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MECHANICAL PROPERTIES AT HIGH TEMPERATURE OF AN AA3004 AFTER ECAP AND COLD/HOT ROLLING

M. Cabibbo[1], M. El Mehtedi[1], L. Barone[1], E.F. Prados[1,2] and M. Ferrante[2]

[1]Department of Mechanical Engineering, Marche Polytechnic University, Via Brecce Bianche, 60131-Ancona, Italy
[2]Department of Materials Engineering, Federal University of São Carlos, Avenida Washington LuHs km 235 , 13565-905, São Carlos (SP), Brazil

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Abstract. Mechanical properties of aluminium alloys can be considerably improved using severe plastic deformation techniques, due the induced dramatic grain refinement. In the present work an extruded AA3004 was severely deformed by Equal Channel Angular Pressing (ECAP) to reduce the grain size down to sub-micrometric scale. Route A was used and the alloy was deformed up to $\varepsilon = 4.32$ (4 passes). The ECAP alloy was then cold and hot rolled (CR and HR). Tensile tests were conducted at two temperatures (523 and 573K) at strain rates of $1 \cdot 10^{-2}$, $1 \cdot 10^{-3}$, and $1 \cdot 10^{-4}$ s^{-1}. The CR and HR tensile response, prior and after ECAP, were compared and discussed. The best mechanical response was obtained in the ECAP+CR and ECAP+HR at 573K. Conversely, the obtained good ductility was insensitive to the strain rate.

1. INTRODUCTION

The 3004 aluminium alloy is widely used in the container, packaging, and automobile industry, because of its excellent specific strength, corrosion resistance and formability. In the last decades, several studies focused on the mechanical behaviour and the microstructural evolution of the AA3004 [1-4]. Yi-Lin *et al.* [1,2] studied the microstructure evolution of the AA3004 submitted to tensile tests, after a pre-deformation cold rolling. They reported a strong influence of the pre-deformation on the resulting alloy mechanical response. Peng *et al.* [3] investigated the temperature and strain rate effect on the alloy mechanical properties. They found that AA3004 shows serrated flow behaviour in the temperature range of 253–423K at strain rates of $5.56 \cdot 10^{-5}$ to $5.56 \cdot 10^{-3}$ s^{-1}. Dirras *et al.* [4] studied cyclic shear tests at various amplitudes showing the macroscopic behaviour being strongly dependent on the alloy initial state (recrystallised, recovered or extra-hardened).

Severe plastic deformation processes, such as Equal-Channel-Angular-Pressing (ECAP), are currently the subject of extensive investigations. ECAP is able to produce alloys with improved mechanical properties, such strength, yield stress, toughness and hardness. These mechanical properties are directly linked to the dramatic grain refinement the material experience during the severe deformation [5]. Although ductility loss appears to be a general trend, it is important to note that the reduction in

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Corresponding author: M. Cabibbo, e-mail: m.cabibbo@univpm.it ductility is generally less than in more conventional deformation processing techniques such rolling, drawing, and extrusion [6]. For instance, Horita *et al.* [7] in an room temperature deformed AA3004 found that the total elongation is significantly lower by cold-rolling than by ECAP, with a stronger effect as the accumulative strain rises.

As for the industrial application of ECAP, there is some evidence that the microstructure grain refinement dos not involve any significant sample size modification. This was pointed out by Yamagushi *et al.* [8] on a series of ECAP experiments dealing with samples of different cross section, in which like tensile strength and grain size were obtained. However, practical applications of ECAP will strongly depend on the resulting mechanical and microstructure properties achievable by combining the ECAP process to conventional forming operations (such CR and HR). With this respect, in an AA6101 it was observed that 1 ECAP pass followed by conventional extrusion led to better mechanical properties in the extruded materials with only a limited reduction in ductility [9]. On the other hand, Akamatsu *et al.* [10], in an Al-3Mg+0.2Sc alloy found that ECAP process can exhibit superplastic behaviour. The superplasticity was also reported for the same alloy subjected to 8 ECAP passes followed by cold rolling.

The aim of this study is to explore the possibility of improving the CR and HR of an Al-Mn alloy using the ECAP process prior rolling. Route A was used in order to keep the uniaxial plastic deformation material path into the ECAP die, before entering the rolling gates, and to make the ECAP + rolling process as simple as possible. The AA3004 was deformed through ECAP up to a strain of $\varepsilon = 4.32$. The results focused on the role of ECAP in the CR and HR alloy mechanical response.

