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Annealing Effects on Creep and Rupture of Polycrystalline Alumina-Based Fibers

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ANNEALING EFFECTS ON CREEP AND RUPTURE OF POLYCRYSTALLINE ALUMINA-BASED FIBERS

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SUMMARY

Continuous-length polycrystalline aluminum-oxide-based fibers are being considered as reinforcements for advanced high-temperature composite materials. For these fine-grained fibers, basic issues arise concerning grain growth and microstructural instability during composite fabrication and the resulting effects on the fiber's thermo-mechanical properties. To examine these issues, commercially available Nextel 610 (alumina) and Altex (alumina-silica) fibers were annealed at 1100 and 1300 °C for up to 100 hr in air. Changes in fiber microstructure, fiber tensile creep, stress rupture, and bend stress relaxation (BSR) that occurred with annealing were then determined. BSR tests were also used to compare as-received and annealed fibers to other polycrystalline oxide fibers. Annealing was shown to have a significant effect, particularly on the Altex fiber, and caused it to have increased creep resistance.

INTRODUCTION

Previous studies [1-3] on fine-grained, polycrystalline alumina-based fibers have demonstrated that the physical and mechanical properties of the fibers are dependent on thermal history. Since these fibers are being considered as reinforcements for high-temperature structural composites, it is important to develop an understanding of the effects of thermal exposure during composite fabrication on such fiber properties as tensile strength, creep, and stress rupture.

Prior work on the polycrystalline alumina fiber Nextel 610 demonstrated that little grain growth occurred at temperatures up to 1200 °C and times of 10 hr [2,3]. However, the same studies showed that under no-load conditions, the fiber's tensile strength degrades due to the thermally activated growth of critical flaws. For the fine-grained 85% Al_2O_3–15% SiO_2 Altex fiber, heat treatments above 1127 °C transformed the amorphous silica and its transition alumina mixture to mullite [1]. The as-produced Altex fibers tested at 1000 to 1200 °C [4] exhibited significantly greater creep rates than did Nextel 610 in the same temperature and stress range [5]. However, no creep experiments were performed on Altex fibers after they were annealed to the mullite composition.

Another 85% Al_2O_3–15% silica fiber with a mullite-alumina composition was shown to have creep resistance superior to that of the polycrystalline alumina fibers [1]. This led to the development of commercially available alumina-silica fibers such as 3M's Nextel 720 fiber [6]. Presumably, during high-temperature exposure, the unstable, amorphous Altex fiber microstructure also converts to a more stable, creep-resistant mullite-containing microstructure.

The purpose of this investigation was to determine the effects of thermal exposure on the creep and rupture behavior of two commercially available oxide fibers, Nextel 610 and Altex. To put these results in perspective, the creep behavior of these fibers is compared to other oxide fibers, including yttrium aluminum garnet (YAG) fibers. Conclusions are drawn about microstructural sources and the implications of these effects on composite fabrication.

EXPERIMENTAL PROCEDURE

Table 1 summarizes the properties of as-received fibers, as reported by their respective manufacturers (Nextel 610 is a trademark of the 3M Company and Altex, of Sumitomo). Tensile creep, stress rupture, and bend stress relaxation (BSR) studies were conducted on as-received and annealed Nextel 610 alumina fiber and Altex alumina-silica fiber. All fibers tested were from single lots supplied by their respective manufacturers. The annealing took place in air over periods of 1 to 100 hr at 1100 °C and for 3 hr at 1300 °C. These temperatures were chosen to represent upper temperatures for fabrication of metal- and ceramic-matrix composites, respectively.

The tensile creep and rupture data were obtained between 900 and 1200 °C in air for times ranging from 0.1 to 100 hr. Stresses of 140 to 280 MPa were applied. Figure 1 shows the experimental setup for tensile creep testing in air. Individual fibers were glued to paper grips that were attached to hooks well outside the hot zone of the molybdenum disilicide element furnace, which can reach temperatures up to 1600 °C. The tensile creep deformation measurements were made at the lower hook using a linear variable differential transformer (LVDT) with a free-floating core. The signals from the LVDT were acquired and stored via a direct sensor input card mounted in and controlled by a personal computer. Measurements were taken every minute for the first half hour and then every half hour thereafter. The thermocouples used for temperature control and measurement respectively, were placed at the center of the hot zone, which was determined by thermal profiling to be 25.0 mm long. For the creep-strain calculations, the effective gauge length was taken to be 25.0 mm.

