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CREEP PROPERTIES OF COARSE-GRAINED Al(Sc) ALLOYS AT 300°C

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Introduction

Most precipitation-strengthened aluminum alloys currently being used are limited to relatively low temperature usage, because of the dissolution and/or rapid coarsening of their precipitates (1). Two-phase Al(Sc) alloys represent an exception, because they contain very fine coherent cuboidal Al_3Sc precipitates, faceted on {100} planes (2), with a melting temperature of 1320°C (3) and very low coarsening rates (2). Studies of the precipitation kinetics (1,4) demonstrate that Al_3Sc precipitates ($L1_2$ crystal structure) form in an Al(Sc) solid solution, resulting in a two-phase microstructure, which is qualitatively similar to that found in γ/γ' Ni-based superalloys. In order for scandium, however, to dissolve completely and form a primary single-phase solid-solution the scandium concentration must be less than 0.38 wt.% (2), corresponding to the maximum solid solubility of Sc in Al at the eutectic temperature (660°C) (2). After quenching an alloy with a Sc concentration of less than 0.38 wt.% from the primary single-phase solid-solution region and aging in the two-phase region, cuboidal Al_3Sc precipitates readily nucleate and grow.

The ratio of proof stress to atomic concentration of alloying element demonstrates that scandium has the highest strengthening effect of any alloying element currently added to aluminum alloys (4,5). Al_3Sc precipitates are also found to influence the microstructure by increasing the recrystallization temperature (6) and by severely limiting grain growth after recrystallization (7). Furthermore, because the density of scandium is only 11% higher than that of aluminum (8), Al-Sc alloys have approximately the same density as pure aluminum.

Much of the recent work on high temperature mechanical properties of Al(Sc) alloys has focused on diffusional creep and superplastic deformation with fine recrystallized grains (9–11). The purpose of this report is to explore the dislocation creep behavior of coarse-grained binary Al(Sc) alloys and to discuss the strengthening effect of the Al_3Sc phase.

Experimental Procedure

Al-0.07 wt.% Sc and Al-0.21 wt.% Sc alloys were produced by diluting an Al-2.1 wt.% Sc master alloy supplied by Ashurst Technology Ltd. (Baltimore, MD) with 99.99 wt.% pure aluminum. The elemental composition was verified employing chemical mass emission analysis from samples located near the center of the ingot. Alloys were melted in air in a high-purity alumina crucible and cast into ingots in a boron nitride-coated graphite mold. The mold was placed on a large copper plate to promote directional solidification. The resulting ingots were subjected to a homogenization and grain-coarsening treatment at 640°C for 24 hours under argon, quenched into water at 23°C, and then aged in air at 350°C for one hour. The mass density of an ingot was measured by Archimedes' method.

Creep specimens were machined from the heat-treated ingots into tensile bars with a gauge length of 18 mm and a gauge radius of 2 mm. Tensile creep testing was performed in accordance with ASTM #E139 specifications. Specimens were tested at 300°C employing constant loads (5-29 MPa) in air in a three-zone resistively heated furnace, with a temperature stability of $\pm$1°C after an 85 minute anneal at the test temperature. The specimen displacement was recorded through a linear voltage displacement transducer with a resolution of 2.5 μm connected to an extensometer, which was attached to the gauge length.

During creep tests, the strain and strain rate were continuously monitored. At any given stress value, sufficient time was allowed to establish a minimum creep rate. After the minimum creep rate was found, the load was changed and the primary and secondary creep rates were again measured at the new stress value. Three specimens were used for the creep study of Al-0.21 wt.% Sc and two specimens for Al-0.07 wt.% Sc. To study aging effects for the latter alloy, one of the deformed Al-0.07 wt.% Sc specimen was rehomogenized at 640°C after having been tested at various stress values without fracturing. The specimen was then aged for 1 hour at 350°C, followed by 34 hours at 300°C (expected to give a condition closer to peak-aging, based on room-temperature hardness curves (6)) and retested at 300°C and stress levels increasing from 8.5 MPa. After a second rehomogenization and re-aging under the same conditions, the aged condition was verified by a second creep test at 8.5 MPa.

