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# Effect of nano-scale particles on the creep behavior of 2014 Al

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#### Abstract

The creep behavior of two grades of 2014 Al was investigated over six orders of magnitude of strain rate. While these two grades were prepared by powder metallurgy (PM), they have different oxygen levels (0.3 and 1.0 wt.% oxygen). The creep data show that the creep behavior of these two grades of PM 2014 Al is similar to that of dispersion strengthened (DS) alloys with regard to the following characteristics: (a) the apparent stress exponent is high and variable, and (b) the apparent activation energy is higher than that for self-diffusion in Al. An analysis of the experimental data indicates that there exists a threshold stress for creep whose temperature dependence is strong. The values of threshold stress increase noticeably with the increase of oxygen content in the alloy. This finding together with the observation of extensive interaction between moving dislocations and nano-scale dispersion particles indicates that the presence of oxide particles is responsible for the threshold stress creep behavior in 2014 Al. By considering the effect of the threshold stress, it is demonstrated that the true creep characteristics of PM 2014 Al are consistent with those reported for Al solid-solution alloys.

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#### 1. Introduction

The creep behavior of powder metallurgy (PM) Al alloys has been extensively investigated [1–5] due to their use in several structural applications. These alloys can be utilized in lightweight high-strength structures or as matrices in the development of discontinuous SiC reinforced Al composites [4–9].

As a result of recent creep investigations [1–3], it has been found that PM Al alloys, unlike conventional solid-solution alloys of the same compositions, exhibit apparent stress exponents and apparent activation energies that are high and variable. In particular, the apparent activation energies reported are higher than that for self-diffusion in Al. The difference in creep behavior between PM Al alloys and conventional solid-solution Al alloys has been attributed to the

existence of a threshold stress in creep [1–3]. It has been suggested [1–3] that the origin of the threshold stress may be related to the interaction between moving dislocation and dispersion particles. Substructural data using transmission electron microscopy have revealed [1,2] clear evidence for such an interaction, which resemble in character those reported for DS alloys [10–12]. On the basis of the morphology of these particles and the processing history of PM Al alloys, it further suggested [1,2] that such dispersion particles most likely represent oxides, which are introduced in Al alloys as a result of processing the alloys by powder metallurgy.

In earlier investigations [2,3], oxides (Al and Mg oxides) were inadvertently introduced in PM Al alloys as a result of using Al alloy powders in preparing the alloy by PM. According to available information [13], during the atomization process, Al powders exhibit a stable surface oxide due to the reactivity of aluminum with oxygen. This oxide layer is an amorphous Al<sub>2</sub>O<sub>3</sub>-H<sub>2</sub>O film that contains small amounts of MgO crystallites. During the step of pressing (cold or hot), the surface layer breaks up into Al<sub>2</sub>O<sub>3</sub> fragments and MgO

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crystallites. Finally, hot extrusion results in further breaking up Al<sub>2</sub>O<sub>3</sub> fragments and Al<sub>2</sub>O<sub>3</sub>/MgO crystallites and in dispersing oxide particles over a wide area.

In the present investigation, oxide particles are purposely introduced in an Al alloy in order to study the effect of their volume fraction on the creep behavior of the alloy and the value of the threshold stress. It is the purpose of this paper to report and analyze the data obtained in the investigation.

#### 2. Experimental procedure

In this section, experimental procedures including those dealing with material preparation, creep testing, and substructure examination are described.

#### 2.1. Materials and processing

The alloy selected for the present investigation is 2014 Al that has the following major solute elements in weight percent: 3.8 Cu; 0.8 Mg; 0.3 Mn; 0.2 Fe; 0.5 Si; 0.1 Cr; and 0.15 Ti. Two grades of the alloy were specially processed by PM in a materials laboratory at the National Taiwan University using two different oxygen levels: 0.3 and 1.0 wt.%. H<sub>2</sub>O moisture was used to introduce oxides. The starting material was prealloyed 2014 Al powders whose average particle size was about 29 µm. The PM process employed consisted of powder blending, cold isotropic pressing, sintering, degassing, and hot extrusion. In order to obtain different mounts of oxide in the materials, the sintering process was performed in air. The compacted billet received a degassing treatment at 673 K for 1 h. Then, the sintered compacts were extruded at 723 K with a reduction ratio of approximately 36:1 to form bars of 15 mm in diameter.