2. EXPERIMENTAL DETAILS

Experiments were conducted on an AA3004 whose composition (wt.%) is as follows: 1.09Mn, 1.08Mg, 0.55Fe, 0.20Si, 0.19Cu, 0.01V, 0.01Ti, Al balk. The material was supplied by Hydro Aluminio Acro S.A. Brazil as extruded rods of 20 mm in diameter. Extruded mean grain lateral spacing was (10 ± 1) μm. For the Equal-Channel Angular Pressing, the material was machined into rods with a diameter of 10 mm and a length of 90 mm. The material was pressed at room temperature into a solid die using a hydraulic press machine of maximum capacity of 150 kN with a linear speed of ~ 4 mms^{-1}. The die L-channel had an angle of $\Phi = 90°$ and an outer arc of curvature of $\Psi = 20°$ at the two channels intersection. The two channels had an equal cylindrical cross section of 10 mm. Using this die configuration the imposed strain at each pass was of $\varepsilon = 1.08$ [11]. Each sample was repetitively pressed up to four passes (in the following indicated as 1X to 4X), that is to a maximum strain level of $\varepsilon = 4.32$. Samples were pressed along the extruded direction using the Route A, i.e. no rotation of the rods between passes [12].

Microstructure inspections were carried out using polarized optical microscopy (POM) along the Y-plane (i.e. the plane containing the pressing and the transverse directions) [13]. Surfaces were chemically etched for few seconds using a solution of 5 pct. of fluoroboric acid (HBF_4) in ethanol at room temperature and V=18 V.

ECAP specimens were reduced to a thickness of 9.5 mm in order to cold rolling (CR) and hot rolling (HR) at 473K in either case to a final thickness of 2 mm. So that, the specimens were rolled to a thickness reduction of 80% and to an equivalent strain of $\varepsilon = 1.80$ ($\varepsilon = 1.15\ln(t_i/t_f)$, where t_i and t_f are the initial and final specimen thickness respectively). Rolling was performed along the ECAP pressing direction, inducing a thickness reduction on the Y-plane.

Vickers hardness measurements were carried out along the ECAP Z-plane (plane containing the pressing and normal directions), and accordingly on the rolled materials. Hardness values were averaged from a minimum of 15 measurements.

Tensile tests were performed parallel to the ECAP pressing and the CR/HR directions. All the specimens had gauge length of 5 mm and cross-section area of 12 mm^2. Tests were conducted to failure at 523 and 573K at a constant displacement rate and strain rates of $\dot{\varepsilon} = 1\cdot10^{-2}$, $1\cdot10^{-3}$, $1\cdot10^{-4}$ s^{-1}. Tests were conducted on ECAP rods (1X and 4X), on CR and HR plates, and on the ECAP+CR, ECAP+HR specimens.

3. RESULTS AND DISCUSSION

Fig. 1 shows representative POM images of the as-extruded microstructure (Fig. 1a), ECAP-4X (Fig. 2b), and ECAP-4X+HR (Fig. 2c). The as-extruded microstructure is highly anisotropic with a grained structure extremely elongated (aspect ratio >5 and lateral spacing of 10 μm). Grain lateral spacing distribution (inset in (a)) shows that the spacing spans from a minimum of 2 to a maximum of 27 μm. This relatively narrow size range reveals a sufficient homogeneity in the grained elongation induced by the extrusion process. In the ECAP-4X, shear bands

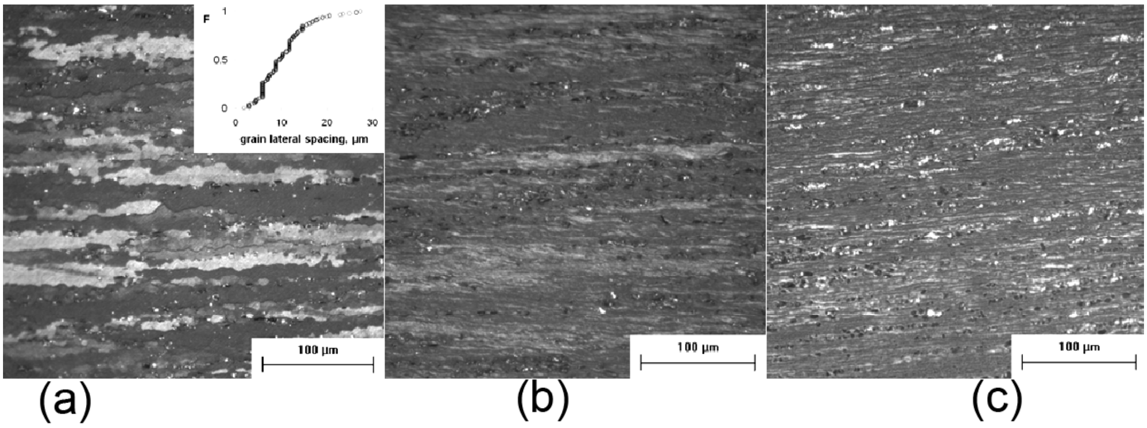

Fig. 1. POM images of the AA3004 alloy: (a) as-extruded, (b) ECAP-4X, (c) ECAP-4X+HR at 473K. Inset in (a) shows the grain lateral spacing distribution.