The BSR test [7] is another method for evaluating fiber creep and for ranking the creep resistance of various fiber types. The parameter used to index the creep resistance is the bend stress relaxation ratio m, which is defined as the relative amount of elastic stress remaining in the fiber after a time-temperature treatment under a constant bend strain. The BSR ratio is

$$m = 1 - \frac{R_o}{R_a} \tag{1}$$

where R_o and R_a are, respectively, the radius of curvature for the initially imposed bend strain and the residual radius of curvature after thermal exposure and strain removal. If $m = 1$, no relaxation has occurred; if $m < 1$, some relaxation has occurred; and if $m = 0$, complete relaxation has occurred ($R_o = R_a$). The BSR tests were conducted in air at an imposed radius of 9.5 mm.

Micrographs of the fibers before and after annealing were obtained by using transmission electron microscopy (TEM) on thin foils prepared by ion milling of random cross sections of the fiber. In addition, thin foils were used for x-ray analysis of selective fibers.

RESULTS AND DISCUSSION

Typical tensile creep curves for the as-received Nextel 610 fibers at various stress levels are illustrated in Fig. 2. These curves, taken at 980 °C, show the effects of the 1100 °C air anneals for 100 hr. The curves display steady-state creep with relatively little primary creep strain. The as-received fiber, tested only at 275 MPa, went into tertiary creep with failure at a total creep strain of 5.6%. However, at the same stress, the annealed fiber displayed a much lower creep strain, just under 0.8%. Moreover, the annealed fiber showed no indication of tertiary creep, even after more than 100 hr. The stress on the annealed fibers was increased to 413 MPa, which resulted in a factor of 3 strain increase after 50 hr. When the creep temperature was raised to 1090 °C, the strain in the annealed fiber exceeded 0.3% within 2 hr at 138 MPa (Fig. 3). In contrast, the as-received fiber failed after only 10 min at the same stress. Doubling the stress on the annealed fiber increased the creep strain by a factor of 5.

Figure 4 illustrates the Altex fiber creep response at 1090 °C. Both as-received samples and those annealed at 1100 °C for 100 hr were tested. Altex required 50 hr instead of 2 hr to achieve strain levels comparable to annealed Nextel 610 (cf. Fig. 3). A comparison of both the as-received and annealed Altex fibers at 1090 °C with the same 138 MPa load showed approximately one order of magnitude less creep strain in the annealed fiber. These results suggest that thermally activated mechanisms play an important role in the creep behavior of these fibers.

The effects of annealing on the 0.2% creep strength of the Nextel 610 and Altex fibers are illustrated in Fig. 5 parts (a) and (b), respectively. Nextel 610 at 980 °C required from 1 to 200 hr to reach 0.2% creep at stresses from 700 to 100 MPa. After Nextel was annealed at 1100 °C for 100 hr, the 0.2% creep times increased from approximately 2 to 500 hr for the same stress. However at 1090 °C, no large annealing effect could be discerned in the Nextel 610 creep strength data. In contrast, the annealed Altex fibers show a marked improvement over both the as-received and annealed Nextel 610 fibers in the same stress range, with annealed Altex exhibiting about an order of magnitude increase in the time needed to reach 0.2% strain at both 980 and 1090 °C. In fact, the annealed Altex fiber tested at 1090°C behaved similar to the as-received Altex at 980 °C.

Actual stress-rupture data were limited, but Fig. 6 indicates that, in all cases, annealing increased rupture time in comparison to the as-received fibers. However, annealed Altex shows only a slight increase in rupture time at 980 °C.

The BSR test results were consistent with the tensile creep data. Figure 7 illustrates the m-ratio data for Nextel 610 fiber after 1-, 10-, and 100-hr annealings at 1100 °C in air. The duration of the stress relaxation tests was 1 and 100 hr in air. If we take $m = 0.5$ as an arbitrary value from which we can compare test results, two trends are evident. First, as the anneal time increases, so does the temperature for $m = 0.5$. Second, the 1-hr BSR tests show a higher relaxation temperature than do the 100-hr tests. The first effect may be related to an improvement on the fiber atomic level, that is, the amorphous interface, which controls creep, as is also seen in Fig. 3. The second effect occurs because stress relaxation and creep are controlled by thermally activated mechanisms.

Figure 8 shows the results of 1-hr BSR tests on polycrystalline oxide fibers in their as-received condition as compared with Nextel 610 and Altex fibers before and after annealing at 1100 and 1300 °C [8,9]. The results show only a slight difference in creep resistance between the as-received Nextel 610 and Altex fibers. However, after being annealed at successively higher temperatures, the Altex fibers showed increases in relaxation temperature that were dramatic improvements over those observed in Nextel 610. At these test conditions, Altex annealed for 3 hr at 1300 °C showed relaxation temperatures comparable to those of a developmental polycrystalline YAG fiber [9]. Because of the YAG fiber's 1600 °C processing temperature, subsequent annealing in the temperature range used in this investigation should not increase its relaxation temperature.

