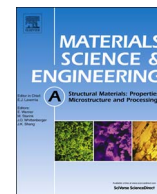




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Effects of Sb micro-alloying on precipitate evolution and mechanical properties of a dilute Al-Sc-Zr alloy

Jeffrey D. Lin^{a,*}, Philipp Okle^{a,1}, David C. Dunand^a, David N. Seidman^{a,b}^a Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108, USA^b Northwestern University Center for Atom-Probe Tomography, Northwestern University, 2220 Campus Drive, Evanston, IL 60208-3108, USA

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ABSTRACT

An Al-Sc-Zr aluminum alloy with Sb micro-addition (Al-0.066Sc-0.050Zr-0.021Sb at%) is cast and heat-treated to study the effects of Sb on the nucleation, growth, coarsening kinetics and precipitate morphology, and resulting mechanical properties at ambient and elevated temperatures. When isochronally aged, the Sb-containing alloy exhibits a peak microhardness (607 ± 12 MPa) at 475 °C, which is greater than that of a comparable Sb-free alloy at the same temperature (549 ± 17 MPa), and a smaller rate of decrease of microhardness values due to precipitate coarsening for aging temperatures > 475 °C. When isothermally aged, the Sb-containing alloy achieves larger peak microhardness values at 300 °C for more than a month (~80 MPa difference) and 400 °C for ~8 h (~200 MPa difference) than the Sb-free alloy. Atom-probe tomography of the peak-aged Sb-containing alloy demonstrates that Sb partitions to the precipitates, and is enriched in the Zr-rich shell (up to 0.35 at% Sb). For creep testing at 300 °C, the Sb-containing alloy exhibits smaller steady-state strain-rates than the Sb-free control alloy at applied stresses > 15 MPa. It is hypothesized that the effects of Sb micro-alloying (partitioning to precipitates, enhanced precipitate coarsening and higher creep resistance) are linked with the following mechanisms: (i) enhanced Zr diffusion in the matrix due to attractive Sb-Zr interactions; (ii) reduction in matrix/precipitate interfacial free energy, when Sb is present; and (iii) an increase in precipitate/matrix lattice parameter mismatch resulting in stronger elastic interactions with dislocations.

1. Introduction

Precipitation-strengthened aluminum alloys microalloyed with scandium and zirconium have been developed as a promising family of lightweight alloys for use at temperatures as high as 400 °C, with applications in aerospace and automotive structural components [1–10]. Current, traditional age-hardened aluminum alloys cannot be used at high temperatures for long times since the precipitate coarsen rapidly above ~230 °C, thereby weakening the alloy [11–16]. Scandium and zirconium form nanoscale trialuminide Al₃X (X=Sc or Zr) precipitates with the L1₂ structure, which are coherent with the face-centered cubic (f.c.c.) crystal structure of aluminum, and displays slow coarsening rates due to the diffusion of Sc and Zr in Al [1,17–30]. Scandium has the greatest ability to strengthen aluminum on a per atom basis by grain refinement and formation of ordered (L1₂-structure) nanoscale precipitates that are stable after prolonged aging at 300 °C [17,18,22–25,27,31–34]. Zirconium forms metastable L1₂ precipitates, which are coarsening-resistant at temperatures below 500 °C, before transforming to the D0₂₃ tetragonal structure [19,21,29,35–38].

Combining Sc and Zr in aluminum yields an alloy with L1₂ Al₃(Sc, Zr) precipitates, displaying superior heat-resistance and high strength [33,39–46]. Precipitates with a core-shell morphology are formed in these alloys during aging, where the core is Sc-rich and the shell is Zr-rich due to Scandium's higher diffusivity, and thus its earlier precipitation in aluminum [3,10,39,40,47–51]. The Zr-rich shell provides a smaller lattice parameter mismatch with the matrix, a diffusion barrier against Sc atoms, resulting in coherent Al₃(Sc, Zr) precipitates with small coarsening rates at 400 °C for up to 2 mos. Recently, additional elements, such as erbium and silico, have been added to Al-Sc-Zr alloys and studied for their effects on precipitate morphology, nucleation and growth rates, coarsening resistance, and strengthening at ambient and elevated temperatures [47,52–54]. Erbium improves the creep resistance by the formation of a higher mismatching Er-rich inner core within the precipitates, Si accelerates the nucleation, growth and coarsening of the precipitates, and improves the peak strength achieved during thermal aging due to an attractive binding free energy between Si atoms and vacancies [47,52–55]. Booth-Morrison et al. [53] attribute the effects on the early stages of nucleation and growth of Sc-rich precipitate cores to: (i) the favorable formation of Sc-Si and Si-

