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Superplastic behaviour of Al-Mg-Zn-Zr-Sc-based alloys at high strain rates

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ABSTRACT

Superplastic deformation behaviour of two aluminium-based AA7XXX type alloys with Sc and Zr additives distinguished by the presence and absence of coarse eutectic Al_9FeNi particles are compared. An alloy with Al_9FeNi particles exhibits high strain rate superplasticity at constant strain rate range of 5×10^{-3}–$8\times10^{-2}\,s^{-1}$ with elongation of 915% due to extensive dynamic recrystallisation. Effective activation energy of superplastic deformation, surface relief of deformed samples, and grain structure evolution are analysed. Particle-depleted zones are found in the alloy with Al_9FeNi particles at strains higher than 1.25.

1. Introduction

Among various aluminium alloys, AA7XXX series of alloys Al–Zn–Mg base system with Sc and Zr additions possess superior strength [1] and high formability in the conjunction with superplasticity [2–5]. Such alloys can be used to produce a variety of superplastically formed (SPF) parts for airspace industries. The strain rates at which superplasticity occurs are low $(1–5)\times10^{-4}\,s^{-1}$ for thermo-mechanically proceeded sheets of AA7XXX alloys without Sc and Zr and the strain rate is significantly higher of $1\times10^{-2}\,s^{-1}$ for alloys with Sc and Zr. Either Zr or Sc, or a combination of both Zr and Sc in aluminium alloys is attributed to high density of nanoscale-coherent [6,7], $L1_2$-ordered particles that control grain growth via the Zener pinning mechanism and increase the recrystallisation resistance [8,9]. The microstructure of these materials ('Supral' type [10–12]) at the start of superplastic deformation consists of bands (*e*longated grains) of similarly oriented subgrains and static recrystallisation does not occur or only partially occurs during heating to superplastic deformation temperature. Many studies [5,13–16] indicated that coarsening resistance of $Al_3(Sc,Zr)$ ($L1_2$) precipitates is higher compared to Al_3Sc ($L1_2$). As a result, $Al_3(Sc, Zr)$ particles are good grain growth inhibitor and they ensure strong recrystallisation resistance.

Numerous studies discuss a possible dominant mechanism of superplastic deformation in dynamically recrystallised alloys with "banded" grain structure [17] before the start of the superplastic deformation, but experimental study yields inconsistent results. Liu and Chakrabarti [18], Cao [19] and Duan [20] concluded that the grain boundary sliding is a predominant mechanism of superplastic deformation of Al–Zn–Mg–Sc(Zr) type alloys. Bate [21] considered that an intragranular dislocation slip could be the principal mechanism in the superplastic deformation of Al–6Cu–0.4Zr alloy. Sotoudeh and Bate [17] and Bricknell [1] established that superplasticity of 'Supral type' alloys with "banded" microstructures is primarily a result of diffusion creep. del Valle [22] founded that the symbiosis of GBS and slip creep allows having superplastic properties at high-strain rate in Supral 2004 and Al–Li 8090. Authors [22] believed that continuous dynamic recrystallisation have an important contribution during superplastic deformation of these alloys. Katsas [12] found that superplastic deformation of Al–1Zr alloy is a result of dynamic recrystallisation process. It means that dislocation slip/creep should be an important mechanism of superplastic deformation such alloys. Recently, a new high strength superplastic alloy of Al–Zn-Mg based system, containing coarse Al_3Ni particles was developed [23]. The alloy exhibits mainly non-recrystallised grain structure before the start of the superplastic deformation and very high stable and fine grains due to dynamic recrystallisation process during superplastic deformation. As a result, sheets of the alloy after simple thermomechanical processing exhibit high strain rate superplasticity at strain rate up to $10^{-1}\,s^{-1}$.

In current study, two alloys of Al–Zn–Mg based system in the

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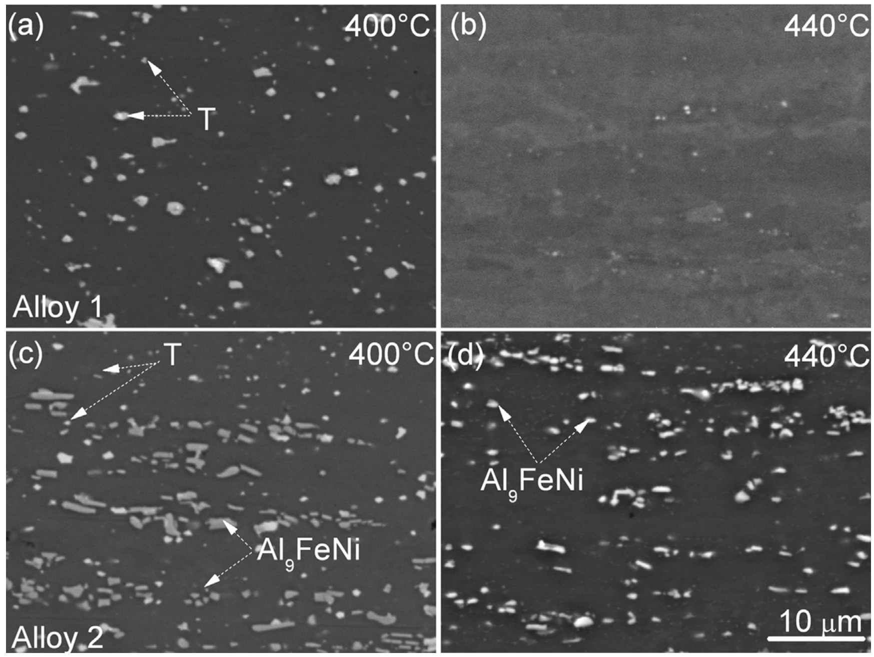

