Study of Creep in Al_2O_3 Thermally Grown on β-NiAl

B.W. Veal, A.P. Paulikas, J. Linton, G. Jennings
Argonne National Laboratory, Argonne, IL, U.S.A.

Introduction

Thermally grown aluminum oxide, which forms naturally on a variety of commercially important structural alloys, provides essential corrosion protection that enables extended operation at elevated temperatures. For example, the successful operation of turbine blades for aircraft engines and land-based energy generation systems depends on the formation of this adherent, self-healing protective oxide. Higher operating temperatures, needed for improved efficiency and longer lifetime operation, require further improvements in the protective coatings.

One of the important issues influencing the behavior of the protective coatings is the creep response to stress perturbations. It is known, for example, that failure often occurs in association with thermal cycling, where large stresses can build up because of differences in thermal expansion coefficients between the metal substrate material and the oxide layer.

Methods and Materials

We have developed a technique to measure, *in situ*, with high precision and accuracy, the evolution of strains that develop in the oxide during growth and in response to stress perturbations. The technique exploits synchrotron radiation utilizing bending magnet radiation (beamline 12-BM) at the APS. We have applied the technique to the measurement of creep phenomena in Al_2O_3 that is thermally grown on β-NiAl.

Results

In Fig. 1, data obtained in the first 400 minutes show measurements of the in-plane strain as the oxide grew and evolved at 1100°C. Initially, a large tensile stress can be observed; this rapidly relaxed to zero. This initial tensile stress was attributable to the transformation of transition aluminas, which form at temperatures below 1000°C, to the stable α-phase. Since the transition aluminas (e.g., γ, θ, κ) were low-density cubic phases, a volume contraction was associated with the transformation. This resulted in the formation of a tensile stress in the converted α-phase. However, once the phase transformation was completed, very little stress remained in the oxide. Consequently, creep processes could be investigated without the need to separate behavior associated with growth strains.

Figure 1 shows measurements from two samples of single crystal β-NiAl. For the first sample (black dots), the temperature was abruptly lowered to 950°C. This temperature excursion applied a compressive stress to the oxide because the thermal expansion coefficient of the relatively massive metal substrate was larger than that of the thin oxide. The creep response in the oxide was then monitored for about 400 minutes at 950°C. The temperature was then returned to 1100°C, where a small and rapidly decaying tensile stress was observed. The second sample (red dots) was also initially oxidized at 1100°C, followed by heat treatments at 1050, 1000, and 950°C.

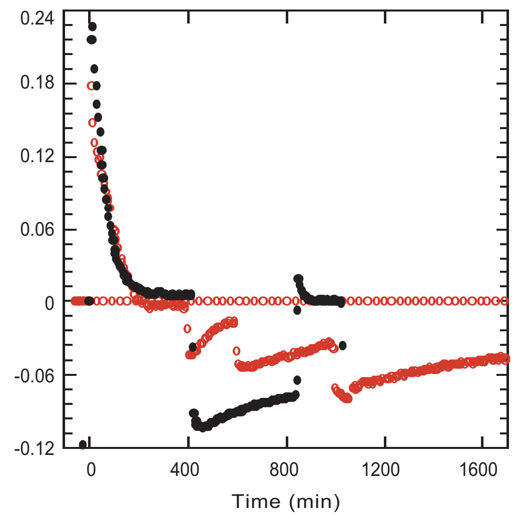

FIG. 1. In-plane strain in alumina thermally grown on two β-NiAl samples measured versus oxidation time. After 400 minutes, the temperatures were changed, and creep was monitored (see text).

Discussion

The data in Fig. 1 can be analyzed to yield a creep rate, at a given temperature, as a function of applied stress σ (using Young's modulus to obtain stress from the measured strain ε). At low stress levels (less than about 100 MPa), the creep rate was typically proportional to the stress, and it converted to power law behavior at high stress levels (i.e., $d\varepsilon/dt = A\sigma^n$).

Figure 2 shows creep rate versus stress obtained at three different temperatures from those considered in Fig. 1. At low stress levels, linear behavior occurred. For the 950°C measurements, power law behavior was observed, with n = 4 in the high-stress region (>300 MPa) and n = 2 at intermediate stress levels.

The Coble model [1], applicable in the low-stress,

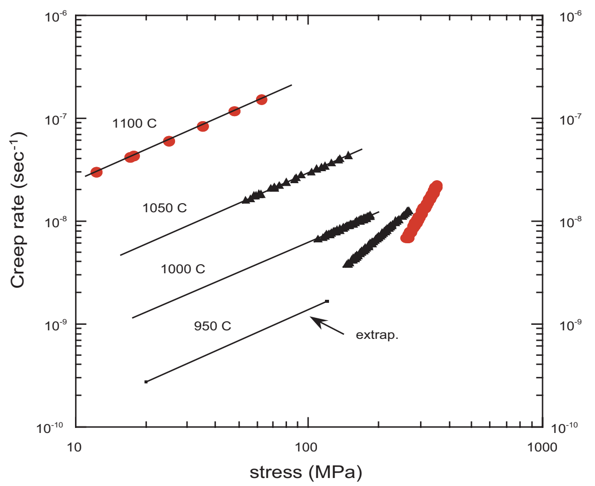

FIG. 2. Creep rate versus stress. At low stress levels, the creep rate is linear. At high stress levels, the creep rate acquires power law behavior.

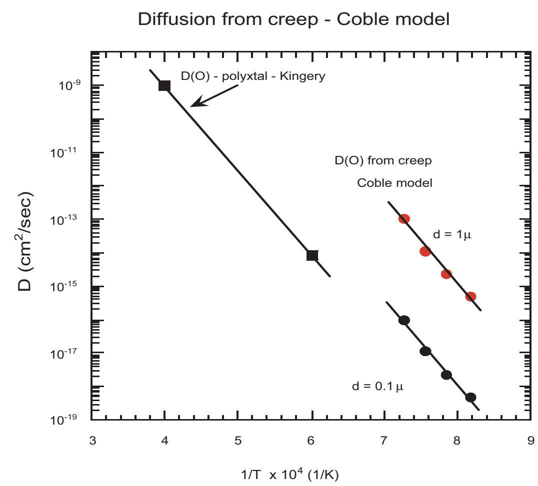

FIG. 3. Oxygen grain boundary diffusion coefficient obtained from the creep data and application of the Coble model compared to tracer diffusion measurements from Kingery et al. [2].

linear region, relates the creep rate to the grain boundary diffusion rate D_{gb} of the slow-moving species (oxygen). In other words, $d\varepsilon/dt = (150\Omega\delta D_{gb}\sigma)/(\pi d^3 k_B T)$, where δ is the grain boundary width, d is the grain size, and Ω is the vacancy volume. Figure 3 shows the diffusion coefficient obtained from the Coble model and measured creep rates obtained when n = 1. Results are shown on the basis of the assumption that the grain sizes are 0.1 and 1.0 µm; these are the grain sizes that appear to bracket the grain size distribution in the oxide. (However, d = 0.1 appears to be closer to the average grain size.) Results are compared to the oxygen grain boundary diffusion data of Kingery et al. [2]. Although they were obtained at significantly higher temperatures, the Kingery data appear to be in reasonable agreement with the diffusion results obtained from our creep measurements. The activation energy, obtained from the creep relaxation times measured at different temperatures, is 4.2 eV. This value is also in reasonable agreement with reported activation energies (typically 4 to 6 eV) for Al_2O_3 that were measured by using a variety of techniques [3].

Acknowledgments

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