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Microstructure and creep behaviour of an Osprey processed and extruded Al–Cu–Mg–Ti–Ag alloy

M. Eddahbi *, J.A. Jiménez, O.A. Ruano

Departamento de Metalurgia Física, Centro Nacional de Investigaciones Metalúrgicas, CSIC, Avda. de Gregorio del Amo 8, 28040 Madrid, Spain

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Abstract

This paper reports the microstructure and creep behaviour of an Al–Cu–Mg–Ti–Ag alloy processed via Osprey forming and extrusion. The microstructure of the as-received extruded bars showed elongated grains towards the extrusion direction, which contained a fine substructure. Elongation to failure values of about 25% were obtained at test temperatures up to 400 °C and for strain rates ranging from 10^{-4} to 10^{-2} s^{-1}. In this temperature range, the mechanical behaviour of the alloy was similar to that of particle-strengthened aluminium alloys: (1) the apparent stress exponents of $n = 17$ and 7 were higher than that reported for most aluminium alloys; (2) the microstructure and the texture did not change significantly during tensile deformation. At temperatures higher than 400 °C, the elongated grains of the microstructure started to recrystallize into smaller grains and a maximum elongation to failure of 170% was observed at 520 °C and 10^{-4} s^{-1}. Tensile tests performed at higher strain rates in this temperature range revealed a stress exponent of 4.5, which corresponds to a slip creep mechanism.

Keywords: Aluminium alloys; Microstructure; Texture; Creep; Deformation mechanisms; Osprey process

1. Introduction

During the last decades various processing techniques have been developed to produce ultra-fine grained materials, since this microstructure improves their properties at low and high temperature [1,2]. At low temperatures, a sub-micron grain size leads to higher elongation to failure and yield strength. Furthermore, at high temperature a fine-grained microstructure usually enables superplastic forming.

Processing new light aluminium (Al) and magnesium alloys have received considerable attention and the number of published papers has been increased considerably [3,4]. Rolling, extrusion, equal canal angular extrusion, accumulative roll bonding, rapid solidification, casting followed by rolling or extrusion and powder metallurgy (PM) are some of the processes used to develop new materials combining ultra-fine grained microstructures and high strength and ductility. A promising processing route to produce high quality and low cost Al alloys is the spray forming technique since the processing steps are reduced compared to other PM processes [5]. The application of high cooling and solidification routes result in an extension of solid solution and the formation of non-equilibrium phases that enhances the properties of the material.

Creep behaviour studies on a variety of different materials and metals systems have increased significantly the understanding of the deformation mechanisms responsible for the evolution of the microstructure during plastic flow. Specially, Al-based solid solution alloys, such as Al–Cu or Al–Mg alloys, and PM Al-alloys have been the subject of many creep investigations [6–8]. Coarse grained Al-based solution alloys exhibit generally three regions of deformation, depending on the stress σ (or strain rate $\dot{\varepsilon}$), characterized by its stress exponent, n [9,10]. At low applied stresses, region I, $n = 5$ and plastic deformation is controlled by dislocation climb mechanism [11]. In the intermediate stress region II the stress exponent is $n = 3$ and the dominant creep process is attributed to viscous-glide of dislocation creep [12]. In the high stress region III, creep is controlled by dislocation climb ($n = 5$) like in the low stress region. The creep behaviour in any of these regions is not depending on the grain size. However, when the grain size is fine, less than 15 μm, region II can be characterized by a stress exponent of 2 and high elongation to failure values. Under these conditions, grain boundary sliding is

* Corresponding author. Tel.: +34 91 5538900; fax: +34 91 5347425.
E-mail address: eddahbi@cenim.csic.es (M. Eddahbi).

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the dominant deformation mechanism and superplastic forming can be achieved [13–15].

The aim of this work is to study the mechanical and microstructural behaviour of a PM Al–Cu–Mg alloy produced by the Osprey process followed by extrusion. This alloy is similar to the as-wrought 2014 alloy widely used in the automotive industry in camshaft bearing caps. An approach to quantify the operating deformation mechanisms is presented.