Micrographs of the Nextel 610 in the as-received and annealed condition (100 hr at 1100 °C) are seen in Fig. 9. The initial average grain size of 0.1 µm appears unchanged. In contrast, the Altex microstructure shown in Fig. 10 reveals the initial 0.02-µm grain size increased fourfold after a 3-hr, 1300 °C exposure. Unlike Nextel 610, the Altex grain morphology remains equiaxed after being exposed to this temperature. In addition, the x-ray analysis of the as-received Altex fiber shows alumina, but x-ray results from the annealed fiber, whether from TEM foils or the powder diffraction method, indicate a mostly mullite phase. This is consistent with the results found by Lesniewski et al. [1] in their annealing studies of the Altex fiber. It is likely that the transformed mullite phase, as well as the stabilized transition alumina, is the source of the increase in the creep and relaxation temperatures for the Altex fiber. As expected, Nextel 610 polycrystalline alumina exhibited no such phase transformation under the annealing conditions of this study. The smaller increase in creep resistance in the Nextel 610 fiber after annealing is probably due to changes in the grain boundary morphology and/or the chemistry on the atomic level, both of which directly affect grain boundary sliding.

CONCLUSIONS

The high-temperature creep deformation behavior of commercial polycrystalline-alumina-based fibers can be modified by annealing. This may offer the opportunity to improve high-temperature creep resistance of composites without affecting their stress-rupture behavior. For multiple- and transition-phased fibers such as Altex, annealing produces phase and microstructural changes that allow them to become as creep resistant as polycrystalline YAG. The results of this study also show that the creep properties of the as-received fiber may not be appropriate for modeling composite creep behavior if the fiber creep properties are changed by fabrication or service conditions.

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Table 1.—Properties of As-Received Fibers[a]

	Nextel 610	Altex
Composition, wt %		
Al_2O_3	99	85
SiO_2	0.2–0.3	15
Fe_2O_3	0.4–0.7	*
Average grain size, μm	0.1	0.02
Average diameter, μm	14	16
Density, g/cm^3	3.9	3.2
Tensile elastic modulus, Gpa	380	280
Tensile strength, MPa	2400	1450

[a]As reported by their manufacturers, 3M (Nextel 610) and Sumitomo (Altex).

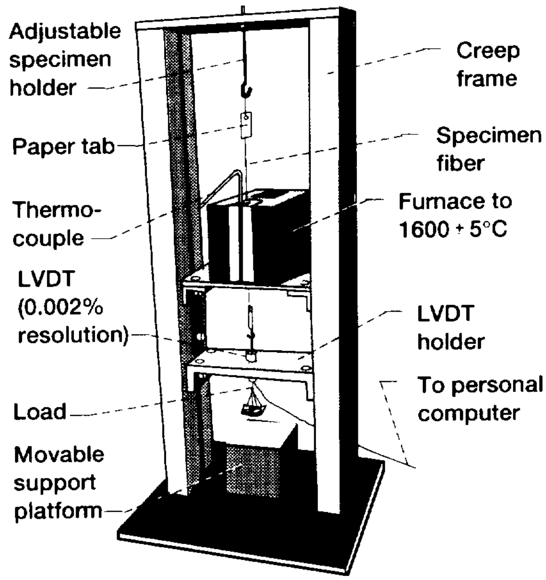

Figure 1.—The experimental setup using a dead-weight loading system for tensile creep testing in air.

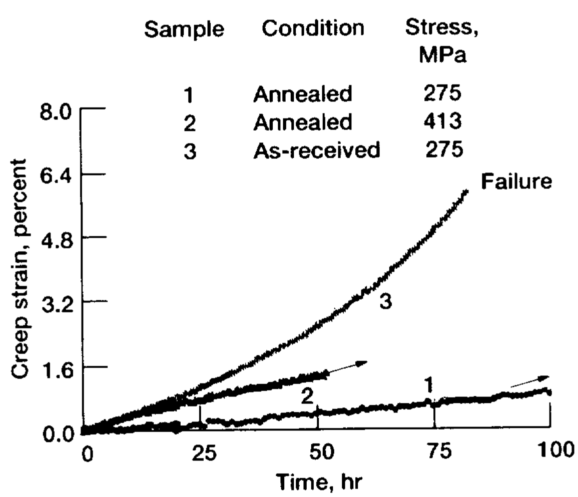

Figure 2.—Typical tensile creep curves for as-received and annealed Nextel 610 fibers under stresses of 275 and 413 MPa at 980 °C (arrows indicate interrupted tests).

