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## Materials Science and Engineering A

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# High-temperature tensile behavior and microstructural evolution of cold-rolled Al-6Mg-0.4Sc-0.13Zr alloy

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#### ARTICLE INFO

#### Article history: Received 8 January 2008 Received in revised form 15 May 2008 Accepted 14 August 2008

Keywords: Al-Mg-Sc-Zr alloy Al<sub>3</sub>(Sc<sub>x</sub>Zr<sub>1-x</sub>) Grain boundary sliding

#### ABSTRACT

The high-temperature tensile properties and microstructural evolution of Al–6Mg–0.4Sc–0.13Zr alloy prepared by conventional cold rolling at various temperatures were studied. At 573 K, the Al $_3$ (Sc $_x$ Zr $_{1-x}$ ) particles stabilized the extremely fine subgrains (SG) and submicrograins (SMG). These SG and SMG boundaries acted as effective dislocation sinks, turning into true high-angled boundaries susceptible to consequent sliding. Therefore, the alloy achieved an acceptable ductility with 300% elongation. At 623–723 K, because geometrical sliding was not feasible with rugged grain boundaries and dislocation movement was strongly hindered by the Al $_3$ (Sc $_x$ Zr $_{1-x}$ ) particles, the alloy exhibited poor ductility. At 773 and 803 K, the Al $_3$ (Sc $_x$ Zr $_{1-x}$ ) particles began to act as grain stabilizers, and retained fine-grain structure at these high temperatures. As a result, a maximum elongation of 1100% was achieved after deformation at 803 K with a strain rate of 5 × 10<sup>-3</sup> s<sup>-1</sup>.

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#### 1. Introduction

The recent years have seen increased attention devoted to energy conservation to meet the environmental concerns. One of the most effective measures is to design lightweight vehicles by using superplastic forming and thereby raise the average fuel efficiency. Superplastic forming requires small and stable grains usually less than 10 µm in diameter [1]. However, superplasticity in a traditional fine-grained Al-Mg alloy is limited due to the significant grain growth during superplastic deformation [2,3]. Therefore, it is important to find an effective grain stabilizing agent for using superplastic Al-Mg alloys. Adding Sc as an alloying element was thought to be the most potent means to improve the properties of Al-Mg alloys. Moreover, the effects of scandium were found to be greatly amplified by Zr [4,5]. For instance, the simultaneous addition of Sc and Zr has a more pronounced effect in grain refinement, leading to higher strength with higher ductility [4]. Furthermore, Lee et al. [5] found that the Al-3Mg-0.2Sc-0.12Zr alloy exhibited superior superplasticity than Al-3Mg-0.2Sc and Al-3Mg-0.2Zr did at high temperature of 773 K. It is due to the grain structure of the Al-3Mg-0.2Sc-0.12Zr alloy prepared by equal channel angular extrusion (ECAE) was more stable than those of the ECAEed Al–3Mg–0.2Sc and Al–3Mg–0.2Zr alloys at 773 K in their study [5]. Besides, Geng et al. [6] also found that for Al–3.3Mg–0.2Sc–0.2Zr alloy, the ultra-fine microstructure afforded by ECAE can remain very fine ( $\sim\!1~\mu m$ ) even after static annealing at 773 K for 1 h. All of the above results were due to the extremely fine and coherent Al<sub>3</sub>(Sc<sub>x</sub>Zr<sub>1-x</sub>) particles, which are more thermal stable than Al<sub>3</sub>Sc or Al<sub>3</sub>Zr particles at elevated temperatures [5,7].

Among the novel Al–Mg–Sc(–Zr) alloys, the 01570 alloy (Al–5.8Mg–0.4Mn–0.25Sc–0.lZr) – containing about 6% Mg with additions of Mn, Sc, Zr, and other elements – has found the widest application due to its high strength [8]. In a study on a cold-rolled Al–6Mg–0.3Sc alloy [9], the alloy achieved its maximum elongation of  $\sim\!1130\%$  at a strain rate of  $1.4\times10^{-2}\,\mathrm{s}^{-1}$  at 793 K. Thus, we are also interested in the superplastic behaviors of cold-rolled Al–6Mg alloys with high proportions of both Sc and Zr.

While the superplastic behavior of Al–Mg–Sc alloys had been widely studied [9–14], there is still a dearth of information on the effects of Sc and Zr on the thermal stability and superplastic behavior of cold-rolled Al–Mg alloys. Besides, most of the available research has focused on the benefit of the impediment on grain growth caused by these fine and coherent Al<sub>3</sub>Sc or Al<sub>3</sub>(Sc<sub>x</sub>Zr<sub>1–x</sub>) particles. In this work, we propose a more comprehensive viewpoint on the interaction between Al<sub>3</sub>(Sc<sub>x</sub>Zr<sub>1–x</sub>) particles and the microstructure at various temperatures.

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Table 1
The chemical composition of the studied Al alloy (in wt%)

Fe	Si	Mg	Mn	Cu	Ti	Cr	V	Sc	Zr	Al
0.08	0.19	6	0.81	0.11	0.18	0.15	0.08	0.4	0.13	Bal.

