Microstructure, accumulated strain, and mechanical behavior of AA6061 Al alloy severely deformed at cryogenic temperatures

D C Magalhães, A M Kliauga, M Ferrante, V L Sordi
Universidade Federal de São Carlos, Department of Materials Engineering, 13565-9058 São Carlos, Brazil

Abstract. The combination of Severe Plastic Deformation (SPD) and cryogenic temperatures can be an efficient way to obtain metals and alloys with very refined microstructure and thus optimize the strength-ductility pair. However, there is still a lack of studies on cryogenic SPD process and their effects on microstructure and mechanical properties, especially in precipitation-hardenable aluminum alloys. This study describes the effect of low temperature processing on microstructure, aging kinetic and tensile properties of AA6061 Al alloy after cryo-SPD. Samples of AA6061 Al alloy in the solutionized state was processed by Equal-channel angular pressing (ECAP) at 77 K and 298 K, up to accumulate true strains up to 4.2. Results indicated that the aging kinetic is accelerated when deformation is performed at cryogenic temperature, dislocation density measurement by x-ray and diffraction analysis at TEM achieved a saturation level of 2x 10^{15} \text{m}^{-2} by ECAP at 298K and 5x10^{15} \text{m}^{-2} after cryogenic ECAP plus precipitation hardening. The same level of yield strength was observed in both deformation procedures but an improvement in uniform elongation was achieved by cryogenic ECAP followed by a T6 treatment.

Introduction

Among the Al alloys the AA6xxx series is usually employed in structural applications due to excellent mechanical strength, high corrosion resistance, good weldability and machinability[1, 2]. In the recent years, different alternative materials have been used to cryogenic conditions, such as aluminum alloys for building ships, liquid natural gas carriers, icebreakers and off shores plants that operated at low temperature in polar and subsea regions [3]. Thus, it is important to understand the mechanical behavior of the AA6xxx group particularly for the determination of processing parameters under different conditions. This alloy is normally thermomechanically processed above the precipitation hardening range, which yields a coarse grained structure, which will be further age hardened in order to achieve higher yield strength. The reported severe plastic deformation (SPD) of this alloys is done in the overaged state [4,5], or shortly after solid solution treatment [6,7] , that is during natural ageing, because their ductility at the peak hardened state its very low. There is few information about the interaction of
precipitation after SPD for this group of alloys. One of the purposes of this work is thus to study the
deformation behavior of the AA6061 alloy at cryogenic temperatures and the influence of post precipitation heat treatments on the mechanical properties.

1. Materials and Methods

Initially, samples of AA6061 Al alloy were submitted to solution heat treatment at 530 °C for 2 hours to obtain a supersaturation solid solution (SSSS). Some specimens were submitted to aging heat treatments at 298 K (natural aging – T4), 173 K (in salt bath – 173K T6), and 443 K (in salt bath – 443K T6).

The tensile tests were performed at 298, 173 and 77 K. The test at 173 K was performed in a closed cryogenic chamber INSTRON model 3119-610; the temperature was measured and controlled throughout the test making use of a type T thermocouple. The 77 K test was conducted with the specimen completely immersed in liquid nitrogen.

Prior to ECAP the samples were solution heat treated at 803K followed by quenching in water; for the room temperature route or in liquid nitrogen for the cryogenic route (see sketches in Figure 1a). The ECAP deformation was performed in a die with a corner angle of 120° and an external fitting angle of 22°, route Bc was applied. The deformation was performed either at room temperature or in the same cryogenic chamber used for the tensile tests, as sketched in Figure 1b. For the low temperature process the samples were maintained in liquid nitrogen between passes and reprocessing took only few minutes. The samples were deformed up to a equivalent deformation of 4.2. After deformation precipitation heat treatments were performed at 298 K (T4) or at 373 K (T6).

![Figure 1. Schematic representation of the thermomechanical procedure (a) and of the cryogenic experiment (b).](image)

Vickers microhardness measurements were carried out after different aging times. Miniature samples with a gauge length of 7 mm and 3 mm x 2 mm cross section area were employed for the tensile tests in the ECAP plus heat treated samples, tested at room temperature and at a nominal strain rate of $1 \times 10^{-3} \text{s}^{-1}$, with the elongation monitored by an optical extensometer.

