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Full length article

Effect of vanadium micro-alloying on the microstructural evolution and creep behavior of Al-Er-Sc-Zr-Si alloys

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A B S T R A C T

Al-Er-Sc-Zr-Si alloys, strengthened by $L1_2$-ordered, coherent $Al_3(Er,Sc,Zr)$ nanoscale precipitates, can be used for automotive and aerospace applications up to 400 °C. Vanadium, due to its small diffusivity in aluminum and its ability to form $L1_2$-ordered tri-aluminide precipitates, is a possible micro-alloying addition for further improving the service temperature of these alloys. Moreover, vanadium-containing $Al_3(Er,Sc,Zr,V)$ precipitates are anticipated to have a smaller lattice parameter mismatch with the matrix, thereby improving the alloy's coarsening resistance. In this study, the temporal evolution of microstructural and mechanical properties of an Al-0.005Er-0.02Sc-0.07Zr-0.06Si alloy micro-alloyed with V are investigated utilizing isochronal, isothermal and double-aging treatments and compared to the results obtained from an alloy that does not contain V, but otherwise has the same composition. Both isochronal and isothermal aging treatments reveal slower precipitation and coarsening kinetics for the V-containing alloy. A peak microhardness value of ~600 MPa is obtained after a double-aging treatment at 350 °C/16 h, followed by aging at 400 °C for 12 h. Transmission electron microscopy reveals a duplex-size precipitate microstructure, with the smaller precipitates having a mean radius <3 nm. Despite the expectation of a reduced creep resistance due to a lower precipitate/matrix lattice mismatch, both alloys have similar creep behavior at 400 °C, characterized by a threshold stress of 7.5 and 8 MPa under peak-aged and over-aged conditions, respectively. Thus, micro-additions of V to an Al-Er-Sc-Zr-Si alloy lead to enrichment of V in the $Al_3(Er,Sc,Zr,V)$ nano-precipitates, improving their coarsening resistance without deteriorating their ability to block dislocations under creep at 400 °C.

1. Introduction

Castable, heat-treatable aluminum alloys are utilized widely in a number of applications; including automotive, aerospace, and power transmission, due to a combination of desired properties, such as low density, high specific strength, good oxidation resistance, high electrical conductivity, and relatively low cost. Their strength and creep resistance is, however, low at temperatures above ~250 °C, due to precipitate dissolution and/or coarsening, which limits the utilization of these alloys, in as-cast or wrought conditions, for high-temperature applications.

Al-Sc alloys, strengthened with $L1_2$-ordered Al_3Sc precipitates, provide a promising alternative to overcome this problem [1–11]. These alloys can be utilized at service temperatures up to 300 °C; however, due to the limited availability of Sc they are rather expensive. There has been extensive research focused on identifying other alloying elements that can further increase the service temperature and replace some of the Sc content, thereby rendering the alloys less expensive. The main requirement for any of these potential substitute alloying elements is that they form $L1_2$-ordered tri-aluminide precipitates. Rare earth (RE) elements, such as Er, Tm, Lu and Yb, are known to replace some of the Sc in the $Al_3(Sc,RE)$

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phase [11–14] and research has demonstrated that Er is the most effective and the least expensive RE [11,12]. However, due to the higher diffusivity of Er in Al compared to Sc, it commences to precipitate at lower temperatures (ca. 250–275 °C), which does not improve the service temperature. Also due to the difference in the diffusivities of Er and Sc they form a core/shell precipitate structure, where Al_3Er forms a core and $Al_3(Sc,Er)$ forms a shell [11]. As a result, the high temperature creep resistance of Al-Er-Sc alloys improves significantly compared to binary Al-Sc alloys, due to a larger lattice parameter mismatch between the precipitate shell and the Al matrix [9,15].

On the other hand, some of the transition metal elements, Zr, Ti, or Hf, substitute for Sc in $L1_2$-ordered-precipitates [16]. Specifically, Zr is known to significantly improve the coarsening resistance, which is attributed to its low diffusivity. In Al-Er-Sc-Zr alloys, Zr promotes the formation of the final shell of core/shell/shell precipitates, which serves as an effective diffusion barrier for coarsening at elevated temperatures [4,7]. These alloys can be used at service temperatures as high as 400 °C [7].

Furthermore, Si is one of the main impurities in Al and microalloying with it in these alloys offer several benefits and can enable the use of less expensive commercial-purity aluminum (99.9%). It can increase the precipitate number density by promoting heterogeneous nucleation [8]. Additionally, Si increases the precipitation kinetics, thereby; reducing the aging time required to achieve peak microhardness [8].