Fig. 1. SEM microstructures of alloy 1 (a, b) and alloy 2 (c, d) after 20 min of annealing at 400 °C (a, c) and 440 °C (b,d).

presence of low 0.1% Sc (wt%) and 0.2%Zr (wt%) were investigated. One alloy contains Al_9FeNi eutectic particles and the other does not contain it. The purposes of this study are: (i) to compare the superplastic behaviour of the Al–Zn–Mg alloys with and without equilibrium eutectic particles at superplastic deformation at various temperatures and strain rates and the role of the coarse eutectic particles at superplastic deformation behaviour; (ii) through evolution of microstructure and surface relief during superplastic deformation, and the effective activation energy to clarify its dominant mechanism of superplastic deformation.

2. Experimental

2.1. Materials and processing

Two Al–Zn–Mg–Zr–Sc alloys with similar concentrations of Zn, Mg, Sc (0.1%), and Zr (0.2%) were studied. Alloys are distinguished by Fe and Ni containing: there is no Fe and Ni in the alloy 1, and the alloy 2 with Fe and Ni contains equilibrium eutectic origin phase Al_9FeNi after solidification [24–31].

Al (99.99%), Mg (99.92%), Zn (99.92%), and Al–20%Ni, Al–5%Zr, Al–5%*Sc* master alloys were used for alloys making. The alloys were melted in a Nabertherm S3 electric furnace with air atmosphere using graphite-fireclay crucibles. The ingots were cast in a water-cooled copper mould with a size of $100 \times 40 \times 20$ mm^3 and casting cooling rate of approximately 15 K/s. Homogenisation annealing in two steps (at 380 ± 2 °C and at 480 ± 2 °C) was applied and performed in “Nabertherm N 30/65 A” furnace. First low-temperature step at 380 °C is required for the formation of high density of fine Al_3(Zr,*Sc*) precipitates and for the dissolving of non-equilibrium eutectic phase T($Al_2Mg_3Zn_3$). Fragmentation and spheroidisation of the Al_9FeNi eutectic particles is the main process at 480 °C. The sheets were produced by simple thermomechanical processing using hot rolling at 400 ± 20 °C and subsequent rolling at room temperature with the reduction of 60%. Final thickness of the sheets was 1.00 mm.

2.2. Microstructure analysis

Polarized light of the optical light microscope (OM) (Axiovert 200MMAT “Carl Zeiss”) was used for the grain structure analysis. Microstructure was analysed in the thickness section parallel to the rolling/stress direction. The samples were prepared by mechanical grinding, polishing, electro polishing in chlorine–alcohol electrolyte at 20 V, and anode oxidizing in water solution of 10% (HF in H_3BO_4).

A Tescan–VEGA3 LMH scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (*e*DS) (X–MAX80) and EBSD–HKL NordlysMax EBSD detector of Oxford Instruments production was used for the microstructural investigations and the phase composition analysis, respectively. The parameters of EBSD maps were: a scan size of 375×375 μm^2 and a scan step of 0.3 μm. The samples for EBSD analysis were sliced parallel to the rolling plane or to the stress direction and were prepared by mechanical grinding, polishing, and electro polishing in nitric acid- methanol electrolyte at 15 V and temperature of (-25)°C.

Thin foils 3 mm in diameter and major axis parallel to the stress direction were used the for TEM analysis in JEOL JEM–2000 EX. The samples were prepared by mechanical grinding and electro polishing in A2 electrolyte (Struers produced) at 21 V and at temperature of (0 ± 2)°C.

The average grain size and particle parameters (size and volume fraction) were determined by the random secant method using more than 300 measurements. Error bars were calculated using an experimental standard deviation and a confidence probability of 95%.

2.3. Superplastic indicators

The superplastic behaviour was characterized by means of uniaxial tensile tests on a Walter Bay LFM-100 test machine with a program service Dion-Pro for the control of the traverse motion in real time. Tests at the constant strain rates were carried out at strain rate range of 5×10^{-3}–8×10^{-2} s^{-1} and a temperature range of 400–500 °C with step of 20 °C for the analysis of superplastic behaviour. The accuracy of the constant strain rate was not