The dislocation density was measured by x-ray diffraction in a SIEMENS D5000 equipment using Cu Kα radiation. Background subtraction was performed by using, alternatively, Si powder standard. The instrumental contribution was subtracted from the peak breadth, according to Cagliotti’s equation [8]. On those diffractograms, peak broadening was analyzed by the Williamson-Hall (W-H) method [9], modified by Warren [10] and Ungár [11-12] by using the following equation:

$$
\frac{B_{\text{peak}} \cos \theta}{\lambda} = \beta W_g = \frac{1}{d} + \left( \frac{\pi M^2 b^2}{2} \right) \rho^{1/2} K^2 C
$$

(Equation 1)
where $d$ is the diffraction domain size, $\lambda$ is the wavelength of the radiation, $\beta$ is the twin density, $W_g$ are the Warren constants related to the stacking faults, $p$ is the dislocation density, $b$ is the Burgers vector, $M$ is a constant related to the cut-off radius of dislocations (which is smaller for more compact arrays), $C$ are the average contrast factors of dislocations and $K=2\sin(\theta)/\lambda$.

Samples for transmission electron microscopy (TEM) were prepared by electrolytic polishing (20% HNO$_3$ in methanol, 20V - 30°C) and observed in a CM120 FEI microscope.

2. Results and Discussion

Figure 2 shows the hardening behavior of the 6061 alloy at room temperature (T4) and at 373 and 443 K (T6). The temperature is an important factor to aging kinetics for AA6061 Al alloy. Detailed studies of precipitation sequence of AA6061 alloy can be found in [13-16]. The simplified precipitation sequence of AA6061 [13] can be described as super-saturated solid solution (SSSS) $\rightarrow$ clustering stage $\rightarrow$ Mg, Si co-clusters $\rightarrow$ Guiner–Preston (GP)-I zones, pre-$\beta'' \rightarrow \beta'' \rightarrow B'$, $\beta' \rightarrow \beta$-$Mg_2Si$.

Various other precipitates [14] have also been reported. The early stages of aging resulting in the formation of Mg, Si co-clusters [15]. The natural aging, or T4 treatment, is very accentuated for this alloy, and after three hours it is already effective. In this condition, the microstructure is formed mainly cluster of Si and Mg, also called co-clusters. Other important feature is about the T6-443K treatment which reach a peak around 18 hours. This maximum is associated with formation of $\beta''$ needles precipitates in the aluminum matrix. For the T6 treatment at 443K, shown in Figure 3, the major part or the precipitates are the pre-$\beta''$ and $\beta''$ needles are in the early stage of formation.

![Figure 2. Hardening curves of the AA6061 alloy at room temperature (T4) and at 373K (T6).](image1)

Xu, Roven and Jia [17], using an AA6060 alloy in T6 condition, determined significant changes in fracture surfaces after tensile tests at 77 K in comparison to room-temperature. Furthermore, the mechanical behavior was strongly affected by testing temperature, and the deformation mechanism by dislocation slip is more homogeneous at low temperature deformation. Esmaeili et al. [18] examined the AA6111 alloy subjected to tensile test between 298 K and 4.2 K under different precipitation treatments, indicating a complex behavior depending on the nature of the obstacles to the movement of dislocations, which implies strong changes in strain rate sensitivity and work-hardening response. Halim and co-authors [19] carried out tensile tests at 298 K and 223 K with AA5754 Al alloy, and their results indicated that the decrease of the test temperature inhibits the Portevin-Le Chatelier effect (PLC), leading to a more homogeneous distribution of solute atoms and dislocations than at room-temperature. All the above investigations concluded that both mechanical strength and ductility increase at low temperatures [17-21]. Fig. 4 shows the true stress true strain curves obtained at 298 K, 173 K and 77 K, with the alloy in the solid solution condition. The stress-strain curve at room temperature is characterized by the presence of the Portevin-Le Chatelier effect, which was suppressed at 173K and 77 K. A higher uniform elongation was obtained at 77 K as well as higher yield stress and strain hardening. This results are summarized in Table 1.
Figure 4. Tensile curves for solution-treated samples of AA6061, at 298 K, 173 K and 77 K, with respective work-hardening exponent. Magnification of the initial region of the curves (insert) shows the PLC effect at room-temperature. Initial strain-rate equal to 10^{-3} \text{s}^{-1}.

