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Microstructural Characterization and Evaluation of the Thermomechanical Behavior of an Al 7075-T651 Alloy Deformed by Two Passes of ECAP

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Billets of an Al 7075-T651 alloy were processed by two passes of equal channel angular pressing, ECAP, by following route A. First pass was performed at 180°C and the second one in the temperatures 130 and 180°C. The resulting microstructures were characterized by optical microscopy, OM, and scanning electron microscopy, SEM. Also, material macroscopic mechanical properties were evaluated by performing uniaxial compression and Vickers microhardness tests. After the second pass, it was not verified a noticeable grain refinement. After second pass at 130ºC, the samples presented higher mechanical strength than observed after their processing at 180°C and the second pass resulted in a decrease in the mechanical properties compared to the deformation by one pass at 180°C.

Keywords: *Equal channel angular pressing, thermal treatments, microstructural characterization, thermomechanical behavior, Al 7075-T651 alloy.*

1. Introduction

The materials are the basis of any civilization and the development of new technologies, fundamental for human evolution, are made possible by science and engineering that seek incessantly to improve the properties of these materials. These improvements make it possible to increase the field of application and in some cases make possible new uses that until today were not possible. Within this context the methods of Severe Plastic Deformation, SPD, are investigated, because they are able to improve the properties of the processed materials.

One of the most widespread techniques among SPD methods is the ECAP technique, where the material is pressed through a die composed of two equal-sized channels that intersect each other at an angle, Φ, and additional angle Ψ, which defines the arc of curvature of the outer intersection of the channels, causing the material to be subjected to the simple shear stress in the intersection plane[1,2].

The main advantage of the ECAP process is preservation of the cross-sectional dimension after processing, thus making it possible to perform repeated deformations until they accumulate at high levels. With this, slip systems can be activated at each pass through the rotation of the samples at the different angles associated with each route. In addition to improving the mechanical properties, produce extremely fine grains and a variety of textures[3,4].

The Al 7075-T651 aeronautical alloy investigated in this work has an excellent balance of properties required in this industry, such as high mechanical strength, moderate fracture toughness and corrosion resistance[5]. It is of great interest to investigate whether the increase in mechanical strength observed in ECAP processed alloys can be added to the increased strength promoted by the aging effect in Al 7075 alloy. Aged hardenable alloys are generally difficult to process by ECAP at room temperature because they invariably fail by catastrophic cracks or segmentation[6]. The aim of the work was to evaluate the thermo-mechanical and microstructural behavior of this alloy processed by ECAP by distinct conditions, that is, as-received, after first pass at 180ºC and after second pass either at 130ºC or 180ºC, by assuming route A. The microstructural characterization was performed using optical microscope, OM, scanning electron microscope, SEM, and energy dispersive spectroscopy, EDS. The material mechanical behavior was evaluated by performing Vickers hardness and uniaxial compression tests after each deformation pass.

This process can be driven by four different routes: A, B_A, B_C and C. On the route A, investigated in this study, there is no rotation of the sample between the deformation passes causing the material distortion to be continuously increased after each pass providing refined and elongated grains in comparison to starting material. On the other hand and after each pass, in the route B_A the workpiece is rotated by 90° in alternate directions while for the route B_C these rotations occur at 90° in the same sense (clockwise or counterclockwise). In addition, in the case of route C, samples are rotated by 180°. Routes B and C were not investigated in the present work.

2. Experimental Procedures

The experiments were carried out using the Al 7075-T651 alloy, whose chemical composition, obtained by means

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of the Oxford Instruments model Foundry Master Pro, is presented in Table 1.

The material was received as a plate with 1000x1000x20mm. The material was machined in the form of solid square section billets with 9.8mm of edge and 40mm of length for ECAP processing, 5 mm of which is milled at the top and bottom of its thickness. The material for the compression tests was machined with the same section, but 20 mm in length.

The samples were prepared for OM observation by means of conventional metallography techniques, subjected to successive grinding, with 500, 1200 and final 2500 granulation sands using running water as a lubricant. After this step they were electrolytically polished with a solution of 59% CH_4O, 35% $C_2H_4(OH)_2$ and 6% $HClO_4$ (in volume). The potential difference applied was 16V for 25s, at room temperature, using a Power supply (Manufacturer Instrutherm, model DC Power Supply FA-3005). The chemical etch was performed by immersion with a solution composed of 10 ml of HF, 15 ml HCl, 25 ml HNO_3 and 50 ml of distilled water known as Keller's reagent for 20s.

The samples were pressed through a H13 tool steel die with a square section with an area equal to 100 mm², intercepted at $\Phi = 90^o$ and $\Psi = 0^o$.

