Processing of a duplex stainless steel by equal channel angular extrusion

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ABSTRACT

A UNS S32205 duplex stainless steel was processed by ECAE in three different velocities, at room temperature, and heat treated in different temperatures and times to evaluate recrystallization. Attrition forces promoted great deformation heterogeneity in the samples sections, with hardness increase, and morphology changes in the grains and changing orientation through the processed samples. In treated samples surface, two types of distinct structures was formed, with surfaces positioned in 90º and 120º angles, probably because the annihilation of pilled dislocations in ferrite based centered cubic structure and austenite face centered cubic structure, respectively. The induced martensite by cold deformation was also observed. Some samples demonstrate located points of recrystallization in grain boundaries for some treatment conditions, the number of recrystallization nuclei increased with the increase of treatment time.

Keywords: Duplex, ECAP, stainless steel.

1   INTRODUCTION

Although the physical and mechanical properties of metals are determined by several factors, the grain size of the material is generally very significant and frequently a dominant factor in determining the properties and final application of the materials [1].

The fact of the mechanical resistance increases with the grain size reduction makes interesting the production of materials with an extremely small grain size. Commercial metallic alloys are generally produced for specific applications where the alloys are submitted to specific mechanical and thermal processes. However this procedures can't be used to produce sub micrometric grain sized materials, because they presents a lower limit fixed, in order of a few micrometers, which represents the minimum grain size that can be obtained by these processes [1].

To produce ultrafine grained materials by a coarse grained material, the imposture of an extreme high strain is necessary, in order to introduce a high density of dislocations, then these dislocations can be rearranged and form ordered grain boundaries. However, in practice, this situation is restrict in conventional conformation process, like the extrusion or stretching, it happens cause of this two reasons: the total strain that can be imposed to the material is limited by the cross sectional reduction in the specimen, and, the total strain imposed is insufficient to introduce an ultrafine grain structure to the material, due the low workability of these materials at room temperature.

Considering these limitations, several researches have been directed to the development of new techniques that can be used to produce ultrafine grained materials and provide the understanding of the behavior during the recrystallization process. Between these techniques, the ECAE (equal channel angular extrusion) was developed, or ECAP (equal channel angular pressing). The principle of ECAE is based on severe plastic deformation application, through the imposture of an extremely high strain in a relatively low temperature, without any significant changes in the cross sectional dimensions of the specimen [1, 2].

During the last decades, ECAE has showed as a very efficient process for grain refinement, through the multi passes operations of the material through an angular channel. Lots of passes are applied to the specimen resulting in a variation of crystallographic during the procedure by the strain accumulation [1-6].

Many studies reported this processing mode in pure metals, but very few have studied this process in commercial alloys. In this sense, a material that have very interesting properties to be observed in relation to the grain size and behavior during the recrystallization process is the duplex stainless steel [7-9].

Duplex stainless steel is having a gradual improvement in his utilization in several applications such as the chemical, energy and oil industries and medical instruments beside a wide range of other products. This is due his high corrosion resistance. The duplex stainless steel are materials that presents a compound

structure by ferrite and austenite, that provides to these steels higher mechanical and corrosion resistance compared to the usual stainless steels, also presents about twice of the yield strength compared to the ferritic or austenitic stainless steels and have more plasticity than martensitic stainless steels. The properties combination of the ferritic and austenitic stainless steels provides to the duplex stainless steels superiors properties compared to these two types of steels [10, 11].

Due his excellent properties combination, duplex stainless steels have been an alternative to monophasic stainless steels, perfectly justified due his combination of excellent mechanical properties and anti corrosive properties. And, beyond the outstanding qualities, the processing complexity of these materials is another motivation for several new researches.

2   MATERIALS AND EXPERIMENTAL PROCEDURE

A duplex stainless steel UR 45N, from URANUS, with an austenitic-ferritic structure, presenting about 50% of each phase was used. These specimens were processed by ECAE in a 120º die, being pressed by a tensile and compression test equipment with capacity for 300KN from Shimadzu, model Autograph II, in the Materials Engineering Department (DEMA) from the UEPG (State University of Ponta Grossa). The pressing was done using a punch made of tool steel AISI M42.

The route A was used in the extrusion, and at room temperature (~20ºC). Three different velocities were used: 1,5, 6 and 12 mm/min., and the operation pressure were controlled. In lower velocities a second pass was done. The heat treatments used were: 7.5; 15 and 30 minutes at 1000ºC, followed by air cooling.

Optical Microscopy was used to analyze the ferritic and austenitic structures and to determine the kinetics of recrystallization and grain growth.

Another analysis of the recrystallization was made by Scanning Electron Microscope (SEM), using a SSX-550 Shimadzu microscope.