The objective of this research is to investigate the effects of V addition on the coarsening- and creep-resistant properties of Al-Er-Sc-Zr-V-Si alloys. Vanadium forms metastable, $L1_2$-ordered Al_3V precipitates and has a diffusivity in Al that is even lower than that of Zr [17]. Therefore, in this context, V has the potential to either create another precipitate shell or modify the Zr-rich shell by forming $Al_3(Zr,V)$. Vanadium is expected to decrease lattice parameter misfit between the precipitates and the Al matrix; hence, its effect on creep resistance must be carefully studied [18]. On the other hand, V is expected to reduce the solubility of Zr in Al, and this may cause the formation of primary precipitates during solidification, which may result in a small grain size, creating an undesirable microstructure for high-temperature creep resistance. We performed isochronal, isothermal, and double aging studies to understand the microstructural evolution and mechanical properties of an Al-Er-Sc-Zr-V-Si alloy, while using an Al-Er-Sc-Zr-Si control alloy to focus on the effects of V additions. Microstructural evolution was studied at four different length scales, utilizing optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atom-probe tomography (APT). Mechanical properties were studied utilizing Vickers microhardness measurements at room temperature and compression creep experiments at 400 °C. Electrical properties, in particular the electrical conductivity, were also measured at room temperature to evaluate the microstructural evolution as a function of aging time and temperature.

2. Experimental procedures

2.1. Casting and aging treatments

Two alloys with nominal compositions of Al–0.005Er–0.02Sc–0.07Zr–xV–0.06Si at.% (Al–0.031Er–0.033Sc–0.236Zr–xV–0.062Si wt%) were fabricated: a control alloy, Q1, was V-free (x = 0) and an experimental alloy, Q2, was V-bearing (x = 0.08 at.% or 0.15 wt%). Both alloys were prepared from 99.99 at.% pure Al, and master alloys consisting of Al–5.9 wt% Er, Al–2 wt% Sc, Al–8 wt% Zr, Al–5 wt% V, and Al–12.6 wt% Si. Pieces from the above metals and alloys were melted in alumina crucibles at 800 °C in a resistively heated muffle furnace and stirred five times using alumina rods with a 15 min hold time between each stir. Subsequently, alloys were cast in graphite molds that were preheated to 200 °C and then placed on an ice-cooled copper platen to promote directional solidification. Upon full solidification, which approximately takes 20–30 s, the alloy ingots were quenched into an iced water bath.

Then a homogenization treatment was performed at 640 °C for 4 h, followed by one of the following three aging treatments: (i) isochronal aging treatments over the temperature range of 200–575 °C with 25 °C increments and a 3 h holding time at each temperature; (ii) isothermal aging treatments at 400 and 425 °C for times up to 264 h; and (iii) double-aging treatments, designed to find the peak microhardness conditions, at a primary aging temperature of 300 or 350 °C for 16 h and a secondary aging temperature of 400, 425, or 450 °C for times up to 200 h. All heat treatments were conducted in air and terminated by water quenching.

2.2. Microstructural characterization

The chemical compositions of the as cast ingots were measured by direct-current plasma atomic-emission spectroscopy (DCP-AES) at ATI Wah Chang (Albany, OR) using two samples taken from the top and the bottom of the ingots and also studied by APT [19,20]. All specimens for OM and SEM investigations were prepared using standard metallographic techniques. To reveal the grain structure, select specimens were dip-etched with Poulton's reagent (60 vol% hydrochloric acid + 30 vol% nitric acid + 5 vol% hydrofluoric acid + 5 vol% water) for 30 s. Electrical conductivity (EC) measurements were performed at 120, 240, 480, and 960 kHz (five measurements per frequency) using a Sigmatest 2.069 eddy current instrument (Foerster Instruments, Pittsburgh, PA).

TEM specimens were prepared by cutting ~1.5 mm thick samples with a diamond saw, which were then mechanically ground with SiC papers to a thickness of ~70 μm. Standard 3 mm TEM discs were mechanically punched from the thin foils and twin jet electropolished using a 30 vol% nitric acid and 70 vol% methanol electrolyte solution at −10 °C. A 10 Vdc potential was utilized, which resulted in a current of 70–90 mA. After electropolishing, the specimens were cleaned with methanol. A JEOL JEM-2010 high resolution TEM and a FEI Tecnai G2-F20 ST scanning transmission electron microscope (STEM) were used for sample analysis. Diffraction spots were confirmed with CaRIne Crystallography (CaRIne Crystallography, Senlis, France) and JEMS simulation software (Interdisciplinary Center for Electron Microscopy, Swiss Federal Institute of Technology Lausanne).

APT specimens were prepared by cutting blanks with a diamond saw to ~$0.5 \times 0.5 \times 10$ mm^3 dimensions and subsequently electropolished in two stages: (i) coarse electropolishing at 20–25 Vdc using a solution of 10 vol% perchloric acid in acetic acid to form a neck; and (ii) fine polishing at 15–18 Vdc using a solution of 2 vol% perchloric acid in butoxyethanol to dissolve the neck and obtain a tip. Picosecond pulsed ultraviolet (wavelength = 355 nm) laser assisted APT was performed using a LEAP4000X-Si tomograph (Cameca, Madison, WI) at a pulse repetition rate of 500 kHz, a pulse energy of 50 pJ, and a sample temperature of −243 °C. The three-dimensional tomographic data was subsequently analyzed utilizing Cameca's integrated visualization and analysis software (IVAS), version 3.6.8.

2.3. Mechanical testing

Vickers microhardness tests were performed utilizing a Duramin 5 microhardness tester (Struers), employing a 200 g load and a