A microstructural investigation of the alloys was performed employing optical microscopy and transmission electron microscopy (TEM). Optical microscopy specimens were polished with SiC paper and an alumina slurry and then etched with Keller's reagent. The grain size was determined by counting the number of grains and dividing by the total surface area of the observed specimen. TEM specimens were cut from the gauge section of unfractured crept samples (Al-0.07 wt.% Sc aged at 300°C and 350°C, and cooled under stress) with foil normals perpendicular to the direction of loading, mechanically polished to a thickness of 130 μm, and then jet polished employing 33% nitric acid in a methanol solution at -50°C.

Results and Discussion

Figure 1 exhibits bright (a) and dark field (b) TEM micrographs of a representative sample of an Al-0.07 wt.% Sc specimen, which demonstrates the presence of coherent Al_3Sc precipitates within the aluminum matrix. Al_3Sc precipitates are indicated by a coherency strain contrast that appears in the micrographs as a pair of lobes or a "coffee-bean" (denoted by the arrows labeled A), demonstrating that Al_3Sc precipitates are coherent with a mean size of 26 $\pm$ 2 nm (20 precipitates were measured) and are present at a number density of $(2.4 \pm 0.9) \times 10^{14}$ cm^{-3} (measured on 6 different 1 μm $\times$ 1 μm representative areas). This strain-field contrast effect was first investigated for electron microscopy by Ashby and Brown (12–14) who extended the dynamical theory of TEM diffraction to explain how the strain contrast pattern of spherical coherent inclusions can exhibit a line of no-contrast between two lobes; this was extended to cuboidal precipitates by Sass et al. (13) Also present in Fig. 1 are several precipitates interacting with dislocations, one of which is indicated by the arrow labeled B. Utilizing the experimentally measured mean size and precipitate number density, the precipitate volume fraction (in percent) was determined to be 0.23 $\pm$ 0.14% (assuming spherical precipitates), in approximate agreement with the value predicted from the overall composition (0.20%). This small volume fraction of the Al_3Sc phase complicated the task of finding precipitates and precipitate-dislocation interactions.