appear essentially oriented along the prior extruding direction. Furukawa and co-workers [13], characterized the shearing patterns for ECAP in various conditions and reported shear band oriented at an angle of 10° to the pressing direction for route A when $\varphi = 90°$. Yet, in this case the ECAP was performed in an extruded alloy and here the shear bands have shown a much lower angle respect to the pressing direction. Microstructure of the ECAP-4X and ECAP-4X+HR reveal a significant grain fragmentation and braking of the highly anisotropic as-extruded microstructure. That is, a refined and more homogeneous microstructure is produced by the severe and rolled uniaxial deformations.

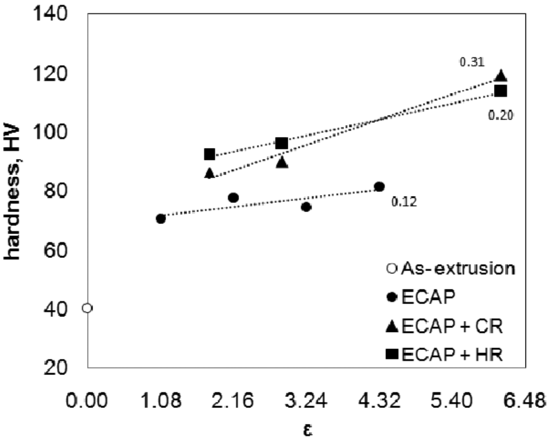

Fig. 2. Hardness vs. accumulative strain for the ECAP (1X-to-4X), ECAP+CR and ECAP+HR conditions. Linear unit increment in the three cases is also reported. Hardness experimental errors are within the datapoint.

Fig. 2 reports hardness as a function of accumulative straining, up to $\varepsilon = 6.12$: ECAP-1X-to-4X, ECAP+CR, ECAP+HR. These plots clearly show an hardness increase with accumulative straining in all the conditions. ECAP-1X hardness increment was more than 50% with respect to the as-extruded value of 40HV. On the other hand, the ECAP+CR and ECAP+HR hardness more than doubled.

The linear unit increment of hardness with strain for the ECAP condition was 0.12, while in the ECAP+CR and ECAP+HR was 0.32 and 0.20, respectively. This ECAP hardness increment with accumulative strain is fully consistent with other literature findings [14,15]. The almost 3-times higher hardness unit increment in the ECAP+CR, with respect to the ECAP, indicated the relevant room temperature microstructure hardening effect of the combined uniaxial plastic deformation to which the material was subjected. The much lower unit increment of hardness in the ECAP-HR is due to a general re-organization of the cell and grain boundaries which tend to evolve into a recovered structure. That is, dynamic recovery occurs during the hot-rolling, which, in turn, induces a lower hardness increment with accumulative straining. Yet, the value of 0.20 is still some 50% higher than the unit increment of the ECAP condition and then recrystallization did not occur during post ECAP hot rolling. (See also Fig. 1c).

Stress-strain curves at the strain rate of $1\cdot10^{-3}$ s^{-1} at 523 and 573K and at 523K with decreasing strain rate ($1\cdot10^{-3}$ s^{-1} and $1\cdot10^{-4}$ s^{-1}) are reported in

