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Creep of Al-Sc Microalloys with Rare-Earth Element Additions

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Abstract. Cast and aged Al-Sc microalloys are creep-resistant to 300°C, due to the blocking of dislocations by nanosize, coherent Al_3Sc ($L1_2$) precipitates. Rare-earth elements substitute for Sc in these precipitates, leading to a higher number density of smaller precipitates, which have a greater lattice-parameter mismatch with Al than in the Al-Sc binary microalloy. This leads to an improvement in both ambient temperature microhardness and high temperature creep. Creep threshold stresses of Al-Sc-RE (RE = Y, Dy, or Er) at 300°C are higher than for Al-Sc and Al-Sc-M (M = Mg, Ti, or Zr) microalloys. This is in agreement with a dislocation climb model that includes the elastic stress fields of the precipitates.

Introduction

Al-Sc alloys have excellent mechanical properties at ambient temperatures due to elastically-hard Al_3Sc ($L1_2$ structure) precipitates [1], which remain coherent with the α-Al matrix at elevated temperatures [2, 3]. Also, a coarse-grained (≈1 mm grain diameter) structure forms after conventionally casting Al-Sc microalloys, within the maximum solid-solubility (0.23 at.% Sc at the eutectic temperature), and a high temperature homogenization within the single-phase α-Al region. Because of these two factors, Al-Sc microalloys are creep resistant at high temperatures [4–6].

Ternary additions to Al-Sc microalloys can decrease costs by replacing Sc, and improve mechanical properties. Mg additions lead to solid-solution strengthening [7] and Ti [8] or Zr [9] substitute for Sc in Al_3Sc precipitates. While this increases ambient temperature strength, it decreases the lattice-parameter mismatch between the α-Al matrix and the Al_3Sc precipitates [10]. This, in turn, decreases the creep resistance of the microalloys because it reduces the elastic stresses between dislocations and precipitates during climb bypass, as we observed both experimentally [8, 9] and via modeling [11]. However, the decreased lattice-parameter mismatch and the smaller diffusivity of Zr and Ti in Al [12] lead to an increased coarsening resistance.

Ternary additions to Al-Sc microalloys that substitute for Sc in coherent Al_3Sc ($L1_2$) precipitates and increase the lattice-parameter mismatch should improve creep resistance. The rare-earth (RE) elements Y, Dy, and Er are highly soluble in Al_3Sc and increase the lattice-parameter of $Al_3\,(Sc_{1-x}RE_x)$ [13–15]. While the diffusivities of these REs in α-Al are unknown, it is anticipated that they are similar to that of the light REs, which diffuse more slowly than Sc in α-Al [16, 17]. This implies that the REs should also increase the coarsening resistance. Finally, these REs are less expensive than Sc.

Sawtell and Morris [18] found that additions of 0.3 at.% Er, Gd, Ho, or Y improve the ambient-temperature tensile strength of hypereutectic Al-0.3 at.% Sc alloys by 11–23%. They attributed this to elastic effects associated with the larger atomic radii of RE atoms. Their alloys were chill-cast, but no primary precipitation was observed and they did not homogenize their alloys. Given the high partitioning ratio of Al-RE alloys [12], a highly inhomogeneous distribution of precipitates is anticipated for their work.

In the present study, we investigate the dilute hypoeutectic Al-0.06 at.% Sc alloys, with additions of 0.02 at.% RE (RE = Y, Dy, or Er), thereby permitting an homogenization heat-treatment at 640°C prior to precipitation strengthening. We report on the compressive creep properties of these microalloys at 300°C.

Experimental methods

Materials Preparation and Heat Treatments Al-0.06 at.% Sc-0.02 at.% RE (denoted by Al-Sc-RE) microalloys were studied for three RE additions (Y, Dy, or Er). The low RE concentration was chosen to increase the probability of being in the single-phase α-Al field during the homogenization treatment at 640°C, as the exact solid-solubility of these REs in α-Al is very small and unknown.

The microalloys were dilution-cast from 99.99 at.% Al and Al-1.2 at.% Sc and Al-1 at.% RE master alloys. The Al-1 at.% RE master alloys were produced by non-consumable electrode arc-melting from 99.99 at.% Al and 99.9 at.% RE; the latter were supplied by Stanford Materials. Each microalloy was melted in a zirconia-coated alumina crucible in a resistively-heated furnace at 750°C in air. After thoroughly stirring, the melt was cast into a graphite mold resting on a large copper platen to insure both relatively rapid solidification and cooling rates. The chemical compositions of arc-melted master alloys and cast microalloys were determined by ATI Wah Chang (Albany, OR).