Table 1. Summary of the tensile properties of AA6061 Al alloy in the solution heat-treated condition, at different testing temperatures. Strain rate = 10^{-3} \text{s}^{-1}.

| Test temperature | \(\sigma_o\) (MPa) | \(\sigma_u\) (MPa) | \(\varepsilon_t\) (%) | \(\varepsilon_u\) (%) | RA (%) |
|------------------|------------------|------------------|---------------------|---------------------|-------|
| 298 K            | 54.5             | 150.2            | 30.1                | 20.2                | 76.9  |
| 173 K            | 60.1             | 158.0            | 33.2                | 23.0                | 72.0  |
| 77 K             | 72.3             | 233.6            | 46.6                | 35.1                | 61.6  |

Note: \(\sigma_o\)= yield stress; \(\sigma_u\)= ultimate stress; \(\varepsilon_t\)= total elongation; \(\varepsilon_u\)= uniform elongation; RA = reduction of area.

Figure 5 shows the samples deformed after 6 passes at room temperature and at 123 K. The room temperature sample presented cracks after the fourth pass and fracture was very evident after the sixth pass, whereas no fracture was observed at 123 K. Observation of the deformed samples in optical microscope showed that the strain was more homogeneous distributed at room temperature than at 123 K where deformation in macroscopic was more concentrated in macroscopic shear bands, as shown in Figure 6. Complementary to this information the hardness measurements just after deformation shows that the saturation level was reached faster at room temperature than for the cryogenic route. (see Figure 7). This indicates that the higher ductility at low temperature may be correlated to the strain distribution as well as to the absence of precipitation hardening.

After 6 passes the hardness for the two sets of samples was very similar, but the degree of grain subdivision at room temperature was more accentuated than at 123 K (see the TEM micrographs in Figures 8a - and 8c with the respective diffraction patterns in Figures 8b and 8d).

Figure 5. ECAP deformed samples after 6 passes

Figure 6. Optical microscopy of ECAP samples after 2 passes. a) room temperature; b) 123K
Figure 7. Hardness evolution after ECAP at room temperature and at 123K. Measurements performed just after deformation for both temperatures.

Figure 8. TEM micrographs of the AA6061 alloy after ECAP at room temperature and at 123K plus (T4).

There was no accentuated difference in the dislocation density after 6 passes at room temperature and at 123K in the natural aged condition. The recovery process at 373K (T6) decreased the dislocation density at the same rate for both samples, as shown in Figure 9a. Figure 9b shows the hardness evolution after six ECAP passes for the T4 and the 373 K - T6 treatments. The hardness evolution at T4 condition was very similar for both sets of samples. Whereas for the T6 condition the cryogenic samples presented a faster precipitation kinetics, which demonstrates the effectiveness of the cryogenic process in suppressing the early stages of clustering.

Figure 9. a) Dislocation density evolution after 6 ECAP passes at room temperature and 123 K plus heat treatments in T4 and T6 conditions. b) Hardness evolution.

As for hardness, the yield strength for the room temperature and cryogenic routes were very similar after 80 aging hours. Figure 10 shows the results of uniform elongation after ECAP plus T4 and T6
heat treatments for both processing routes. The cryogenic deformation showed an improvement of this property in comparison with the room temperature route.

![Figure 10](image.png)

**Figure 10.** Uniform elongation obtained in tensile tests after 6 ECAP passes at room temperature and 123 K plus heat treatments in T4 and T6 conditions.

**Conclusions**

Deformation at cryogenic temperatures successfully suppressed the formation of \( \beta^" \) and made possible the SPD process without the superimposed effect of precipitation hardening of the AA6061 alloy. The subsequent T6 treatment yielded higher uniform elongation for the deformed material.

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