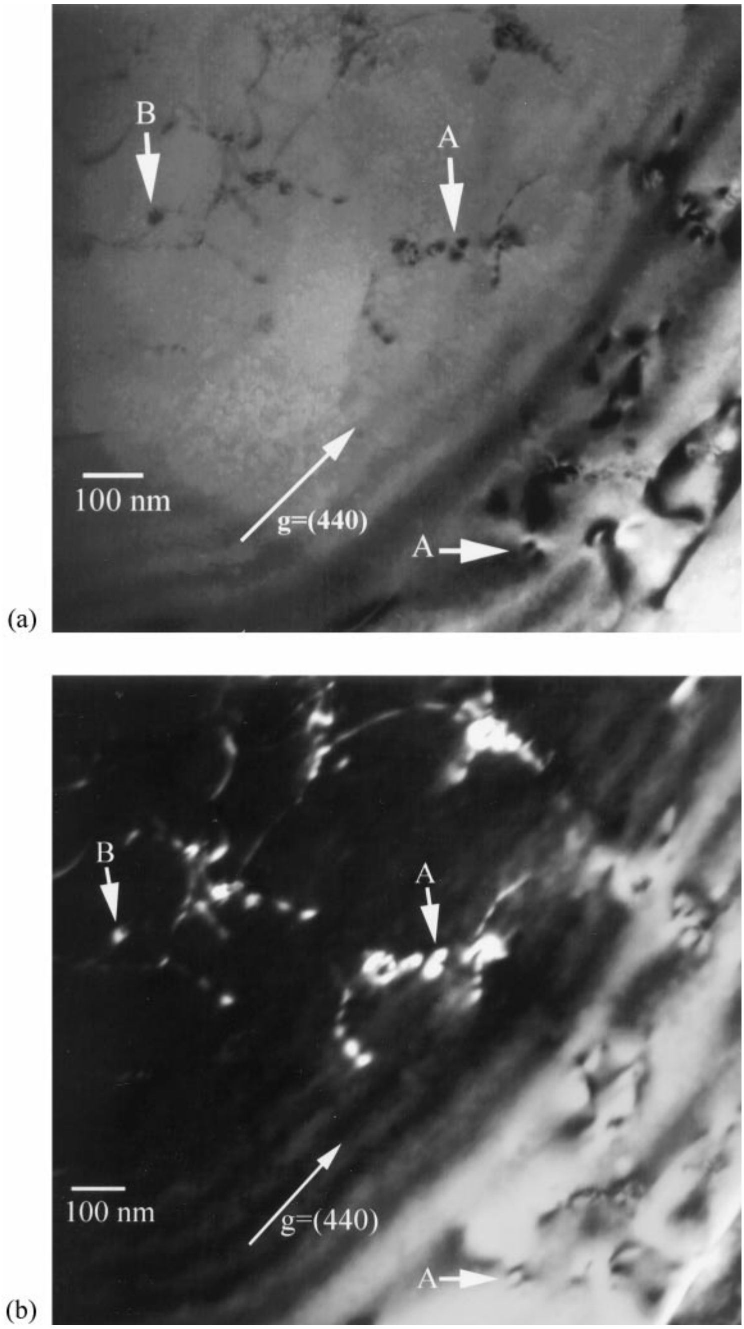

Figure 1. Bright-field (a) and dark-field (b) two-beam TEM micrographs, recorded employing a 440 operating reflection, of a crept Al-0.07 wt.% Sc tested at 300°C and cooled under load. Arrows on micrographs denote the presence of Al_3Sc precipitates with strain-field contrast (A) and an Al_3Sc precipitate interacting with a dislocation (B).

Density measurements demonstrate that the as-machined samples have a density which is 99.90 ± 0.03% of the theoretical value of 2.702 g cm^{-3} for Al-0.07 wt.% Sc. Optical microscopy of an Al-0.21

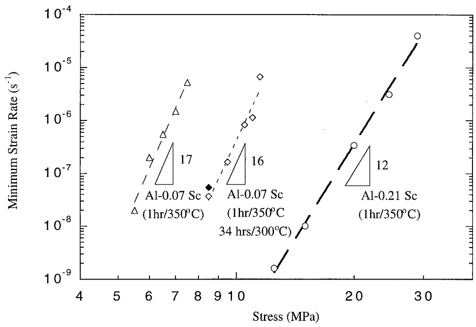

Figure 2. Minimum creep rate of Al(Sc) alloys at 300°C as a function of applied tensile stress (aging time and temperature are given in parentheses). Second data point (◆) for Al-0.07 wt.% Sc was used for verification of aging characteristics.

wt.% Sc sample reveals that the grain-coarsening treatment at 640°C gave a grain density of 16 cm^{-2} (corresponding to a grain size of about 0.25 cm), with no evidence of further grain coarsening after the creep experiments. Given the very coarse grain size, grain-boundary diffusional creep can be neglected and creep deformation can be assumed to result primarily from dislocation creep mechanisms. The creep behavior of the two-phase Al(Sc) alloys is presented in Figs. 2 and 3 on double logarithm plots of stress vs. minimum strain rate. Figure 2 illustrates that creep resistance is sensitive to both the Sc concentration and heat-treatment. Figure 2 also demonstrates that the apparent stress exponent of the alloys ($n = 12$ for Al-0.21 wt.% Sc and $n = 16–17$ for Al-0.07 wt.% Sc) is much higher than for pure aluminum [$n = 4.4$ (15)], indicative of the existence of a threshold stress. Dispersion-strengthened alloys have been shown to exhibit a threshold stress, below which dislocation creep does not take place because dislocations do not have sufficient energy to bypass precipitates (16). Also Fig. 2 indicates that the threshold stress, estimated by extrapolating the creep curve to a strain rate of 10^{-10} s^{-1} (17), increases from 4 to 10 MPa by increasing the Sc concentration from 0.07 to 0.21 wt.%, for the same initial heat treatment (350°C for 1 hr). The threshold stress in Al(Sc) alloys will be modeled in future publications.

