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# Influence of microstructural stability on the creep mechanism of Al–7 wt% Si alloy processed by equal channel angular pressing

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# ABSTRACT

A Na-modified, as-cast Al–7 wt% Si alloy was processed by equal channel angular pressing (ECAP) up to 8 passes by route A at ambient temperature using a 90° square section die, obtaining improved strength, ductility and work of fracture. From the first pass, porosity is removed, the eutectic constituent is refined and the eutectic silicon particles are partially redistributed. Additionally, a fine and homogeneous strain-induced silicon precipitation occurs in the supersaturated solid solution retained in the casting. These fine precipitates assist in grain refinement, resulting in a 250 nm grain size after one pass and 210 nm after 8 passes. This microstructure cannot sustain grain boundary sliding because it coarsens rapidly even at the lowest testing temperatures. Deformation at high temperatures gives values of n of about 8 and values of the activation energy corresponding to the self-diffusion of aluminum, 142 kJ/mol, which can be rationalized by a constant substructure slip creep mechanism. These values are influenced by the presence and evolution of the fine intradendritic silicon precipitates. Coarsening of these precipitates with time and temperature increases their interparticle distance causing variations in experimental n and Q values.

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# 1. Introduction

Al–Si casting alloys are extensively used in the automotive and aerospace industries. This family of alloys has a wide range of possible industrial applications as a result of their low cost, good castability, moderate strength at room temperature and good wear resistance [1]. However, Al–Si casting alloys are poor in ductility and toughness. The influence of microstructural parameters such as dendrite size and Si particles shape and size have been studied extensively [2,3]. Many solutions have been proposed for controlling these parameters and improving the mechanical behavior, such as variations of solidification rates [4,5], additions of microstructure modifying elements [6], and performing various heat treatments [7].

Another approach to improve the mechanical properties of Al–Si casting alloys is to employ severe plastic deformation (SPD) which has been very effective in many alloy systems [8]. In the case of the Al–Si alloys different SPD techniques have been applied

<sup>1</sup> Present address: IMDEA Materials Institute, Eric Kandel 2, Getafe, Madrid 28906 Spain. showing how microstructural homogenization and refinement lead to improvement of the mechanical properties [9,10]. Techniques such as friction stir processing may be applied to selected areas of a casting to achieve localized modification of microstructure and improvement of properties [11]. However, such a process involves a complex thermomechanical cycle. In contrast, equal channel angular pressing (ECAP) involves essentially isothermal deformation and a well-controlled strain history. This technique consists in pressing a billet through a die formed by the intersection of two channels. The billet cross section remains constant after one ECAP pass, and so the processing potentially can be repeated many times. ECAP processing has been widely applied to many metals and alloys, and the grain refinement obtained has been shown to increase the room temperature strength [12]. Nevertheless, not all the potential of ECAP processing has been revealed when applied to hypoeutectic Al-Si alloys.

The study of mechanical properties at intermediate to high temperatures has received little attention for these alloys. However, the beneficial microstructural changes produced by severe plastic deformation may improve considerably the creep resistance as observed in aluminum alloys [13,14] or may lead to superplastic behavior [15,16], which are of interest for high temperature applications or processing, respectively. Therefore,

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the aim of this paper is the mechanical properties improvement of the Al–7 wt% Si alloy at intermediate-high temperatures by modifying its microstructure employing ECAP processing using route A, as well as the determination of the operating creep mechanisms.

# 2. Experimental procedure

The hypoeutectic Al–7 wt% Si alloy was prepared by melting appropriate proportions of pure aluminum (99.99 wt%) and eutectic Al–12.3 wt% Si master alloy and casting into an ingot 350 mm  $\times$  210 mm  $\times$  70 mm in size. A 0.02 wt% Na addition to the melt was employed in order to obtain a finer eutectic constituent. X-ray radiography was employed to assess porosity. The samples were machined from areas with the least apparent porosity.

ECAP billets,  $90 \times 10 \times 10$  mm<sup>3</sup>, were machined from the ascast ingot. ECAP processing was performed at room temperature using a sharp-cornered 90° ECAP die having die channels of square cross section. A schematic of the ECAP die used in this study is shown in Fig. 1. In this diagram, the plane where the simple shear occurs is located (marked with arrows) in the intersection of the two channels. FP, TP and CP denote the flow plane, top plane and cross plane after pressing respectively. Up to 8 repetitive ECAP passes were imposed using route A, which involves pressing without sample rotation between consecutive passes leading to a monotonically increasing strain.