To analyze the mechanical properties microhardness tests were done, providing results about the mechanical resistance and hardness of the processed materials.

3   EXPERIMENTAL RESULTS

3.1   Material Characterization as Received

As received Duplex stainless steel microstructure is shown in Figure 1, and presents the longitudinal (a), superior (b) and cross (c) faces, whose is observed flattened and elongated grains due the rolling process. The brighter region in the image is the austenite grain over a darker region of the ferritic matrix.

Before processing, the Vickers micro hardness was measured in each face of the material. The average results in the longitudinal and cross section was 275HV±15, in agreement with the values found in literature, but the superior face showed an average hardness of 306HV±22. This difference can be explained by the several grain boundaries perpendicular to the forces applied in the indentation in this face.
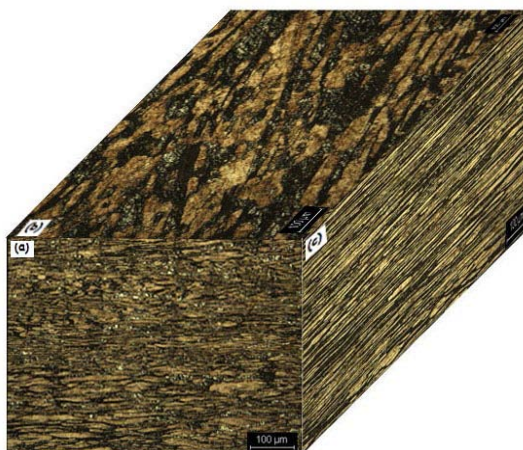

Figure 1: Tridimensional presentation of duplex stainless steel as received, with the directions: (a) longitudinal, (b) superior and (c) cross.

Stress reached behaviour information during the extrusion was collected by an universal mechanical test equipment. During the duplex stainless steel processing by ECAE, in three different speeds, tension reached as function of displacement was measured and the results are presented in the Figure 2, which shows the development of resistance levels up to 3GPa.
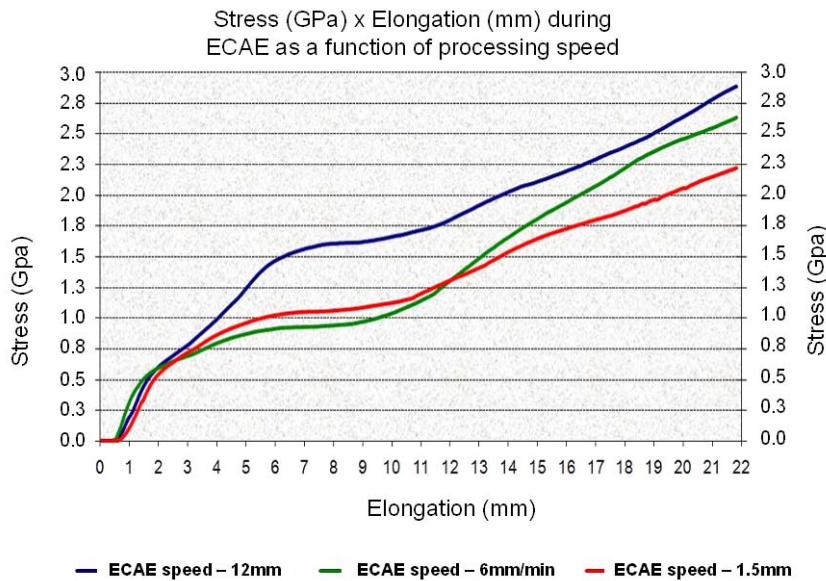

Figure 2: Graphic of stress as a function of displacement during duplex stainless steel ECAE at speeds of 1, 5, 6 and 12 mm / min.

In the graphic, the increase of the stress with the increase of the extrusion speed is evident, reaching over 2GPa in the lower processing speed and reaching near 3GPa in the maximum speed. The extrusion speed is an extremely important factor to consider in the project of the processing die.