2. Experimental procedures

The alloy of the composition Al–4.3%Cu–0.36%Mg–0.19Mn–0.44%Ti–0.3%Ag (wt.%) was produced by Osprey rotatory atomization and extrusion and supplied by PEAK Werkstoff GmbH, Germany. Details of this process have been described elsewhere [16]. The alloy was received as extruded bars of 20 mm in diameter. Chemical analysis of the as-received bars has shown that the material contained between 0.2 and 0.3 wt.% of oxygen and small amounts of nitrogen, inferior to 0.1 wt.% resulting from the spraying atmosphere. Scanning electron microscope (SEM) examination showed that no evidence of pores in the matrix and the dissolved oxygen is retained in the matrix as oxides particles.

It was reported that spray forming process results in microstructures containing metastable phase of Al_2Cu particles. To avoid these particles in the material, the as-received bars were solution annealed at 480 °C for 2 h followed by cooling in air to room temperature. This treatment assures a homogeneous microstructure that is alike for all samples prior to mechanical testing.

For mechanical characterization, cylindrical samples of 28 mm in gauge length and 3 mm in diameter were machined from the annealed bars with the extrusion direction (ED) parallel to the tensile axis. Elongation to failure tensile tests were carried out at temperatures ranging from 250 to 520 °C in the strain rates range from $\dot{\varepsilon} = 10^{-4}$ to 10^{-2} s^{-1}. Prior to tensile tests, the samples were heated at the test temperature for less than 7 min to stabilize the test conditions.

In addition, strain rate changes tests, SCT, were carried out in tension at different temperatures to determine the strain-rate-sensitivity exponent, m (or the stress exponent, n, which is the reciprocal value of m). In order to achieve a steady state equilibrium microstructure, each test was initiated at the strain rate of $\dot{\varepsilon} = 3 \times 10^{-3}$ s^{-1} until a steady state was reached. Then, the strain rate was decreased to $\dot{\varepsilon} = 10^{-5}$ s^{-1}, and the test was maintained until a new steady state was reached. After few percent of strain, the strain rate was increased stepwise up to $\dot{\varepsilon} = 4 \times 10^{-3}$ s^{-1}, and this procedure was repeated several times to obtain sufficient creep data (strain rate and the corresponding steady state stress σ). It is worth noting that the grain structure was kept constant during SCT at all test temperatures. This was supported by two mains facts. The first one is that the SCT was carried out in a quasi-steady state, varying the strain rate from low to high values in a small strain interval of less than 0.3. The second one is that equal values of stress were obtained at the strain rate of $\dot{\varepsilon} = 3 \times 10^{-3}$ s^{-1} at different strains during the SCT. At 520 °C, however, some dissolution of the Al_2Cu phase (particles) occurred during the SCT. Therefore, a second sample was used for testing to avoid the effect of the Al_2Cu dissolution on the microstructure, specifically on the formation of (sub)grains and grain growth. One of them was subject to SCT performed at lower strain rates ranging from $\dot{\varepsilon} = 10^{-5}$ to 10^{-4} s^{-1}, and the other at moderate and high strain rates ranging from $\dot{\varepsilon} = 10^{-4}$ to 10^{-3} s^{-1}. Both SCT were carried out applying a similar amount of strain of $\varepsilon = 0.3$. This strain was 0.15 and 0.2 at 300 and 400 °C, respectively.

