suspension. The data was processed with TSL OIM 7.3b software. The data processing parameters for OIM software were: maximum mis-orientation threshold angle of 5° for grain size calculation and clean-up subroutine by grain dilation with a minimum grain size of 4 points. For characterizing the local texture three EBSD maps (S1, S2, and S3) were obtained with 1 μm step size covering the edges (S1 and S3) and the center of the sheet thickness (S2).

For the phase identification X-ray diffraction measurements of the processed samples were carried out on a Philips X pert Pro MPD diffractometer with Cu-Kα radiation at 40 kV and 15 mA, X-ray lenses, parallel plate collimator and Xe detector. The specimens were mechanically polished to 1000 grit and then etched with a mix of 5 mg FeCl3 + 50 ml HCl and 100 ml H2O. The quantitative phase volume fraction was obtained using the methodology described by De et al. [22]. This method has been used in different studies to calculate the phase volume fraction [3,23]. The volume fraction of phases was calculated with Maud software [24] through a Rietveld refinement [25] in the normal direction (ND) plane.

The strength of the processed material was characterized by tensile tests and hardness measurements in the Knoop scale. Three different hardness profiles were done along the sheet thickness to evaluate the hardening gradient between the zones closer to the edges and the sheet core (TD plane). Tension tests at room temperature at a constant strain rate of 1.1 × 10−3 s−1 were performed using an Instron 3362 universal testing machine. The specimens were machined out of the 5 mm thickness sheets (gauge dimension of 12 mm × 3 mm × 5 mm) by means of wire electrical discharge machining.

3. Results and discussion

A first approximation of the material heterogeneity after the ECASE process Fig. 1b indicates the Knoop hardness evolution for the initial (0P) and deformed material (1P). The hardness evolution across the material thickness (TD plane) for the initial material seems to be quite homogeneous with a mean value of 223.7 and a standard deviation of 17.8. On the other hand, the material with one ECASE pass reached a mean and standard deviation of 322.7 and 44.7 respectively. These differences in the mean and standard deviation values are consequence of the material deformation which not only
improve the material strength but also demonstrate the effectiveness of the ECASE process to introduce heterogeneity in the material.

The heterogeneity in the material can be corroborated in Fig. 1b by the higher hardness increments in the zones closer to the edges in comparison with the lower increments in the center zone. A similar behavior was found by Fandiño et al. [26] with a magnesium alloy processed by ECASE. The higher hardness values in the edge neighborhoods are due to the mode of deformation of the ECASE process, which concentrates higher deformations in the regions closer to the edges, as was demonstrated in a previous study [27].

In Fig. 2 the microstructure evolution at three points over the TD plane can be observed for both initial and one ECASE pass materials. As a first observation, the material with one ECASE pass presents a higher fraction of \( \alpha' \) martensite in the regions S1 and S3 in the vicinities of the inner and outer edges. Meanwhile, in the region S2, a more homogeneous distribution of phases can be observed, similar to the distribution in the initial material. This high fraction of \( \alpha' \) martensite in the inner and outer edges is not the only microstructure change promoted by the heterogeneity in the ECASE process. Misorientation angle distribution, and texture changes can be also observed in this figure, where smaller fractions of high angle
grain boundaries (HAGB) were calculated in the S1 and S3 zones with values of 38.8% and 33.6% respectively in comparison with the 65.8% and 45% in the S1 and S3 zones in the initial material. For the S2 zone, only minor differences were found between one pass and initial material with values of 60% and 67.34% respectively.

The misorientation distributions in Fig. 2 also show the reduction in the fraction of twins (peaks at misorientation values of ~44° and ~60°) for the material with one ECASE pass in the region S1 and S3 with respect to the area S2 and the regions S1, S2, and S3 in the initial material. This reduction of twins in the inner and outer edge regions is a consequence of the DIM effect. It has been demonstrated by Shen et al. [28] that in an ASS304 the strain induced transformation mechanism follows the sequence: γ-austenite → stacking fault (SF) → twin → α′-martensite. For that reason, due to the heterogeneity of the process a low fraction of twins was found in the regions S1 and S3 (higher deformation) and a high fraction in the region S2 (lower deformation), because in the regions S1 and S3 the twinned crystals partially transform to α′-martensite.