Is also observed the continuous increasing of the stress over the curves, indicating that the generated friction between the specimen and the die channel walls contribute to the increasing of the stress in the processing. With the advance of the specimen in the output channel, the contact area of processed steel and the die walls increases and the contribution of the friction to increase the stress is necessary to remember that this elevated stress values are very close to the limit for utilization to the tool steels used in fabrication of the punctures in this present work (M2 and D2).
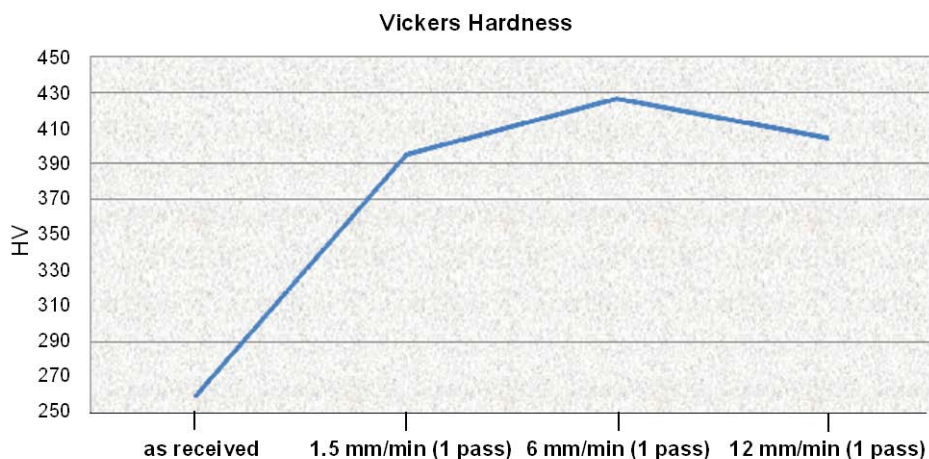

Figure 3: Variations according to processing speeds.

After processing the duplex stainless steel by ECAE the Vickers hardness of the samples were measured. The average result found in the samples cross section processed at speeds of 1,5; 6 and 12

mm/min., is shown in Figure 3 graphics, which shows that all the speed processing used promoted high levels of hardness. Is observed that the highest hardness is found in a speed processing of 6mm/min., in this situation the hardness increased about 64% comparing to the as received material. All the processing speeds presented similar results, with variations smaller than 4% comparing to its average value.

The small difference found between the hardness values in Figure 3 can be related to the same active systems in the speed range investigated. Knowing that the duplex stainless steels are compound by two different phases, with ferritic structures (CCC) and austenitic structures (CFC), and each one is associated with a different slip system.

3.2 Evaluation of the Microstructural Evolution

Figure 4 shows optical microscopy of the complete cross section of the sample during the pass through the ECAE processing die, at a speed of 1.5 mm/min. at room temperature. In this figure stand three different regions by the grain morphology. We can observe in a great extension of the cross section the elongated grains placed at a 45° angle referent to the initial orientation.

Between successive changes promoted in the duplex steel during the plastic deformation, changes can be observed in the grain shape, which become elongated, increasing the grain boundaries area in the material.

In the Area 3 of the section (near to the angle of curvature, Φ) a smaller deformation is observed in relation to the other regions. In this region the process of grain orientation starts resulting in the dominant orientation in the section, as showed in Figure 4. Area 2 of the section presents morphological homogeneity of the grains and represents about 90% of the section area. The aspect of the area 1 of the microstructure from Figure 4, shows that this region is subjected to a higher strength of the friction between the processed material and the die walls. In Figure 4, can be observed, in a region near the surface, grains with a great severity of deformation, which shows the actuation of the extremely high friction strengths. We can also observe a region approximately at 300μm from surface, a darker region with a severity more pronounced in the deformation.

The effect of stress applied on the surface of the solids does not propagate continuously over the entire volume under solicitation. So, the superficial friction stress is propagated until a limited depth of the material. In consequence of this friction, the material flow is restricted in this region, establishing a strain heterogeneity, which can be observed by the difference of deformation of the grains in the cross section in Figure 4. Hardness values verified in this region reach values much superior than the rest of the section.
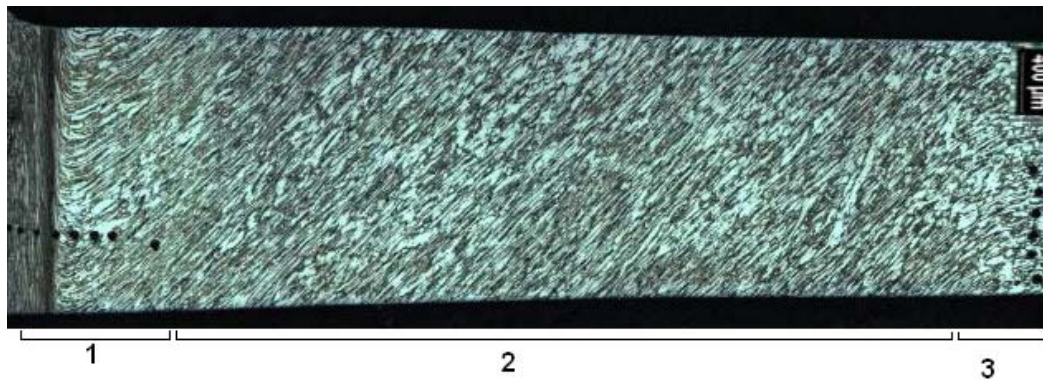

Figure 4: Cross section of the sample after processed at 1.5mm/min.