The eutectic microconstituent distribution of the as-cast, 1p and 8p ECAP processed material was examined using an Olympus BH-2 optical microscope. For that purpose, the samples were ground and polished to a colloidal silica finish.

The (sub)grain size and small precipitates in the ECAP processed material were analyzed by means of a Zeiss Neon 40 field-emission scanning electron microscope (SEM) using the backscattered electron (BSE) signal. The samples were ground and polished to a colloidal silica finish and then electropolished using a solution consisting of 200 ml perchloric acid, 700 ml ethanol and 100 ml glycerol. Electropolishing was conducted at a potential of 15 VDC and temperature of -20 °C.



**Fig. 1.** Schematic illustration of an ECAP pass. The plane of the die channel intersection, where simple shear occurs as the billet passes through, is marked with arrows. FP, TP and CP denote the flow plane, top plane and cross plane after pressing respectively.

Grain size was measured on the primary Al constituent in the processed condition using BSE images. Scanning electron micrographs were analyzed using the Sigma Scan Pro software in order to obtain the size distribution of the aluminum matrix grains. The grains are not equiaxed in the processed alloy and so the minimum axis dimension of the grain was used as the grain size. More than 400 grains for each processing condition were analyzed. Size distribution histograms obtained from these measurements were conducted. Grain size data fell into lognormal distributions, so the geometric mean value was chosen as a measure of their size.

Planar dog-bone tensile samples with  $6 \text{ mm} \times 2 \text{ mm} \times 1.8 \text{ mm}$ gage dimensions were electro-discharge machined. The samples were machined parallel to the flow plane (FP), in such a way that the gage section coincided with the middle region of the ECAP samples avoiding frictional effects. Tensile samples were tested using a universal Instron 1362 testing machine equipped with a four-lamp ellipsoidal furnace to evaluate the mechanical behavior in the temperature range 200-400 °C. A set of tensile tests was performed at elevated temperatures and at constant cross-head speed of 0.065 mm s<sup>-1</sup>, equivalent to an initial strain rate ( $\dot{\varepsilon}$ ) of  $10^{-2} \text{ s}^{-1}$ . Work of fracture values,  $U_{\rm T}$ , were determined by computing the area under the  $\sigma$  vs  $\varepsilon$  curve. Additionally, another set was performed to determinate the apparent stress exponent,  $n_{\rm ap}$ , and the apparent activation energy,  $Q_{\rm ap}$ , using strain-rate-change (SRC) tests from  $10^{-1}$  to  $10^{-5}$  s<sup>-1</sup>. Limited ductility and the presence of porosity in the as-cast material made it difficult to perform SRC tests in this material, so a complementary set of tensile tests at constant strain rate of  $10^{-4} \, s^{-1}$  was performed to determine the values of  $n_{\rm ap}$  and  $Q_{\rm ap}$ .

#### 3. Results

## 3.1. Microstructure

Fig. 2a and b shows optical micrographs corresponding to the as-cast, hypoeutectic Al–7 wt% Si alloy. This microstructure consists on primary Al matrix dendrites surrounded by the eutectic constituent. The primary spherical-shaped Al dendrite cells are about  $60-100 \ \mu m$  in size. Fig. 2b is a higher magnification image of eutectic constituent showing a distribution of irregularly shaped Si particles, about 5  $\mu m$  in size (major axis).

It should be noted that a metastable super-saturated solid solution ( $\sim$ 1.6 wt% Si) is retained in the aluminum matrix due to the relatively fast cooling rate following casting [17,18]. This supersaturated solid solution will help in the grain refining process during subsequent severe plastic deformation. The effects on the microstructure in the flow plane (FP) after one and eight ECAP passes by route A are shown in Fig. 3. Fig. 3a and b are optical micrographs showing the distribution of the primary Al matrix and eutectic constituents of the one ECAP pass (1p) and eight passes (8p) materials, respectively. After one pass the primary and eutectic constituents are elongated and inclined with respect to the axis of the die exit channel. Eight ECAP passes lead to a further elongation of both constituents and a lower inclination, reducing the separation between any two adjacent eutectic areas. A more detailed description can be found elsewhere [10,19]. SEM micrographs in Fig. 3c and d show eutectic Si particles after one and eight passes, respectively. The shear stress imposed on the plane defined by the intersection of the two channels into the ECAP die disrupts the 3-dimensional Si particles morphology in the eutectic structure. These particles are progressively refined and become smaller and more equiaxed with increasing number of passes (Fig. 3c and d) [18-20]. Fig. 3e and f present the (sub)grain structure in the Al primary matrix after one and eight passes,

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