#### 2. Experimental procedure

The alloy used in this study was provided by Chung-Shan Institute of Science and Technology (Taiwan). The raw material was produced by direct chill casting followed by homogenization at 813 K for 12 h then under extrusion at 693 K. The alloy was received in an extruded form with a thickness of 15 mm and its chemical composition is listed in Table 1. This alloy was treated by two-step mechanical processing. The procedure in our work was as follow: First, the alloy was hot-rolled at 723 K to a thickness of 8 mm ( $\sim\!47\%$  rolling reduction) followed by water quenching at room temperature. Second, cold-rolled for 40 passes to a final thickness of 2 mm which was with a reduction of 75%. The cold-rolled sheet were then cut for isothermal annealing and machined for high-temperature tensile tests.

Isothermal annealing was carried out at temperature between 373 and 773 K for 30 min in an air furnace and the hardness curve of the annealed samples was measured.

Tensile specimens with a gauge length of 10 mm and cross-section of 6 mm  $\times$  2 mm were machined from the rolled sheets and the loading axis was parallel to the rolling direction. Tensile tests were performed at temperature between 523 and 803 K with the initial strain rates ranging from  $5 \times 10^{-4}$  to  $1 \times 10^{-1}$  s<sup>-1</sup>. To avoid the liquid formation, the highest working temperature 803 K was picked according to the DTA experiment that the alloy began to occur partial dissolution at temperature above 831 K. All high-temperature tensile tests were performed on a Hung-Ta HT8150 materials test system with a five-zone furnace operating at a constant crosshead displacement rate. The testing temperature was controlled within  $\pm 3$  K. The value of the strain rate sensitivity, m, was also determined according to the following equation:

$$m = \frac{\partial \log \sigma}{\partial \log \dot{\varepsilon}}$$

Transmission electron microscope (TEM) was also used to examine the microstructure after static annealing and high-temperature tensile tests. The samples chose for TEM observations were prepared by mechanical grinding to a thickness of about 10  $\mu m$  and then further thinning to a thickness of electron transparency was carried out using double-jet electropolishing in a 25% nitric acid and 75% methanol solution operated at around 253 K and 20 V. Samples were examined on a JOEL-100CXII microscope operating at 100 kV.

#### 3. Results

#### 3.1. Static annealing

Fig. 1 shows the TEM micrograph of the Al–6Mg–0.4Sc–0.13Zr alloy after cold rolling. The alloy shows a typical dramatically deformed grain microstructure with elongated grains parallel to the rolling direction and a considerable number of introduced dislocations [12]. The microhardness curve of the cold-rolled Al–6Mg–0.4Sc–0.13Zr alloy after static annealing for 30 min at different temperatures is illustrated in Fig. 2: a conventional Al–Mg alloy annealing curve is presented in which recovery occurs at lower temperatures (<523 K) and recrystallization occurs at temperature above 573 K [11]. Recrystallization was not inhibited in our study, unlike in the study of the Al–4.2Mg–0.2Sc alloy by Park et al. [11]. This difference may be caused by their homogenization step, 683 K

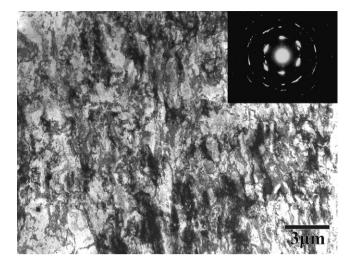
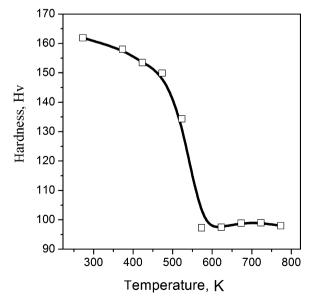


Fig. 1. The TEM micrograph of the cold-rolled Al-6Mg-0.4Sc-0.13Zr alloy.

for 24 h, which may have been suitable for the primary Al<sub>3</sub>Sc particle to precipitate sufficiently [15] and then seriously impede the consequent softening process.

of Fig. 3(a)-(g)shows the grain structure Al-6Mg-0.4Sc-0.13Zr alloy after annealing for 0.5 h at various temperatures. When the alloy was annealed at 473 K (Fig. 3(a)), dislocations were partially recovered and no distinct grains were formed. And on annealing at 523 K, as present in Fig. 3(b), partial recrystallization started to emerge and some newly formed submicrograins (SMG,  $\sim$ 0.3  $\mu$ m) were found. Fig. 3(c) shows the microstructure of the Al-6Mg-0.4Sc-0.13Zr alloy after annealing at 573 K for 0.5 h. Here, the grain structure was extremely inhomogeneous: a small number of huge grains (>10 µm) surrounded fine-grain bands similar to the duplex structure in the ECAEed Al-3Mg-0.2Sc-0.12Zr alloy after static annealing at 673 K for 1 h [5]. Closer observation of these fine-grain bands revealed that subgrain boundaries (SGBs) divided small micrograins (MGs) into subgrains (SGs), and the existence of the  $Al_3(Sc_xZr_{1-x})$  particles at the triple point of the SGBs was also noted [9]. These Al<sub>3</sub>( $Sc_xZr_{1-x}$ ) particles were coherent with the matrix, spherical in shape, and



**Fig. 2.** The vickers hardness curve of the cold-rolled Al-6Mg-0.4Sc-0.13Zr alloy after static annealing for 30 min at various temperatures.

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