The sections of the transversal and longitudinal processing direction of the samples are presented next. Figure 5 shows the optical microscopy of the duplex stainless steel processed by ECAE at 1,5mm/min. speed. The longitudinal section presents a great elongation and grain reorientation. The transversal section does not present changes in grain orientation, but, in result of the great elongation, a grain refinement in the material was promoted. This same behavior was verified in the same duplex stainless steel processed by ECAE at 6 and 12 mm/min. speeds.
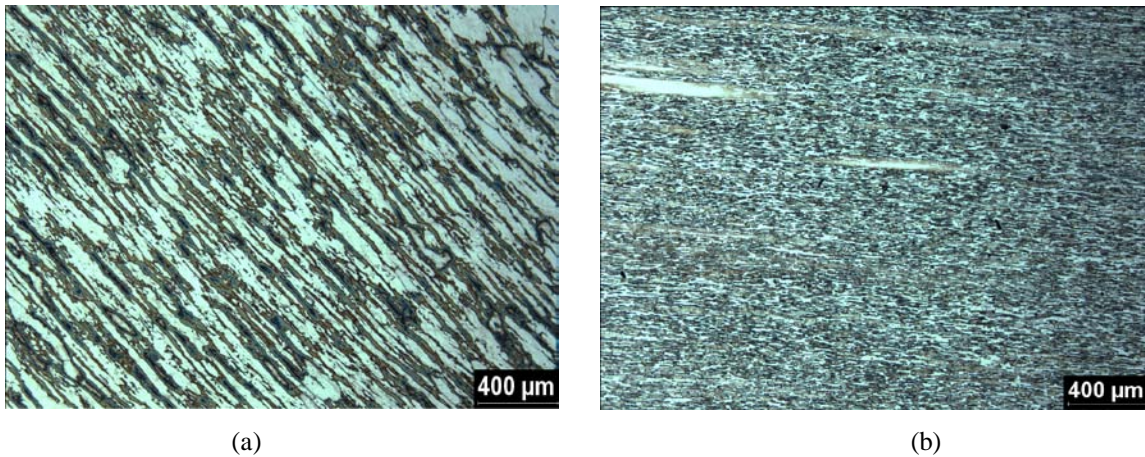
(a)   (b)

Figure 5: Transversal (a) and longitudinal (b) sections of the duplex stainless steel processed by ECAE at 1.5mm/min.

3.3   Microstructural Analysis of Heat Treated Samples at 1200ºC

Just after heat treatment at 1200ºC, the processed samples presented in its surface a structure compound by coarse grains and colored, visible by eye, as showed Figure 6 (a), (b) and (c), related to the samples processed at 1,5; 6 and 12 mm/min. and heat treated.
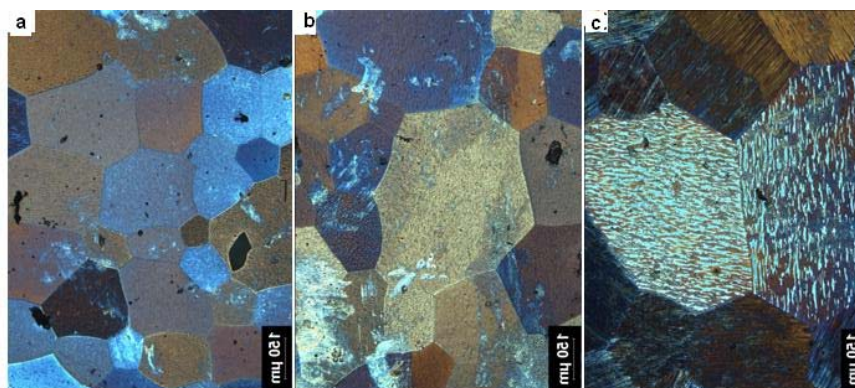

Figure 6: Surface microstructure of the samples processed at 1,5 (a), 6 (b) and 12 mm/min.(c), after heat treatment, without any metallographic preparation.

These microstructures were evaluated at SEM (Scanning electronic microscope) and the obtained images are presented in Figure 7(a), (b) and (c) related to Figure 6(a), (b) and (c) respectively.

The sequence of SEM images indicates, as function of heat treatment time, the sequence of formation of the structures compound by small surfaces forming well defined angles between them. Also can be observed, that this surfaces trend to form 90 and 120º angles, as showed in Figure 7(a).