The billets were pressed by H13 tool steel punch 110 mm in length. Figure 1 shows the schematic drawing of one side of the die with the material positioned to the bottom of the vertical channel.

Optical microscopy was performed on a Nikon Eclipse LV150 reflective light microscope, associated with Nikon DS-Fi1 camera and NIS-Elements D 3.2 software. To obtain data on the morphology, the samples were submitted to SEM in the Secondary Electrons mode (SE), EVO model MA10 of the manufacturer Zeiss with LaB_6 filament with acceleration voltage 10 kV. In order to detect the present components, analyzes by dispersive energy spectroscopy, EDS, were obtained using a JEOL- JSM model 6460LV microscope. All the analysis were done on TD x DN plane.

Samples were deformed by ECAP using a universal machine EMIC model 23-600, with a maximum capacity of 600kN. The preparation of the extrusion was first done by lubricating the inner channels of the die with a dry lubricant, the MoS_2 base, then with the die still open, the die also lubricated and placed inside the die channel. After the adjustment, the punch was positioned exactly on the specimen, 70 mm from the top of the die and 40 mm from the bottom of the vertical channel. The test speed was maintained at 0.5 mm/min, that is, by assuming a nominal strain rate of $3.33 \cdot 10^{-4}$ s^{-1}. Each sample was deformed by two pressing passes. The first pass was performed at 180ºC for all the samples and the second pass, assuming route A, was performed at 130 and 180ºC. These

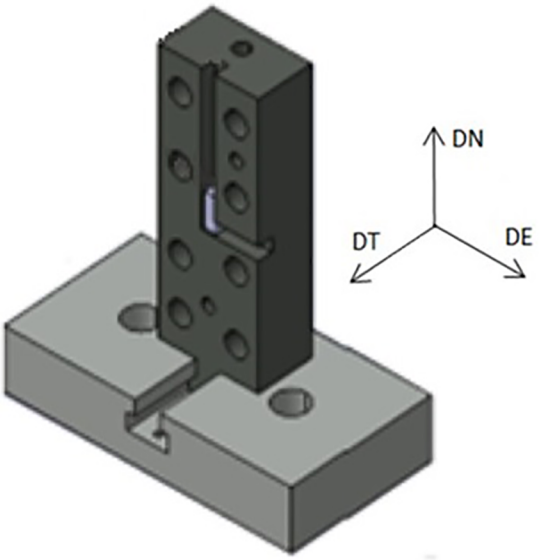

Figure 1. Schematic drawing of the matrix within sample positioned in the vertical channel[7].

temperatures were measured with a K-type thermocouple placed at the end of workpieces and the temperature control was performed by the on/off method on a circuit breaker connected to two 650W tubular resistors placed in the specific openings for them in the ECAP die. The tests were interrupted when the punch reached 35 mm from the stroke, 5 mm to the end of the inferior channel.

ECAPed samples Vickers microhardness were measured by performing 16 indentations, with a load of 100gf and a loading time of 20s in the TD x ND plan of the sample with a distance of 1mm from the edge, thus neglecting the edge effects. Compression tests were also realized in a universal machine used on ECAP process. These tests were conducted at a constant speed of 0.5 mm/min. The criteria for stopping the tests were displacement of 15mm of the movable (upper) plate. A typical value of 71.7 GPa was adopted for the elastic modulus of Al 7075-T651 alloy. Then, the yield stress, $\sigma_y^{0.2}$, was calculated by offset method. The material plastic parameters, i.e., mechanical strength, K, and hardening exponent, n, were obtained by fitting the nonlinear true stress-strain curves through Hollomon model.

3. Results and Discussion

3.1 Microstructural characterization

The second pass by ECAP was performed at 130°C and 180°C, due to material low ductility observed at lower temperatures and therefore failed to crack[7,8].

Table 1. Chemical composition of alloy Al 7075 (in mass fraction, %)

Al	Zn	Mg	Cu	Fe	Cr	Si	Mn	Ti	Others (total)
90.1	5.52	2.29	1.41	0.209	0.203	0.12	0.0473	0.0208	0.0799

The analysis by optical microscopy in the TD x ND plane is shown in Fig. 2, with a magnification of 100x and etch with Keller reagent for 20s. Fig.2a reveals the microstructure in the starting condition, this being the first pass by ECAP at 180ºC. Figure 2b presents the microstructure after the second pass at 130°C and Fig. 2c depicts the microstructure after the second pass at 180°C. One can observe that there is no significant changes between the as-received material and the second pass at both 130°C and 180°C. It can also be seen that in all the micrographs the grains are elongated as a consequence of the material pre-processing by hot rolling in the as-received condition.