#### 2.2. Creep testing

Experiments were conducted under conditions that were similar to those used in an earlier investigation on PM 2124 Al [2]. The advantages for using double-shear specimens for creep tests have been described in other reports [14,15]. Prior to testing, all specimens were solutionized in an argon gas atmosphere at 768 K for 2h, water quenched, and left at room temperature for a minimum of 1 week. The purpose of this heat treatment was to eliminate the effects of machining and to stabilize the grain size of the material. Three different testing temperatures of 618, 648, and 678 K were used to conduct tests on a creep-testing machine operated at constant load (for specimens of double shear configuration, constant load is equivalent to constant stress); the details of the testing procedures were described elsewhere [2,3].

#### 2.3. Microstructure examination

For optical microscopy, samples were individually mounted, mechanically polished and finally etched at room temperature using a mixture of 2% HF, 3% HCl, 5% HNO<sub>3</sub> and 90% H<sub>2</sub>O (Keller's Reagent) to check the grain size.

For the purpose of TEM investigation, specimens after straining were cooled rapidly under load to preserve the substructure developed during creep. The samples for TEM examination was prepared by cutting thin slices, approximately 0.4 mm thick, from the gauge length of crept specimens. The thin slices were reduced to a thickness of about 0.1 mm by mechanical polishing. The final thinning of the slice to form a TEM thin foil is carried out in a twin-jet electropolisher. The solution used for twin-jet polishing was a mixture of 25% methanol and 75% HNO<sub>3</sub>. Dry ice was used to keep the temperature of the solution lower than 240 K. The thin foils prepared by this technique were examined in a Philips CM-20 transmission electron microscope operated at 200 kV.

#### 3. Results

#### 3.1. Creep curves

The creep curves obtained for the two grades of PM 2014, like those reported for both PM 2124 A1 [2] and PM 6061 Al [3], exhibit the following characteristics: (a) an instantaneous strain,  $\gamma_0$ ; (b) a normal primary (transient) stage, where creep rate, $\dot{\gamma}$ , decreases continuously with time, t; (c) a steady-state stage (secondary stage), where  $\dot{\gamma}$  is constant; and (d) a tertiary stage, where  $\dot{\gamma}$  increases with time.

### 3.2. Stress dependence of steady-state creep rates

In Fig. 1a and b, the creep data of grades of PM 2014 Al containing 0.3% oxygen and 1.0% oxygen, respectively, are plotted as steady-state creep rate,  $\dot{\gamma}$ , against applied shear stress,  $\tau$ , on a double logarithmic scale at three temperatures. Inspection of these figures reveals that for each testing temperature, the apparent stress exponent,  $n_{\rm a}$ , inferred from the slope of the plot ( $n_{\rm a} = \partial \ln \dot{\gamma}/\partial \ln \tau$ ) is not constant but increases with decreasing applied stress. This behavior is similar in trend to that reported for 2124 Al [2] and 6061 Al [3]. For the purpose of comparison, the data for both grades obtained at a single temperature of 618 K were plotted in Fig. 2 as steady-state creep rate,  $\dot{\gamma}$ , against applied shear stress,  $\tau$ , on a double logarithmic scale. It is clear that the data of the two grades coalesce at high stresses, but they diverge at low stresses.

#### 3.3. Temperature dependence of steady-state creep rate

The data in Fig. 1a and b were used to plot the logarithmic  $\dot{\gamma}$  against 1/T at constant stress, where T is the absolute temperature. The apparent activation energy for creep,  $Q_a$ ,

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