Analyzing the resultant microstructures for the three processing velocities is observed that the structures formations are faster when the material is processed at 1,5mm/min. The first microstructures obtained for all three velocities are equals. But, with the increasing of heat treatment time up to 30 minutes, the lower speed stars to present the formation of structures with a well defined angle. In the higher speeds processed samples, only after 60 minutes of heat treatment starts to present these structures. This fact can be associated to the amount of active slip systems in each different formation speed. Higher processing speeds could just activate the most favorable slip systems, while the lower speeds can activate the less favorable slip systems.
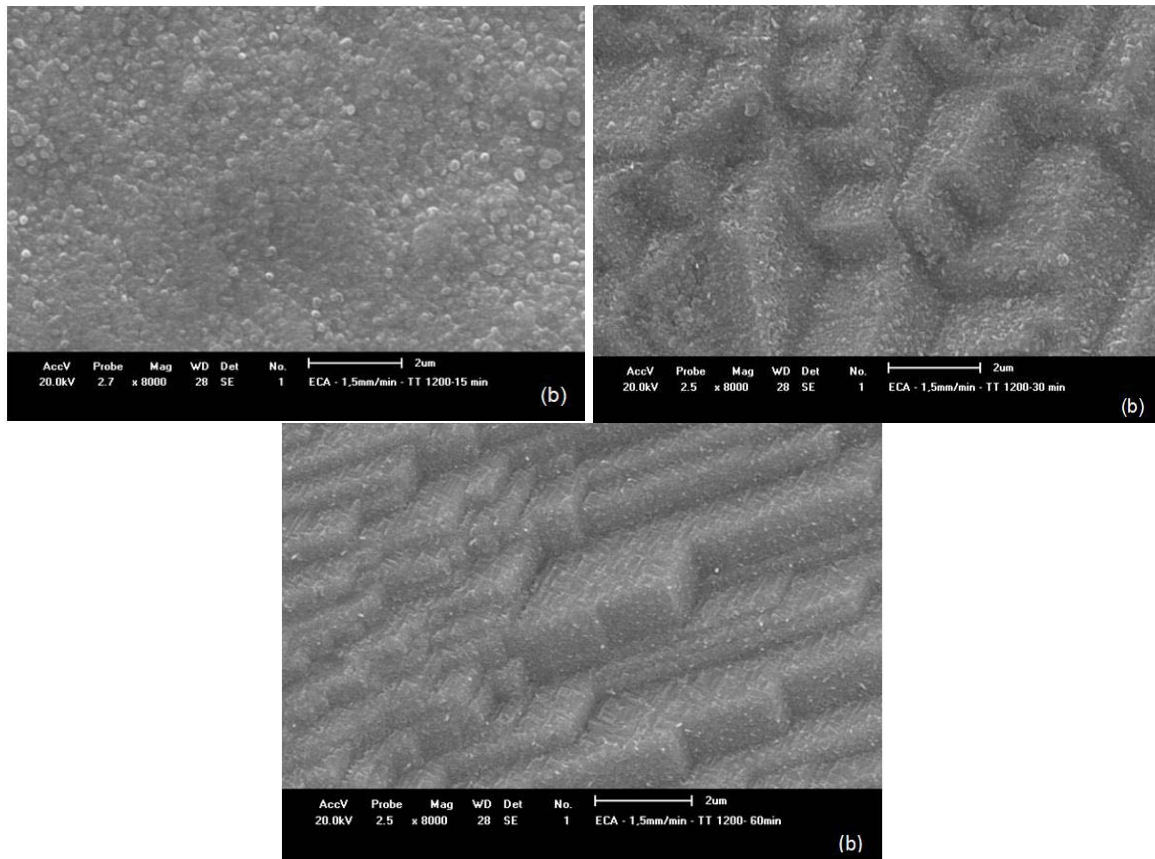
Figure 7: SEM of duplex stainless steel processed at 1,5mm/min. With heat treatments of 15, 30 and 60 minutes respectively.
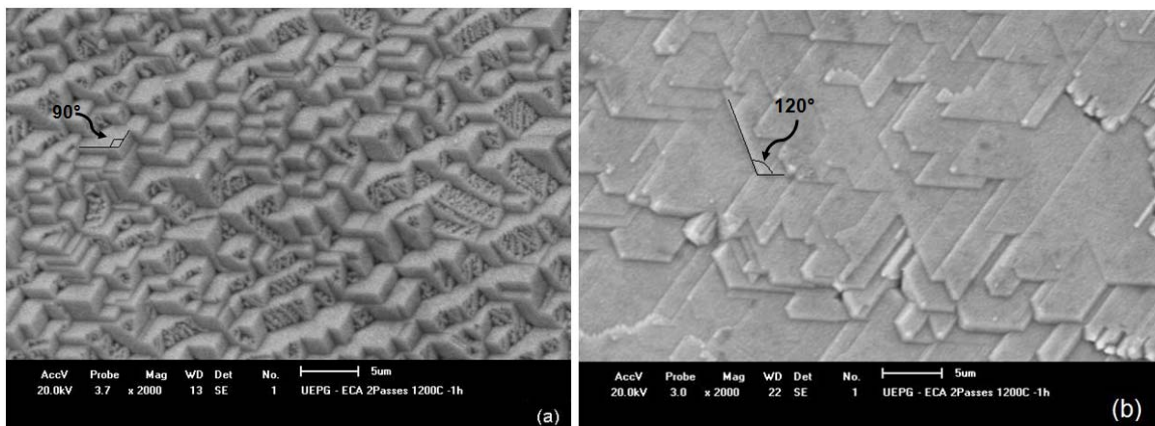