Another effect of the heterogeneity in the material microstructure can be observed in the texture evolution in the three analyzed areas. The inverse pole figures (IPFs) for both phases FCC and BCC are represented in Fig. 2. In the initial material as well as in the deformed one the γ-austenite phase in areas S1 and S3 is mainly oriented to the <101> direction, while in the area S2 both materials share orientations between <101> and <111>. Conversely, the BCC phase for the material with one pass presents a marked difference in orientation toward to <100> in the areas S1 and S3 with respect to the area S2 which is oriented to <111> direction. Those texture changes are a consequence of the combination of monotonic (rolling) and non-monotonic (shearing) deformations. It has been demonstrated by Bobor et al. [29,30] that shearing can be considered as a non-monotonic process which can lead not
only to texture changes but also to microstructure modifications.

Fig. 3 shows in more detail the microstructure characterization in the zone more affected by the ECAP process (S1) after one pass together with the initial material to see the deformation effect. The X-ray diffraction patterns representing the material phases allow to see the great increment in the BCC $\alpha'$-martensite due to the DIM effect. The volume fraction of phases indicates the large transformation from the initial austenite to martensite passing from 4.3% in the initial state to 71% after one pass (close to the inner edge). A similar volume fraction of $\alpha'$-martensite was obtained by Shirdel et al. [3] for a commercial ASS304L after 55% cold rolling. On the other hand, the contribution of characteristic peaks for $\varepsilon$-martensite was very weak in the X-ray diffraction spectra so that its volume fraction was neglected. Besides, the EBSD analysis corroborated the increment of $\alpha'$-martensite by the appearance of bands of martensite inside of the initial austenitic grains because of the lattice distortion. It can be also observed in images, using image quality (IQ), that the $\alpha'$-martensite starts to appear around the $\varepsilon$-martensite and in the austenitic grain boundaries. This effect in the material microstructure can be observed through the grain average misorientation (GAM) approach. In this context, the grain average misorientation, which is the average misorientation between all neighboring pairs of points in a grain [31,32], presents lower GAM values for recrystallized or deformation free grains than those of deformed grains. For that reason, most of the grains in the material with one pass presents GAM values higher than that of the initial material (see the peak shift in the GAM plot of Fig. 3).

Fig. 4 indicates the mechanical properties after the tensile test for both initial and one ECAP pass materials. Firstly, in Fig. 4a it can be observed a remarkable increment in the yield and maximum stresses for the material with one pass with values of 711.3 and 886.4 MPa respectively with respect to 256.7 and 724.1 MPa for the initial material. However, a reduction in the elongation to failure was observed after one pass, moving from ~80% engineering strain in the initial state to ~40% after one pass. In spite of the elongation reduction, the mechanical properties of the present steel are better than the properties of the ASS of Karavaeva et al. [33] processed by ECAP, rolling
and ECAP + rolling. Their results showed a similar elongation to failure but with a great reduction in the strain hardening capacity. Other authors such as Kumar et al. [34] and Zheng et al. [35] reported a good strength–ductility relationship by the adequate selection of deformation process and annealing treatments with values of yield and maximum stresses and elongation to failure similar to the ones in this study.

The heterogeneity effect into the mechanical properties is also reflected in the fracture areas after the tensile test. In Fig. 4a it can be observed how the two materials exhibit different fracture areas (size of fracture area, direction of fracture). In the initial material the fracture propagation (smooth and bright areas) is coming from all four sides of the sample (blue arrows indicating the direction), whereas in the deformed material the propagation just comes from one side (the hardest, inner edge), these areas of propagation look like a brittle material following a veins pattern (orange square). Another characteristic shared by both materials is that the final fracture corresponds well with a ductile material due to the presence of dimples (green square), although the dimples are larger in the initial material than in the deformed one. Besides, the total and final fracture areas (red and yellow dashed lines respectively) are smaller than in the material with one pass due to the intense plastic deformation before fracture. This high intense plastic deformation in the initial material is demonstrated by the Considère criterion [36,37], which indicates in Fig. 4b a higher value of deformation where the plastic instability starts with respect to the material with...
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Conflicts of interest

The authors declare not conflict of interest.

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