The SEM analysis in the TD x ND plane in the secondary electron mode is shown in Fig. 3, with a magnification of 2000x and etch with Keller reagent for 20s. Fig. 3a displays the microstructure in the as-received condition while Fig. 3b presents the microstructure after the second pass at 130°C. Also, Fig. 3c reveals the microstructure after the second pass at 180°C. It can be noted that there were no significant changes between the as-received condition and the second pass at both 130°C and 180°C as the OM analysis. In the Fig. 3b it is possible to observed inside the red circle the presence of intermetallic phase. As previously discussed, in the images performed through optical microscopy the grains are also elongated in the SEM images, see Fig. 2.

The samples in all the conditions presented points and dark micro regions when analyzed by OM and by SEM in SE mode. These regions were better visualized and analyzed with the compositions performed on EDS analysis. Figures 4a and 4b show two distinct regions at different magnifications in the sample TD x ND plane after the first pass at 180°C. Figures 4c and 4f depict the EDS spectra from these evaluated regions. These regions denote insoluble phases and precipitate particles of Mg (Zn, Al, Cu)$_2$[9]. As could be confirmed on point 1, see Fig. 4a, and the respective spectrum shown in Fig. 4c, there is a presence of Mg and Zn, which possibly carry an intermetallic phase of Al-Mg-Zn. Point 2 in Fig. 4b shows the characteristic spectrum of Al-Mg-Cu, as observed in Fig. 4d. Point 3 in Fig. 4b is associated to the spectrum presented in Fig. 4e. Finally, in point 4 it was only noted Al-Cu precipitate, as can be observed in the respective spectrum displayed in Fig. 4f.

3.2 Mechanical properties

Figure 5 shows the material true stress-strain curves for as-received condition and after two ECAP passes at 130 and 180°C. The compression tests showed different behaviors according to the condition of the sample tested. As received material and after first pass at 180ºC it was observed both higher compression limit stress, CLS, yield stress and strength

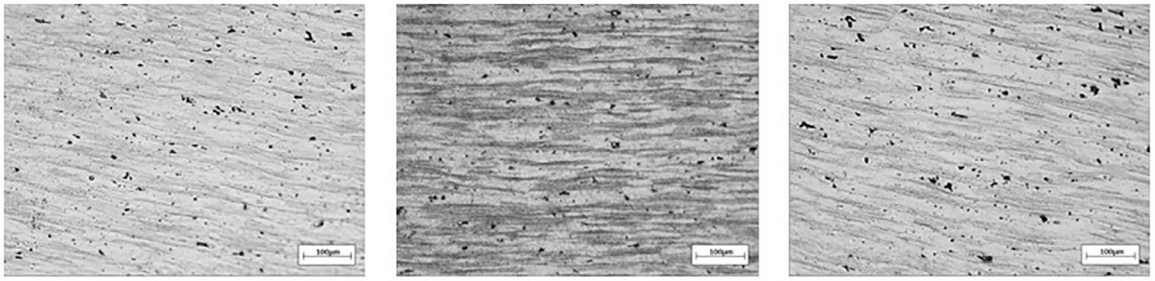

Figure 2. Microstructure analyzed by optical microscopy 100x magnification in TDxND plane, Keller 20s attack through: a) 1st pass at 180ºC b) 2nd pass at 130ºC, c) 2nd pass at 180ºC.

Figure 3. Microstructure analyzed by MEV, SE mode 2000x magnification, Keller 20s attack: a) 1st pass at 180ºC b) 2nd pass at 130ºC, c) 2nd pass at 180ºC.