Temperature changes were performed during tensile tests at strain rates of $\dot{\varepsilon} = 10^{-4}$, 10^{-3} and 10^{-2} s^{-1}. At each of these strain rates, the test temperature was increased and/or decreased sequentially in the temperature range from 250 to 500 °C. This technique permits to determine accurately the activation energy for creep and/or superplastic deformation. Fig. 1 shows a set typical deformation curves recorded from a temperature change test performed at a strain rate of $\dot{\varepsilon} = 10^{-3}$ s^{-1}. The first test was conducted at 500 °C until the steady state was attained. After decreasing the test temperature to 475 °C, a second test was run at the same strain rate of $\dot{\varepsilon} = 10^{-3}$ s^{-1} until a new steady state was attained. For further tests, the temperature was changed sequentially in intervals of 25 °C until the final test temperature of 250 °C. At this temperature, the test was stopped and the sequence of temperature changes was reversed, i.e. the temperature was increased up to 500 °C as illustrated in the figure. At this temperature the test was continued to failure. It should be noted that in each jump the temperature was reached in a short period of less than 3 min and was stabilized in 2 min. Fig. 1 shows that the same values of stress are obtained at a given temperature in the heating and cooling sequence. This proves that the creep data are not influenced by microstructural changes that may occur during the temperature changes.

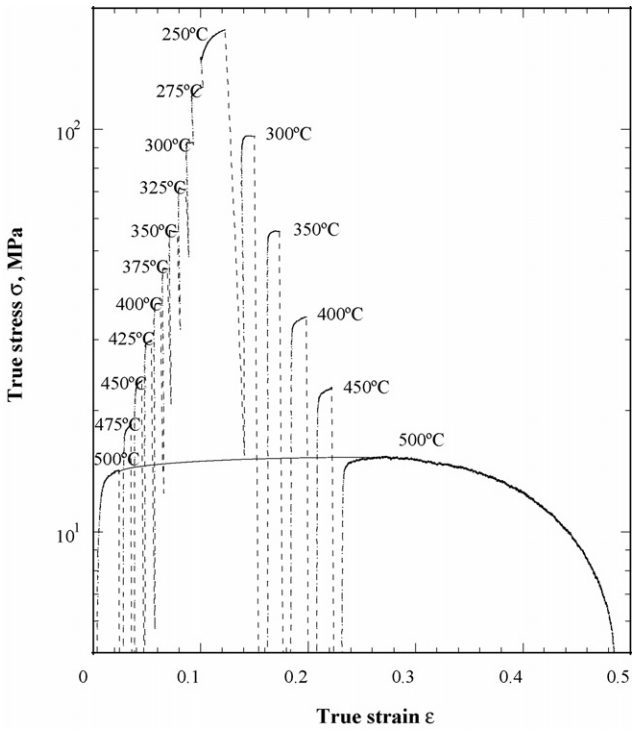

Fig. 1. True stress as a function of true strain corresponding to a series of temperature changes from 500 to 250 °C and vice versa from 250 to 500 °C, strain rate $\dot{\varepsilon} = 10^{-3}$ s^{-1}.

The samples of the as-received material and after deformation were prepared by conventional grinding and polishing techniques, followed by etching using, Keller's and Barker's reagents. Optical microscopy (OM) and scanning electron microscopy (SEM) were performed for metallographic examination.

Texture measurements were carried out by means of the Schulz reflection method, using a Siemens X-ray diffractometer equipped with a D5000 goniometer and an opened Eulerian cradle. Incomplete pole figures {1 1 1}, {2 0 0} were measured over a range of colatitude φ varying from 0° to 75° in the step mode with increment of $\Delta\varphi = 5°$. A textureless standard sample of pure Al was used for defocusing correction.

3. Results and discussion

3.1. Initial microstructure

Fig. 2a and b reveal the distributions of the second phase particles in the as-received material viewed on the longitudinal and transverse sections, respectively. Qualitative microanalysis performed with energy dispersive X-ray spectrometry showed two kinds of particles. Coarse particles, aligned along ED, were identified as Al_2Cu. At the interface between these particles and the matrix some oxygen was detected. The small particles, with a few nanometres in size, aligned in the ED correspond to Al_2O_3 particles. Theses oxides particles were introduced during the spray processing. Fig. 2c shows the microstructure of a longitudinal section of the as-received bar. The original powder particles are elongated as a consequence of the extrusion process.