Figure 8: SEM of duplex stainless steel after 2 passes processing by ECAE at 1,5 mm/min. and treated at 1200°C for 60 minutes.

A second processing pass was only possible at the 1.5 mm/min. speed. The sample treated for 60 minutes presented the formation of regions with two well distinct structures considering the formed angles, as showed in Figure 8(a) and (b). In Figure 8(a) occurs the formation of 90° angles between the surfaces, which promoted the formation of cubic structures. In Figure 8(b) the angles formed are 120° angles, which promoted the formation of "roof manner" structures. These structures can be associated to the ferrictic and austenitic phases, respectively, related to the slip system present in each of them. Ferrite, with a BCC (body centered cubic) structure, with the principal slip system in {110} planes which form 90° angles between themselves. The austenite slip systems, with the FCC (face centered cubic) occurs exclusively at {111} slip systems, which form 120° angles between them.

Both structures visualized may be obtained after a high level of strain imposed by the ECAE processing, which provided a high hardening with accumulated and pilled-up dislocations, which can be confirmed by the expressive increase of hardness. With the heat treatment, this dislocations present in the material trend to annihilate themselves at the surface at 90º and 120º angles from ferrite and austenite slip systems, respectively.

Similar structures to the presented in Figure 8(a) were obtained in the steel surface of 316 austenitic stainless steel during the stress corrosion process, as showed in Figure 9. Where the faces of cubic structures formed correspond to the group of planes {110}.
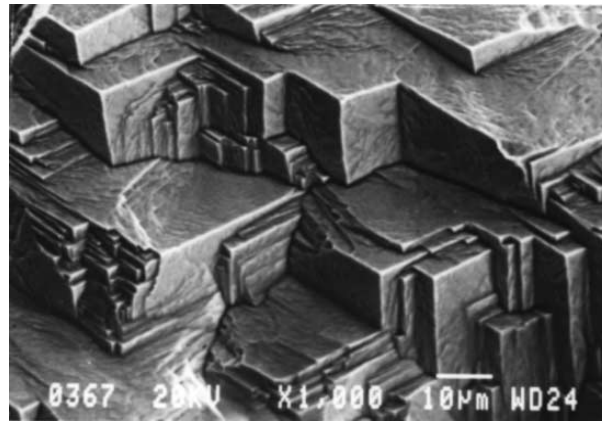

Figure 9: Monocrystal fracture surface microscopy <111> obtained by TEM [12].

Occurrence of these distinct regions was limited to the interior of the grains and exhibit characteristic orientations of the formed structures. Figure 10 shows this characteristics at a triple point found in the sample.
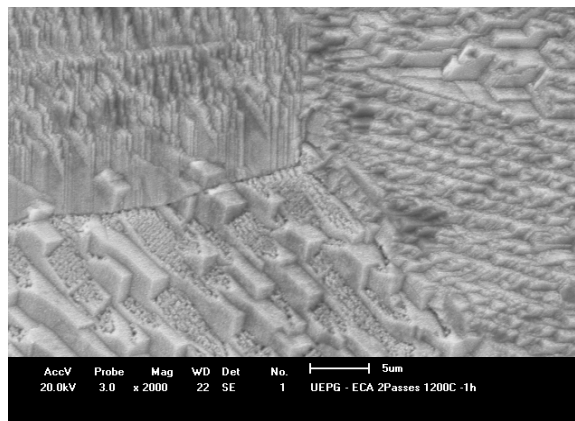

Figure 10: Triple point in a sample processed by ECAE with 2 passes at 1,5mm/min. and heat treated at 1200ºC for 60 minutes.

At surface analysis of the treated samples was also possible to verify typical structures related to transformation of metastable austenite in induced martensite by cold plastic deformation.

