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Effect of eutectic particles on the grain size control and the superplasticity of aluminium alloys

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ABSTRACT

Aluminium alloys containing eutectic particles of the Al–Ni, Al–Mg–Si, Al–Ni–Ce and Al–Cu–Ce systems are investigated. The particles which control grain growth and stimulate grain nucleation are studied. The Zener–Smith law about dependence between grain size and particle parameters is confirmed and experimental coefficients are found. Experimental coefficients of the Zener–Smith equation obtained in this study depend on the particle size and differ from theoretical coefficients proposed by Zener and Smith. Some alloys with grain size about 3 μm demonstrate very good superplasticity indicators, namely: the strain rate sensitivity index m = 0.5–0.6 and the elongation over 400% at constant strain rate $5 \times 10^{-3}\ s^{-1}$.

1. Introduction

Superplasticity is a property through which alloys exhibit very large elongations before necking and fracture during tensile test with very high strain rate sensitivity. This phenomenon is used for the manufacturing of products using superplastic forming. The technology reduces the number of seams in the final product, the weight of products and equipment costs. Superplasticity effect occurs in alloys with grain sizes less than 10 μm, and at particular temperature and stain rate [1–3]. Grain sizes less than 10 μm can be achieved in aluminium by optimizing the structure heterogeneity; this can be done using the hard particle phases of various origins [4,5].

Numerous studies have been conducted on the development of thermo-mechanical processes and severe plastic deformation for the grain refinement of aluminium alloys by recrystallization [6–18]. The grain structure evolution during recrystallization has been investigated extensively in the past. Factors affecting grain structure are complicated as deformation pre-history, annealing temperatures and heating rates and particle structures must be taken into account [19–21]. Many papers focussed on the influence of non-deformable oxides, carbides and other ceramic and non-metallic particles on recrystallization kinetics [22–26]. The effect of particles is two-fold. On the one hand, finely dispersed particles (<0.1 μm) inhibit recrystallization, while on the other, large particles (>1 μm) promote recrystallization through particle stimulated nucleation (PSN) [19–21,29–37]. These studies reveal that new grains tend to nucleate at large particles (PSN). By restricting subgrain boundary migration, fine particles effectively inhibit the nucleation of new grains (Zener pinning). The effect of eutectic particles on the size of recrystallized grains has not been thoroughly studied. However, it is very appealing to use eutectic particles for grain size control. For example, the eutectic Al–Ca, Al–Ca–Zn, Al–Ca–Si alloys have a grain size of about 0.5–3 μm and very high superplasticity [27,28]. Both fine and coarse particles can be obtained in eutectic alloys and both can influence the grain structure. This study investigates the influence of the eutectic particle parameters on the nucleation of new grains and grain boundary pinning. The purpose of this work is to study the influence of size and volume fraction of eutectic particles (such as Al_3Ni, Al_4Ce, Mg_2Si, Al_8CeCu_4) in aluminium based alloys in order to determine their ability to produce fine grains and to contribute to better superplastic properties.

2. Experimental procedure

Our study focused on the aluminium rich corner of Al-based Al–Ni, Al–Mg–Si, Al–Cu–Ce and Al–Ni–Ce [38,39] alloys. These alloys contain the Al_3Ni, Mg_2Si, Al_8CeCu_4 and Al_4Ce eutectic phases (Table 1) and Al-based solid solution. The alloys were cast in water-cooled copper moulds. The ingots had a size of 100 mm × 40 mm × 20 mm. The cooling rate during casting

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was about 15 K s^{-1}. Casting temperatures were in the range of 750–800 °C. All ingots were homogenized at temperatures in between 450 and 620 °C in order to obtain particles of different size (Table 1). Subsequent rolling was carried out at temperature of 450 °C with thickness reduction of 83%. Then cold rolling with a deformation degree of 67% was carried out. The final sheet thickness was 1 mm.

Microstructure characterizations were carried out using JSM35-CF scanning electron microscope (SEM) and Neophot30 optical light microscope (LM). Specimens were etched in the Keller's reagent to study particle parameters or were subjected to oxidation to analyze the grain structure. The average grain size (D), particle size (d), volume fraction of particles (f) and interparticle spacing (l) were determined experimentally. Additionally the inter-particle spacing was estimated using Eq. (1) in paper [40] and the volume fraction of particles was calculated using ThermoCalc software (version TALL5):

$$l = \frac{2}{3} d \left(\frac{1}{f} - 1 \right). \quad (1)$$

The calculated and experimentally measured values of both the volume fraction and inter-particle spacing are in good agreement. Specimens for transmission electron microscopy (TEM) were sliced parallel to the rolling plane. The discs were electrochemically thinned by twin-jet polishing using Struers Tenupol and 20% perchloric acid in methanol at the temperature of 5–10 °C below zero.

The superplastic behaviour of the alloys was characterized by means of tensile tests performed at elevated temperatures (0.95T_{melt}). For the superplastic tests samples with gauge direction parallel to the rolling direction by area 14 mm × 6 mm × 1 mm were used. Elongation and flow stress values were measured in the tests at a constant strain rate, while the strain rate sensitivity index m was determined with incremental increases in the strain rate.