* Corresponding author.

¹ Current address: Eidgenössische Technische Hochschule Zürich, Zurich, Switzerland.<http://dx.doi.org/10.1016/j.msea.2016.10.067>

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vacancy dimers, which decreases the migration energy of Sc in Al from 0.74 to 0.45 eV atom⁻¹ and increases the Sc diffusivity; and (ii) an attractive binding free energy between Si and vacancies leading to vacancy clusters that become heterogeneous nucleation sites for precipitates. Vo et al. [54] propose that the accelerated coarsening of the Al₃(Sc, Zr) precipitates is a result of Si atoms increasing the diffusivity of Sc within the Zr-rich regions in the precipitate, and also possibly Zr.

This article focuses on a study of an Al-Sc-Zr alloy with microalloying addition of Sb, which was selected based on Wolverton's [55] first-principle calculations of binding energies between solute atoms and vacancies in aluminum. Since Sb has a favorable 0.30 eV attractive binding energy, at 0 K, with nearest-neighbor vacancies in Al, we hypothesize that Sb will exhibit a similar effect to that of Si on accelerating the precipitation kinetics of Al₃(Sc, Zr) by binding favorably with Zr, as many intermetallic compounds form in the Sb-Zr binary phase [56]. The Al-Sb binary phase diagram indicates a very limited solid solubility of Sb in aluminum (< 0.02 at% at 627 °C), with AlSb being the only intermetallic line-compound [57–59]. Although it is possible that the solubility of Sb in Al is altered as a result of Sc and Zr additions in the alloy, there currently exist no ternary phase diagrams for the Al-Sc-Sb or Al-Zr-Sb system. Furthermore, it is likely that the effects of Sc and Zr on the solubility of Sb in Al can be neglected due to the small amounts of Sc and Zr in the alloy (< 0.1 at% for either). To disperse the Sb atoms throughout the Al solid-solution and reduce or prevent AlSb primary precipitates formation during solidification and homogenization, a very small concentration of Sb (200 ppm at%) was chosen. Herein, we compare a previously-studied Sb-free alloy to our new Sb-containing alloy with respect to aging kinetics, coarsening resistance, precipitate composition, and microhardness and creep resistance.

2. Materials and methods

2.1. Alloy preparation and heat treatment

A 200 g cylindrical ingot was cast in air for an alloy with a nominal atomic composition of Al-0.06Sc-0.06Zr-0.02Sb at% (Al-0.10Sc-0.21Zr-0.09Sb wt%) by melting pieces of 4N (99.99% purity) Al (Alcoa Inc.), Al-Sc and Al-Zr master alloys (Alcoa Inc.), and ~99.5% pure Sb (Alfa Aesar) in an alumina crucible at 800 °C. The melt was stirred vigorously before being poured into a graphite crucible placed onto an ice-cooled copper platen to encourage directional solidification, thereby preventing the formation of internal shrinkage cavities during solidification. Direct-current plasma optical-emission spectrometry (DCP-OES) was performed on ~0.50 g of the ingot by ATI Specialty Alloys and Components (Albany, OR), and the results are displayed in Table 1, alongside those measured utilizing local-electrode atom-probe (LEAP) tomography. The DCP determined concentrations for Sc, Zr and Sb are close to their nominal concentrations, and the former is used in the remainder of this article.