In general, martensitic transformation is a phase transformation without diffusion, in which atoms move cooperatively by a shear mechanism. In opposition to diffusion controlled processes, which the transformed fraction increases along time and the reaction speed increases with temperature, martensitic transformation can occur at low temperatures and very fast. The amount of martensite increases with the increase of deformation, with decreasing of deformation temperature and with the increase of deformation rates. Other evidence of induced martensite formation in duplex stainless steel processed by ECAE is the comparison of the images obtained using SEM at present work, with Karaman representation in Figure 11 [13]. Is verified a great similarity between the visualized structure and the proposed representation by this author, specially in planes disposition and formed angles between them.
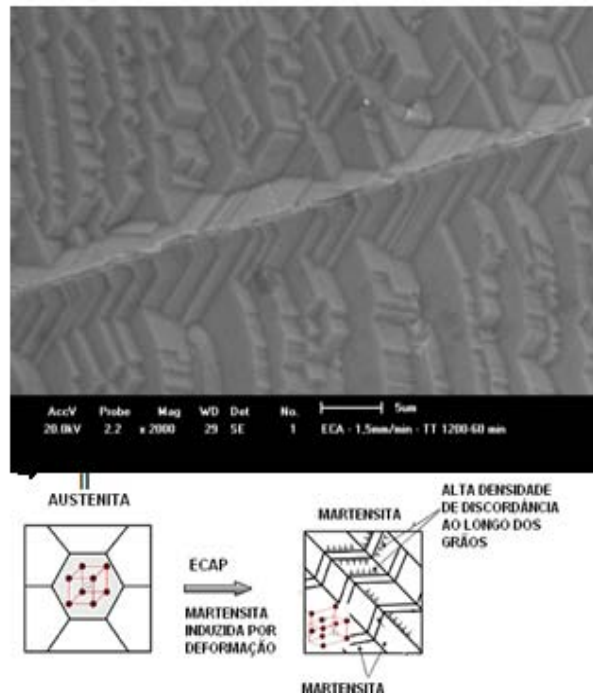
Figure 11: Comparision between duplex stainless steeel processed by ECAE and schematic martensitic transformation from austenite [13].

3.4   Crystallization Primary Evaluation

Some of the processed samples were heat treated at 1000ºC for 7.5, 15 and 30 minutes, as showed in Figure 12. After optical microscope analysis were identified recrystallization spots located at grain boundaries. Considering the increase of time treatment from 7.5 to 15 minutes, is observed the increase of recrystallization nuclei, while after 30 minutes of heat treatment, is verified an increase in the size of the crystals without considerable increase in the number of nuclei.
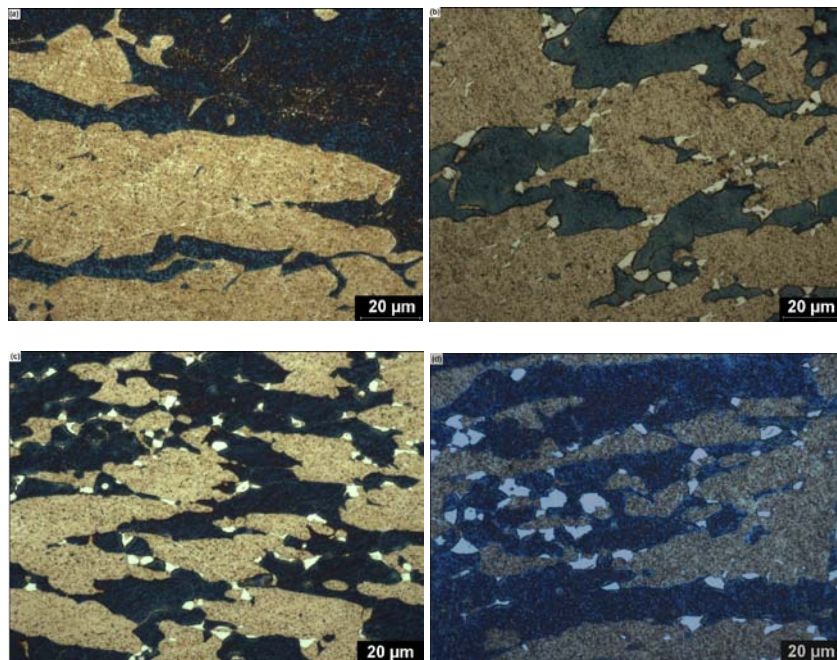