3. Results and discussion

The alloys were selected in hypoeutectic concentrations range for receiving various volume fractions of the particles from f_{min} to f_{max} (Table 1). These particles have different properties and may affect recrystallization differently. For example, the large Al_3Ni particles (with the highest hardness) will generate local rotations. The Al–Ni and Al–Ni–Ce alloys have a low alloyed aluminium matrix, while other Al–Mg–Si, Al–Cu–Ce alloys have higher concentration of solute atoms in aluminium matrix.

After cold rolling, the grains are elongated along the deformation axis. Analysis via TEM identified differences in the dislocation structure of the cold-rolled alloys with different particle sizes. Alloys containing more than 12 vol.% of particles with sizes ranging from 0.7 to 4.0 μm have subgrain microstructures with low densities of free dislocations (Fig. 1a). Cellular dislocation structures with larger amounts of dislocations are formed in the alloys which contain 3–8 vol.% of particles of the same size. Similar structures were observed in alloys with particle size of 0.3 μm (Fig. 1b).

Recrystallization was complete after 20 min of annealing at the temperature of 0.95T_{melt} (Fig. 2a). However, non-recrystallized zones were observed in the Al–Ni alloy which had 10 vol.% of particles with size 0.3 μm (Fig. 2 b).

The grain size D varies linearly with the d/f ratio as (Fig. 3, Eq. (2)),

$$D = k \left(\frac{d}{f} \right) + b. \quad (2)$$

Experimentally obtained values of the coefficient k in Eq. (2) depend on the particle size. Values of the coefficient k higher than

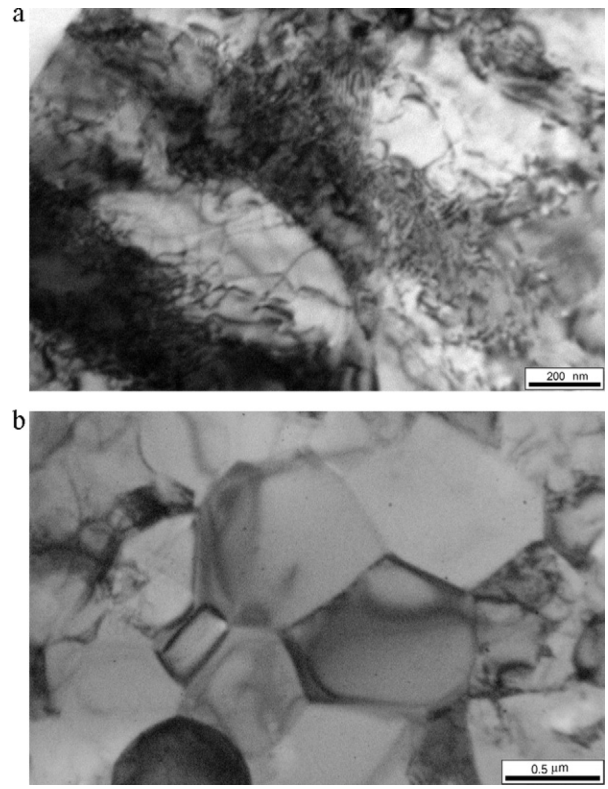

Fig. 1. TEM micrograph of the alloys containing different sizes of Mg_2Si particles: (a) f = 8 vol.%, d = 1.5 μm; (b) f = 15 vol.%, d = 1.5 μm.

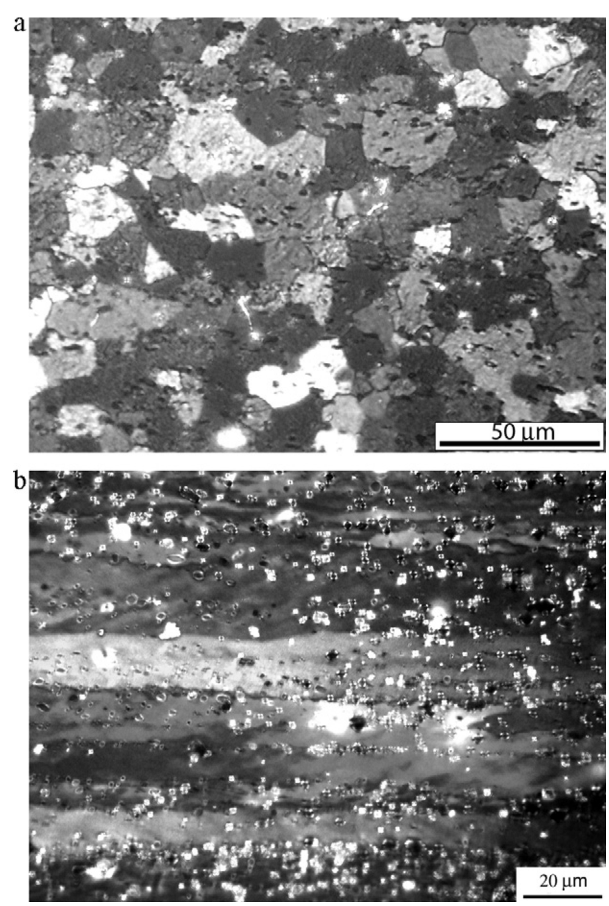

Fig. 2. The structure after 20 min annealing at the 0.95T_{melt} of the Al–6Ni alloy with different size of Al_3Ni particles: (a) d = 1.2 μm and (b) d = 0.3 μm.