The cast alloy was homogenized in air at 640 °C for 3 days and then water-quenched, resulting in a supersaturated solid-solution. Samples of the ingot were cut and heat-treated sequentially from 100 to 575 °C with 25 °C/1 h steps for isochronal aging. Other samples were iso-

thermally aged at 300 and 400 °C for times ranging from 0.5 to 2 months. Double isothermal aging (300 °C for 4 h and subsequently at 400 and 425 °C for 0.5 h to 1 week) was performed to develop a heat treatment sequence for maximum alloy strength with a minimal aging time. Vickers microhardness measurements were made using a Struers Duramin 5 hardness tester with 200 g load, dwell time of 5 s, and a magnification of 40×, while electrical conductivity measurements were performed using a Foerster SigmaTest 2.069 at frequencies of 120, 240, 480, and 960 kHz. Both microhardness and conductivity measurements were utilized to follow the time and temperature evolution of the precipitates.

2.2. Creep experiments

A constant load compressive creep experiment was performed at 300 ± 1 °C on a cylindrical specimen with a 10 mm diameter and a 20 mm height. The specimen had been isochronally aged to 450 °C after homogenizing at 640 °C for 72 h with a final aging temperature well above the creep temperature, to insure no aging during the creep test. The samples were heated in a three-zone furnace, with the temperature monitored using a thermocouple placed within ~1 cm of the specimen, which were placed between two boron-nitride coated alumina platens, and subjected to uniaxial compressive loads by Ni superalloy push-rods employing dead weights. Sample strain was determined by measuring the cross-head displacement using a linear-variable displacement-transducer (LVDT), with a resolution of ~6 μm, corresponding to a strain increment of ~3×10⁻⁴. The specimens were deformed at a constant load until steady-state (secondary) creep was achieved; the load was then increased and the measurement repeated. As a result, several creep rates for sequentially increasing applied stresses were measured from a single specimen without deforming the specimen more than 13%.

2.3. Microstructural imaging

Specimens for three-dimensional (3D) local-electrode atom-probe (LEAP) tomography were prepared by cutting blanks, with a diamond-blade precision saw, to dimensions of ~0.45×0.45×10 mm³. The blanks were cut from samples aged isochronally to 450 °C utilizing 25 °C/1 h steps. They were then electropolished at room temperature by thinning the blanks at 25–20 V DC using a solution of 10% perchloric acid in acetic acid, followed by necking and final nanopit formation at 15–12 V DC with a solution of 2% perchloric acid in butoxyethanol [47,54]. Ultraviolet (UV) laser assisted APT was performed using a LEAP 4000Si-X tomograph (Cameca, Madison, WI) at a specimen temperature of 30 K, a UV laser energy of 25 pJ pulse⁻¹, and a pulse repetition rate of 500 kHz. The data were analyzed using IVAS reconstruction software (Cameca), and measurement errors were calculated using counting statistics and standard error propagation techniques.

A surface of the Al-Sc-Zr-Sb alloy specimen after homogenization at 640 °C was polished to a ~0.05 μm finish, and then etched with Keller's reagent to reveal the grains and dendritic structure. The images were recorded using a Nikon Eclipse MA200 optical microscope with bright- and dark-field modes. Additionally, the base surface of the creep specimen after aging isochronally to 450 °C was polished to a ~1 μm

Table 1

Compositions of the base and experimental alloys given in atomic percent (at%) determined by direct current plasma – optical emission spectroscopy (DCP-OES) and local electrode atom-probe (LEAP) tomography. The detection limit of each method is indicated for Si.

Nominal composition	Measured composition from DCP-OES				Measured composition from LEAP			
	Sc	Zr	Sb	Si	Sc	Zr	Sb	Si ^a
Al-0.06Sc-0.06Zr ²¹	0.067	0.052	–	< 0.005	0.069	0.026	–	< 0.002
Al-0.06Sc-0.06Zr-0.02Sb	0.066	0.050	0.021	< 0.005	0.047	0.034	< 0.002	< 0.002

^a Atomic concentration of ²⁸Si²⁺ atoms from LEAP tomography.

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