Figure 12: ECAE processed duplex at 1,5mm/min. after 1 pass: (a) without heat treatment, (b) 7,5 min. at 1000ºC, (c) 15 minutes at 1000ºC (d) 30 minutes at 1000ºC.
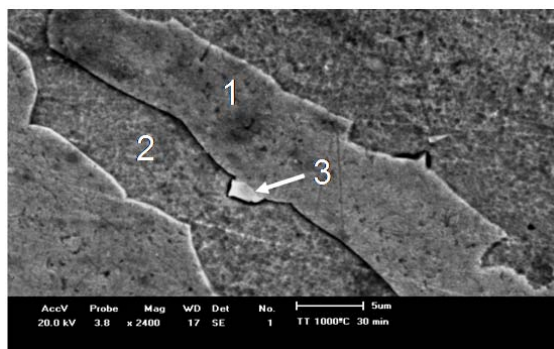
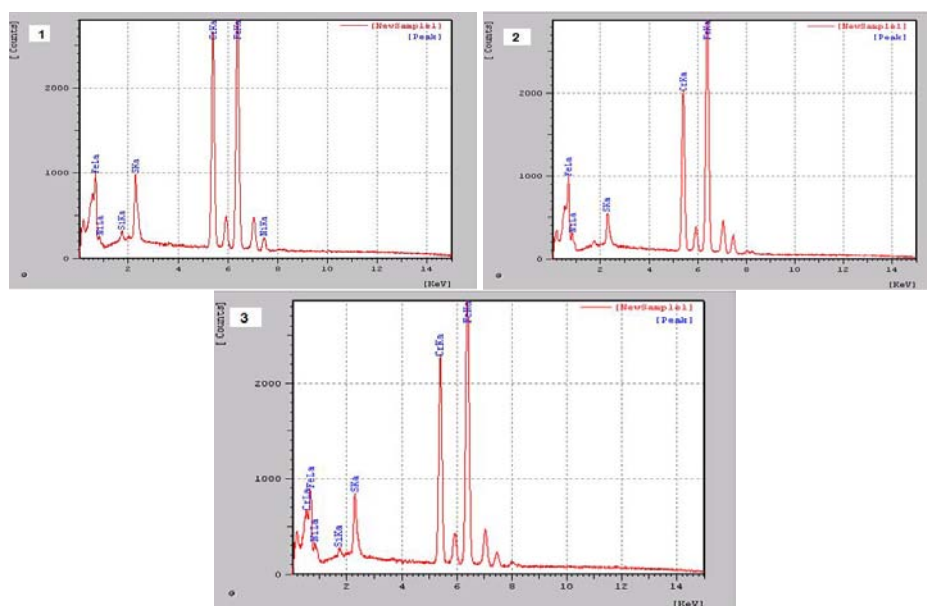**Figure 13:** different regions were the EDS analysis was executed.

Figure 14: EDS analysis results for the three regions

SEM was realized in the sample processed at 1.5 mm/min. and treated at 1000°C for 30 minutes. As shown in Figure 13. EDS analysis were done in the area showed in the figure, in three identified spots, which refers to ferrite grain, austenite grain and the crystal formed during processing. The compositions obtained in the respective spots 1, 2 and 3 of the Figure 13 are described in the Table 1 and the peaks generated by the analysis of these spots are showed in Figure 14.

By the composition obtained in the analysis can be verified that ferrite have greater amount of α phase stabilizer elements (Cr and Fe) and austenite presents greater amount of γ phase stabilizer elements (Ni). Also can be verified by the composition analysis that the formation of new ferrite crystal incorporates more chromium and less nickel due the solubility difference of these elements.

Table 1: Chemical composition obtained by EDS analysis.

Composition (%)	1	2	3
Cr	26.042	27.819	29.091
Fe	64.540	61.465	62.246
Ni	7.221	6.098	5.187

4 CONCLUSIONS

It was possible to obtain a great deformation heterogeneity in the grains due the slip systems, strain and shear bands which determine the microstructural evolution of the material in ECAP process. The

deformation process influences the recovering and recrystallization process, with great heterogeneities and disorientations of structure helping the formation of new grains.

The stress increases with the processing speed and with the number of passes. The stress increases continuously due the friction of the sample with the die walls.

The hardness of the material had a great increase in all three processing speeds, with no significant variations of the hardness achieved with the processing speed.

The regions near to the surface of the sample in contact with the die walls are submitted to higher strains. The strains promoted changes in grain orientations and great elongation in longitudinal sections and this elongation promoted a grain refinement in the cross section of the material.

The heat treatment in the samples showed two distinct structures formed, with 90° and 120° angles well defined. Martensite induced by cold deformation structures was also found. The recrystallization points were observed in grain boundaries, the increase of the time of heat treatment showed an increase in the number of these nuclei and sequent stabilization in the number of new grains formed and growth of the new grains.

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