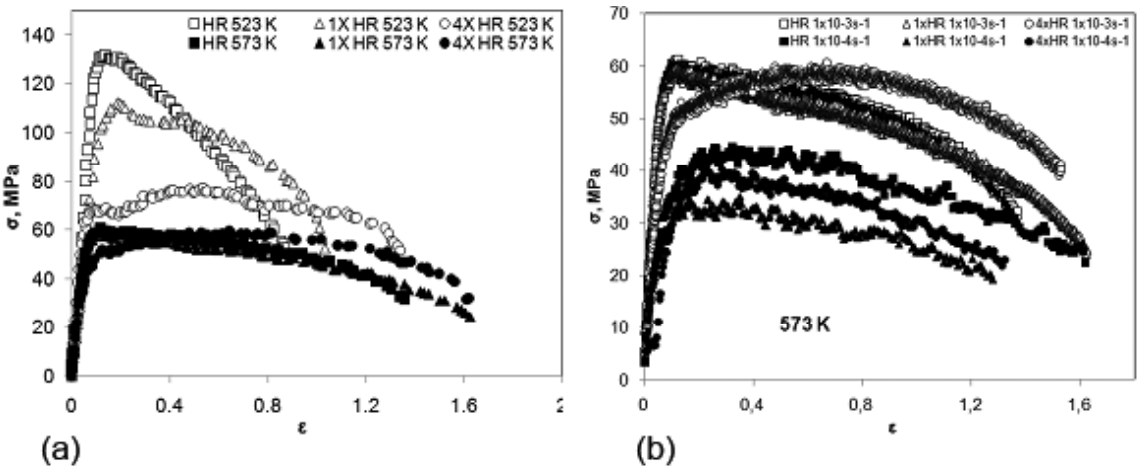

Fig. 3. Tensile stress-strain curves at $\dot{\varepsilon}=1\cdot10^{-3}$ s^{-1} of the AA3004 alloy (523 and 573K) (a), and at 573K for decreasing strain rate ($\dot{\varepsilon}=1\cdot10^{-3}$ s^{-1}, $\dot{\varepsilon}=1\cdot10^{-4}$ s^{-1}) (b). The hot-rolling (HR), 1X and 4X ECAP with subsequent hot-rolling at 473K conditions are reported.

Figs. 3a and 3b. The flow stress decreases with increasing temperature and decreasing strain rate. The effect of ECAP on the flow curves is remarkable at 523K, whereas it tends to be negligible at 573K, because of recovery and recrystallization phenomena. At 523K, the flow stress decreases with increasing prior-ECAP straining. On the other hand, ductility increases, yielding a better hot workability of the alloy in these experimental conditions.

Fig. 4 shows the elongation to failure as a function of the strain rates for all the different experimental conditions of this study. Results show that the material did not exhibit a superplastic behaviour, as the strain rate sensitivity of the flow stress, m, ranged 0.21 to 0.29 at 523K, and 0.12 to 0.31 at 573K.

The highest elongation was recorded at the lowest strain rate in all the experimental conditions: 100 to 150% at 523K, and 120 to 160% at 573K; the elongation to failure increased with temperature, as well. At the same time, elongation reduced with strain rate at both temperatures (Figs. 4a and 4b). The only exception to this trend was at $\dot{\varepsilon}=1\cdot10^{-3}$ for ECAP-4X+HR and ECAP-1X+CR, where the elon-

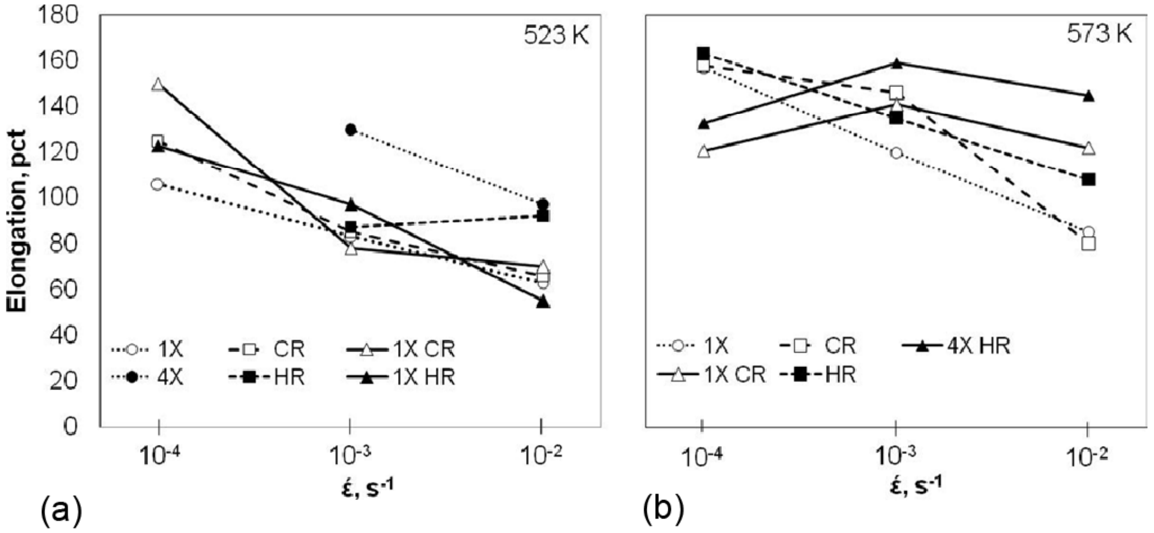

Fig. 4. Elongation to failure *vs*. strain rate ($\dot{\varepsilon}=1\cdot10^{-2}$, $1\cdot10^{-3}$, $1\cdot10^{-4}$) (a) at 523K and (b) 573K.

