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 Al3Sc
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 Al3Sc precipitates (L12
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 Al3Sc
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). Al3Sc
precipitates are also found to influence the microstructure by increasing the recrystallization temper-
ature (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 Al3Sc 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 ±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, mechan-
ically 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 Al₃Sc precipitates within the
aluminum matrix. Al₃Sc 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
Al₃Sc precipitates are coherent with a mean size of 26 ± 2 nm (20 precipitates were measured) and are
present at a number density of (2.4 ± 0.9) × 10¹⁴ cm⁻³ (measured on 6 different 1 μm × 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 ± 0.14% (assuming spherical precipitates), in approximate
agreement with the value predicted from the overall composition (0.20%). This small volume fraction
of the Al₃Sc phase complicated the task of finding precipitates and precipitate-dislocation interactions.
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 1. Bright-field (a) and dark-field (b) two-beam TEM micrographs, recorded employing a 440 operating reflection, of a creep Al-0.07 wt.% Sc tested at 300°C and cooled under load. Arrows on micrographs denote the presence of Al\(_3\)Sc precipitates with strain-field contrast (A) and an Al\(_3\)Sc 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
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 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.

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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...
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 Al₃Sc 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.

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