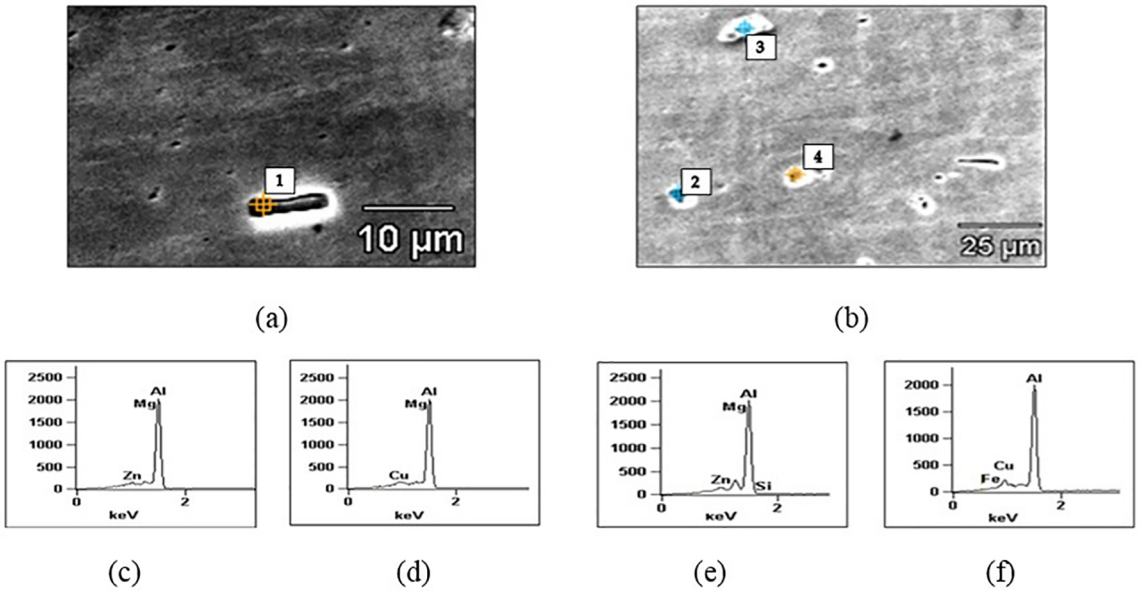

Figure 4. EDS analysis on start condition after 1st pass at 180°C a) Point 1; b) Point 2, 3 and 4; c) Spectrum point 1; d) spectrum point 2; e) spectrum point 3; f) spectrum point 4.

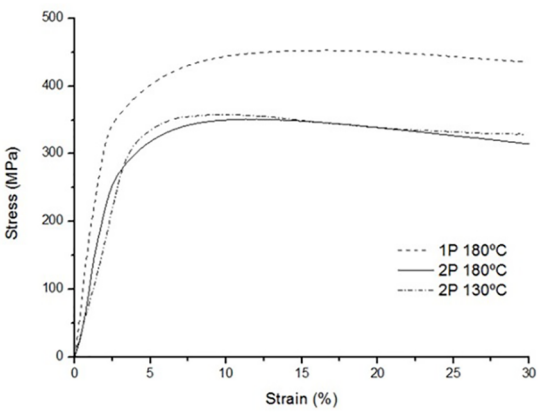

Figure 5. True Stress-strain curve of uniaxial compression test to analyzed conditions.

coefficient, K, but the lowest strain hardening exponent, n. The samples after the second pass at 130 and 180°C showed similar behaviors in the true stress- strain curve. However, the was observed an increasing of mechanical properties at 130°C, that is, CLS 2.0% higher, K 4.7% higher, n 9.6% higher and $\sigma_y^{0.2}$ 29.4% higher. These aspects are listed in Table 2.

The Vickers microhardness test differed from the behavior presented in the compression tests after the second pass through route A at 130ºC since the latter presented greater resistance to indentation. This resistance was close that obtained in the as-received condition, whereas for the true stress-strain curves one can be observe a great difference in the mechanical properties, i.e., it was higher than after pressing for two passes at 130ºC. The sample deformed by two passes at 180°C showed lower indentation resistance,

Table 2. Material mechanical properties.

Test	Compression limit stress (MPa)	K (MPa)	n	$\sigma_y^{0.2}$(MPa)	Vickers microhardness (HV)
1P 180ºC	453	588	0.155	359	129.1 ± 4.7
2P 130ºC	358	486	0.197	306	130.8 ± 4.4
2P 180 ºC	351	463	0.178	216	115.6 ± 7.4

which is consistent with the true stress-strain curve shown in Fig. 5, since this condition provided lower K-values.

The increase of dislocation density attributed to the material during ECAP reaches a saturation point from which the recovery phenomenon stands out on the increase of the mechanical strength and a decreasing in the hardness occurs for more processing passes. It can explain the decreasing in mechanical strength observed from the starting material for the second pass at both 130 and 180°C[7,8,10,11]. Another phenomenon that can occur at these temperatures is overaging with precipitates growth and transformation to more stable phases.

4. Conclusions

The behavior of Al 7075-T651 alloy processed by two passes of ECAP via route A at 130 and 180ºC was investigated in terms of microstructure changings and macroscopic mechanical properties. The following conclusions can be outlined:

- No noticeable microstructural changes were observed by evaluation through MO and SEM techniques. It is necessary complementary evaluations by Transmission Electron Microscopy, TEM and Electron Backscatter Diffraction, EBSD, to confirm grain refinement and quantify high angle boundaries fraction;

- The coarse precipitates observed by OM are composed by of Al, Mg, Cu, Zn. TEM investigations can be employed to determine morphology and dimensions of precipitates;

- In terms of mechanical behavior, it was observed through stress-strain diagrams and Vickers hardness tests, that the material presented higher strength after single pass at 180°C. Also, performing the second pass at 130ºC, it was obtained an intermediary resistance. Then, the material lesser hardening capability was achieved after second pass at 180ºC.

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