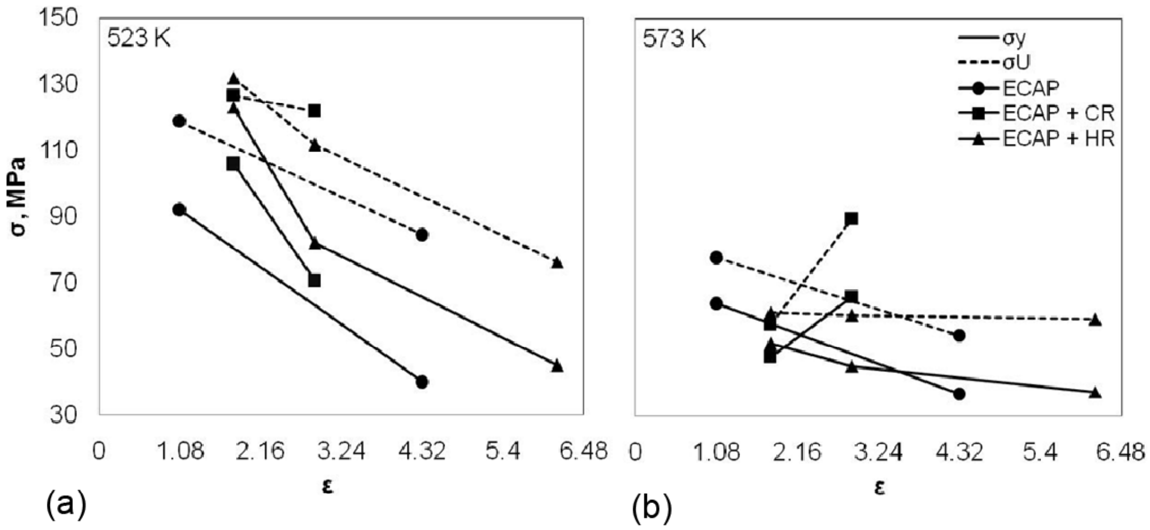

Fig. 5. Yield strength (YS, σ_y) and ultimate strength (UTS, σ_U) vs. accumulative strain at $\dot{\varepsilon}=1\cdot10^{-4}$, for the ECAP, ECAP+CR, and ECAP+HR conditions. Solid lines refer to YS, dotted line to UTS. (a) 523K, (b) 573K.

gation to failure peaked at 160 and 145%, respectively. This is an important result, as it indicates that the best mechanical response was given by ECAP+HR and ECAP+CR, where, at 573K, the good ductility is not reduced with the strain rate. The microstructure refinement induced by ECAP, coupled with the grain boundary sliding and grain rotation of the non-equilibrium boundaries [6], may explain the showed good ductility with the strain rate. Fig. 5 reports the yield strength (YS, σ_y) and the ultimate strength (UTS, σ_U) as a function of strain for both temperatures (523 and 573K). ECAP YS and UTS decrease with accumulative strain at both temperatures. This is a well known and expected behaviour due to the highly refined grained structure which is also thermodynamically unstable [16]. An even more pronounced strength shrink with strain was observed in ECAP+CR and ECAP+HR at 523K. At 573K, the accumulative strain seams to not significantly affect the yield and ultimate strengths in the ECAP+HR conditions.

4. CONCLUSIONS

This paper focused on the possibility of using the ECAP process in the cold and hot rolling of an extruded AA3004. The alloy uniaxial plastic deformation path was maintained using route A in ECAP. It was found that at different strain rates ($\dot{\varepsilon}=1\cdot10^{-2}$, $\dot{\varepsilon}=1\cdot10^{-3}$, $\dot{\varepsilon}=1\cdot10^{-4}$) the best elongation to failure response was given by ECAP+HR and ECAP+CR at 573K, with no significant ductility deterioration with strain rate. At 523K, the flow stress decreased with prior hot rolling ECAP straining, at the same time ductility increased, thus giving a much better alloy hot workability.

The linear hardness unit increment with strain was of 0.12 after ECAP, and 0.32 and 0.20 after ECAP+CR and ECAP+HR, respectively. The highest hardness unit increment in the ECAP+CR indicated a significant hardening effect on the combined two uniaxial plastic deformations.

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