Test specimens were machined from the cast billets. Hardness specimens were larger than $5\times5\times3$ mm^3. Cylindrical compression and creep specimens (8.10 mm diameter and 16.10 mm length) were electro-discharge-machined with their long axis in the main billet direction. The dimensions of each specimen were measured before and after each test. The density of a creep specimen was measured before and after creep via the Archimedes method.

The microalloys were homogenized in air at 640°C for 72 h and then quenched into ambient-temperature water. Aging of each microalloy specimen was performed at 300°C in air for different times and was terminated by an ambient-temperature water quench.

Mechanical Properties The Vickers microhardnesses of the six microalloys were measured using a 200 g weight at room temperature on samples ground to a 1 μm surface finish. Ten measurements were recorded on each sample.

Compressive creep experiments at constant load were performed at 300°C in air in a three-zone resistively-heated furnace with a temperature stability of $\pm$1°C. A superalloy compression-cage was used with boron-nitride-lubricated alumina platens. The platen displacement was transmitted by an extensometer connected to a linear voltage displacement transducer (1.0 μm resolution). Strain and loading times were continuously monitored and recorded by a computer. At any given stress level, sufficient creep time was allowed to establish a minimum creep-rate by a linear-regression analysis. The steady-state creep-rate was determined after at least 2 % strain, over approximately the last 0.5 % strain range. The load was then increased and another minimum creep rate was measured. This procedure was repeated until a strain of 10 % was accumulated or the sample had failed. Thus, a single specimen yielded minimum creep rates for up to four different stress levels.

Results and discussion

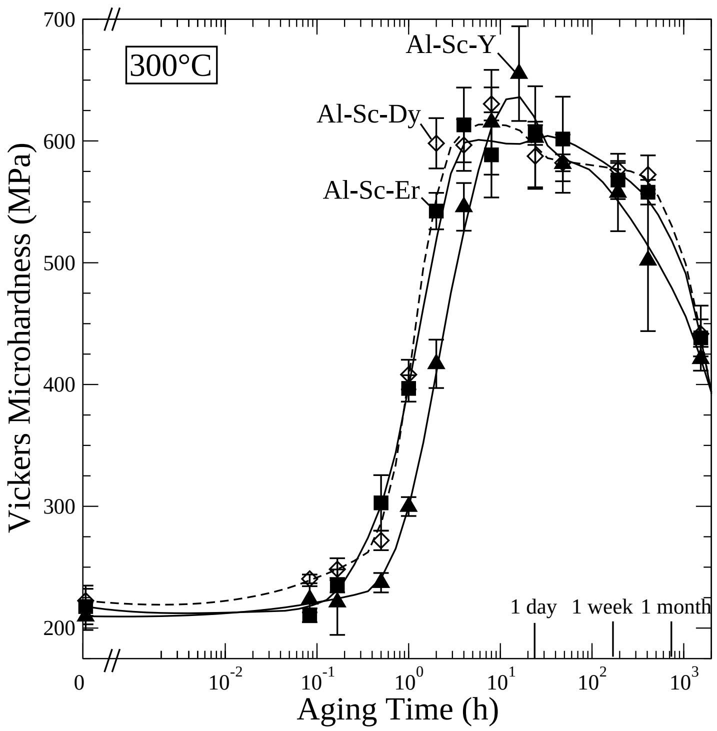

Fig. 1: Microhardness as a function of aging time at 300℃ for Al-0.06 at.% Sc-0.02 at.% RE. Error bars are one standard-deviation from the mean. The three alloys exhibit similar aging responses, but Al-Sc-Y has a higher mean peak microhardness than Al-Sc-Dy, which is harder than Al-Sc-Er.

Microhardness Fig. 1 displays the temporal evolution of microhardness for aging at 300℃ of the three microalloys studied here. The four anticipated stages of precipitation strengthening are observed: (a) a short (0.2 h) initial plateau where the microhardness does not vary (incubation period); (b) a rapid increase in microhardness (under-aged regime); (c) a plateau with high (400 MPa higher than that of the as-homogenized state) microhardness values (peak-aging); and (d) a slow decrease in microhardness (over-aging). Even after aging for 1536 h (64 days), the microhardness is 200 MPa higher than in the as-homogenized state. Although there is a significant sampling error, the mean measured peak microhardnesses for the microalloys increase with the α-Al/$Al_3\,(Sc_{1-x}RE_x)$ lattice-parameter mismatch determined from Vegard's law. Sawtell and Morris [18] also found Al-Sc-Y to be harder than Al-Sc-Er and predicted (from RE atomic radii) that Al-Sc-Dy should have a strength between the two. Our experimental results for these three microalloys are consistent with their simple model.