An exploratory investigation of aging in Al-0.07 wt.% Sc alloys indicates that these alloys must be aged longer than the Al-0.21 wt.% Sc alloys to achieve optimal creep strength. The Al-0.07 wt.% Sc alloy is underaged when undergoing the same aging treatment (350°C for 1 hr) as the Al-0.21 wt.% Sc alloy (Fig. 2), as demonstrated by the improved creep properties after an additional heat treatment at 300°C for 34 hours. The creep resistance increases by three orders of magnitude, and concomitantly the threshold stress from 4 to 6 MPa, between the two aging conditions. These results can be approximately correlated with hardness curves published by Elagin *et al.* (6), where Al-0.1 wt.% Sc and Al-0.2 wt.% Sc alloys have peak hardness values at 6 and 55 hours at 300°C, respectively. A direct comparison is difficult, because all samples in our study were aged for one hour at 350°C (peak-aging for Al-0.2 wt.% Sc (4)) followed by various times at 300°C during creep testing.

Figure 3 compares the creep properties of Al(Sc) alloys with those of other aluminum alloys near 300°C. First, at low stresses, the Al(Sc) alloys exhibit a creep resistance which is significantly higher than pure aluminum. Second, we consider an Al-2 wt.% Mg alloy that is solid-solution-strengthened by magnesium, which produces a change from a power-law creep mechanism ($n = 5$) to a glide creep mechanism ($n = 2.9$) (18). Despite a ten-fold higher alloying element concentration, this alloy has a creep resistance lower than the two Al(Sc) alloys below 12 MPa and 29 MPa, respectively, because it does not exhibit a threshold stress. The third comparison in Fig. 2 is with one of the most creep-resistant aluminum alloys prepared to date, rapidly solidified (RS) Al-11.7 wt.% Fe-1.15 wt.% V-2.4 wt.% Si, which is precipitation-strengthened and also exhibits a threshold stress (19). Not surprisingly, this alloy

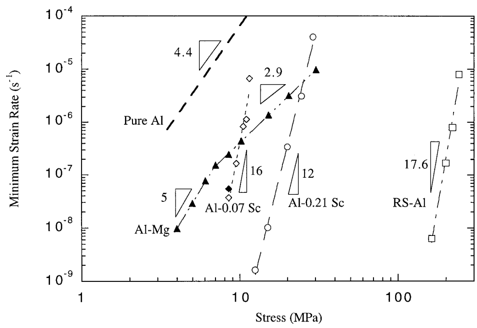

Figure 3. Minimum creep rate of Al(Sc) alloys at 300°C as a function of applied stress. Data for coarse-grained pure Al at 300°C (15), Al-2 wt.% Mg at 300°C (18), and RS-Al (Al-11.7 wt.% Fe-1.15 wt.% V-2.4 wt.% Si) at 314°C in compression (19) are presented for comparison.

has a substantially higher creep resistance than the Al(Sc) alloys due to the much larger alloying content (a total of 15.25 wt.%), increasing the number density of precipitates and thus the efficiency of dislocation pinning.

Conclusion

An initial study of the dislocation creep behavior at 300°C of two coarse-grained Al(Sc) alloys consisting of a pure aluminum matrix with small (26 ± 2 nm) coherent Al_3Sc precipitates is presented. Both Al(Sc) alloys (0.07 and 0.21 wt.% Sc) exhibit a threshold stress that is dependent on the volume fraction and size of the precipitates, which is typical of dispersion-strengthened materials. The combination of low-density, high coarsening-resistance, and high creep-resistance make this alloy potentially attractive for aerospace or automotive structural applications at elevated temperatures.

Acknowledgments

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