Creep An anticipated primary creep region, where the strain-rate decreases continuously, always precedes the steady-state creep regime. All alloys and aging treatments exhibit significant creep resistance at 300℃, as shown in Fig. 2. High apparent stress exponents were measured (n_{ap} = 8–20), which is indicative of a threshold stress, below which creep is not measurable.

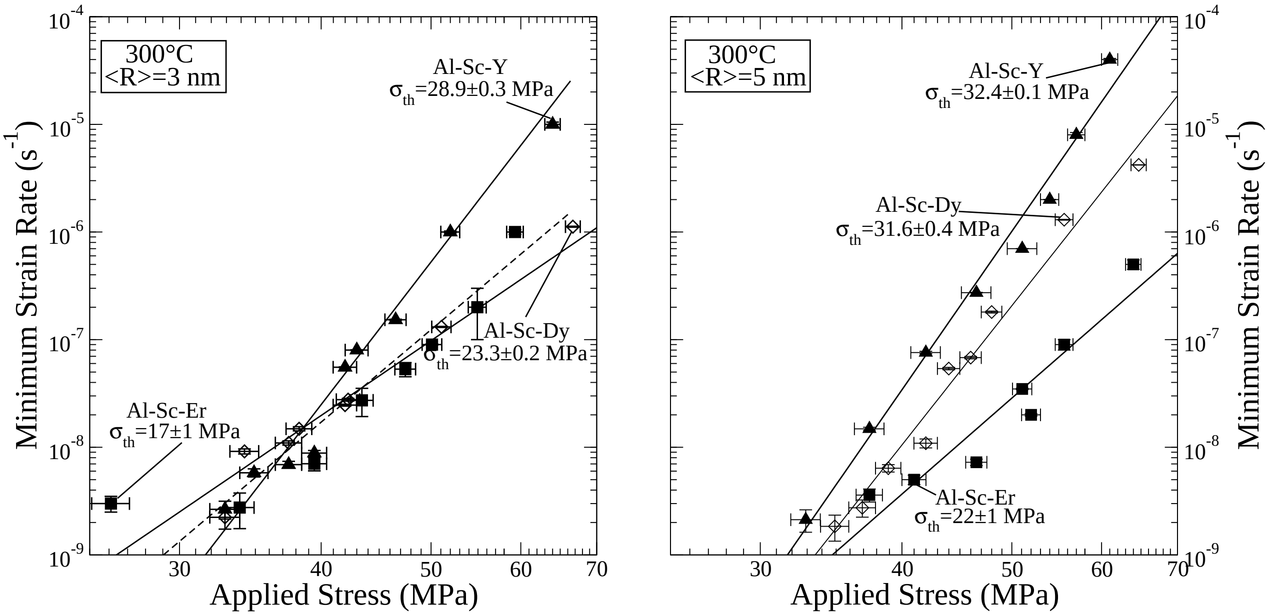

Fig. 2: Double-logarithmic plot of minimum compressive creep strain-rate vs. stress at 300°C for Al-0.06 at.% Sc-0.02 at.% RE. In the left plot, the average precipitate radius, $\langle R \rangle = 3$ nm. In the right plot, $\langle R \rangle = 5$ nm. For any given alloy, the specimen with the larger diameter precipitates is more creep resistant. High apparent stress exponents are indicative of a threshold stress, σ_{th}.

A modified version of the Mukherjee-Bird-Dorn power-law equation states that the minimum strainrate, $\dot{\varepsilon}$, is:

$$\dot{\varepsilon} = A(\sigma - \sigma_{th})^n \exp\left(-\frac{Q}{RT}\right); \tag{1}$$

where A is a constant, σ is the applied stress, σ_{th} the threshold stress, n is the stress exponent, Q the activation energy, R the ideal gas constant, and T the absolute temperature. The value of σ_{th} is calculated by dividing the intercept by the slope employing a weighted least-squares linear-regression of $\sqrt[n]{\dot{\varepsilon}}$ vs. σ [19]. The experimental value for the Al matrix, $n = 4.4$, is used [20]. Threshold stresses for microalloys with $\langle R \rangle = 5$ nm are higher than those with $\langle R \rangle = 3$ nm. This is consistent with our results for Al-Sc [5, 6], Al-Mg-Sc [7], Al-Sc-Zr [9], and Al-Sc-Ti [8] alloys. As was previously observed for the Al-Sc-M alloys, the threshold stresses predicted for shearing and the general dislocation climb models [21] do not explain our results: the former predicts stresses that are too high and the latter does not explain the precipitate radius dependence. The experimental behavior is, however, consistent with a modified general climb model [11], which considers the elastic interactions due to the lattice-parameter and modulus mismatches between the α-Al matrix and the $Al_3(Sc_{1-x}RE_x)$ precipitates. Based on the larger lattice-parameter mismatch exhibited by Al-Sc-RE alloys, this model also predicts correctly larger threshold stresses for these alloys as compared to Al-Sc, Al-Mg-Sc, Al-Sc-Zr, and Al-Sc-Ti alloys. This trend is evident in a normalized threshold plot (Fig. 3).

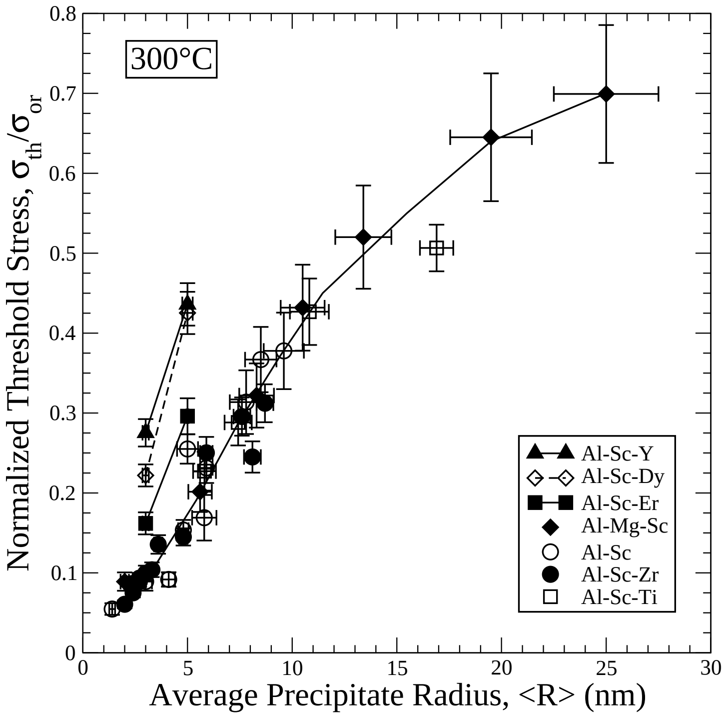

Fig. 3: Creep threshold stress, normalized by the calculated Orowan stress at 300℃, as a function of average precipitate radius, $\langle R\rangle$. Normalized stresses are higher for the three Al-Sc-RE microalloys than any of the previously studied Al-Sc-M alloys [5–9].

Conclusions

The mechanical properties of Al-0.06 at.% Sc-0.02 at.% RE (RE = Y, Dy, or Er) microalloys aged at 300℃ for up to 1536 h were studied at ambient temperature and at 300℃, leading to the following conclusions:

- $Al_3\,(Sc_{1-x}RE_x)$ precipitates lead to high ambient temperature strengths, as measured by Vickers microhardness.
- Even though the Al-Sc-RE microalloys over-age when exposed at 300℃ for over 96 h, the microhardness is greater than the as-homogenized hardness after as long as 1536 h.
- Al-Sc-RE alloys are creep resistant at 300℃ and exhibit a threshold stress that is greater in microalloys containing 5 nm radius $Al_3\,(Sc_{1-x}RE_x)$ precipitates than those containing 3 nm radius $Al_3\,(Sc_{1-x}RE_x)$ precipitates. This same trend was observed in Al-Sc, Al-Mg-Sc, Al-Sc-Zr, and Al-Sc-Ti microalloys we studied previously. The results are also in agreement with a modified general climb model that includes elastic interactions between dislocations and the stress fields of $Al_3\,(Sc_{1-x}RE_x)$ precipitates in an α-Al matrix.

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