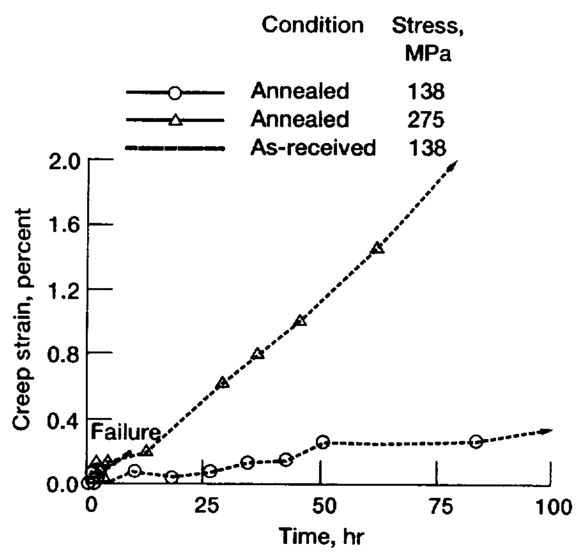

Figure 3.—Tensile creep curves for as-received and annealed Nextel 610 fibers at creep temperature of 1090 °C (arrows indicate interrupted tests).

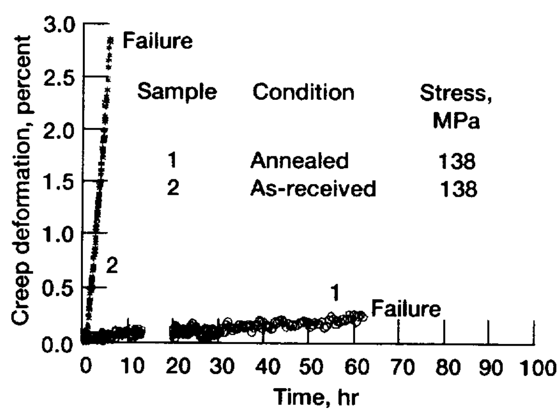

Figure 4.—Altex fiber creep response at 1090 °C as a function of annealing at 1100 °C for 100 hr.

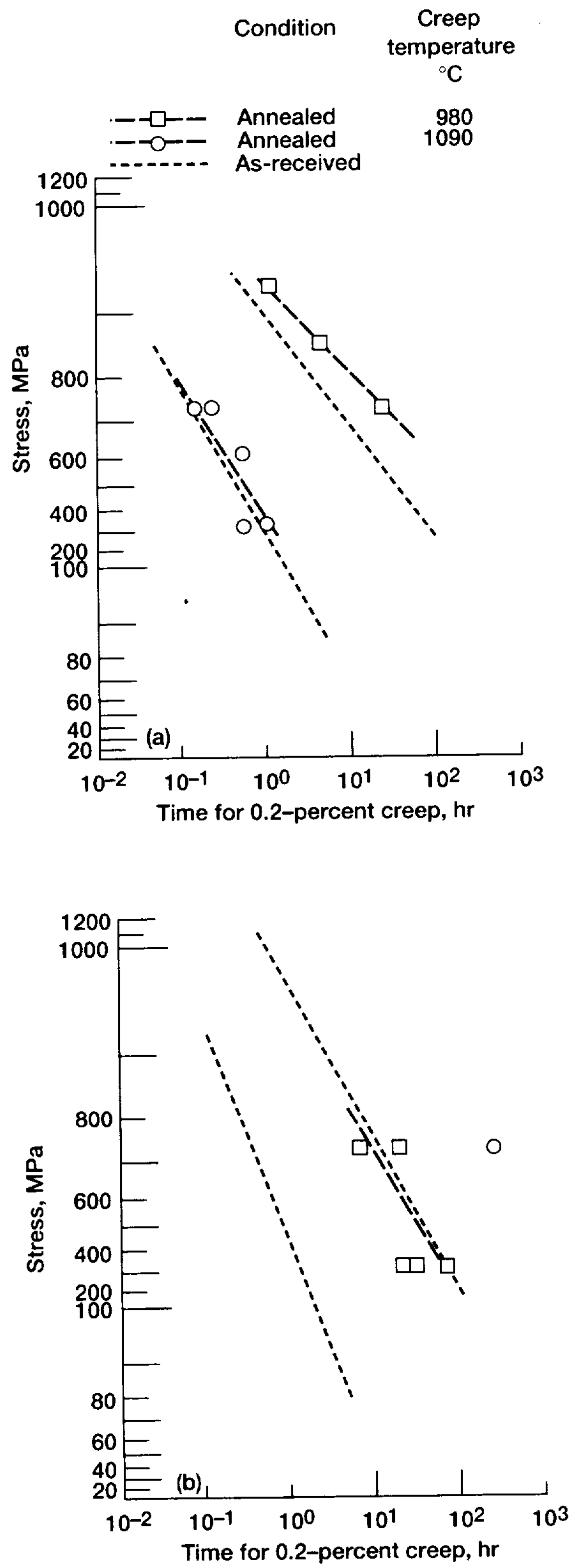

Figure 5.—Effects of annealing on 0.2-percent creep strength. (a) Nextel 610. (b) Altex.

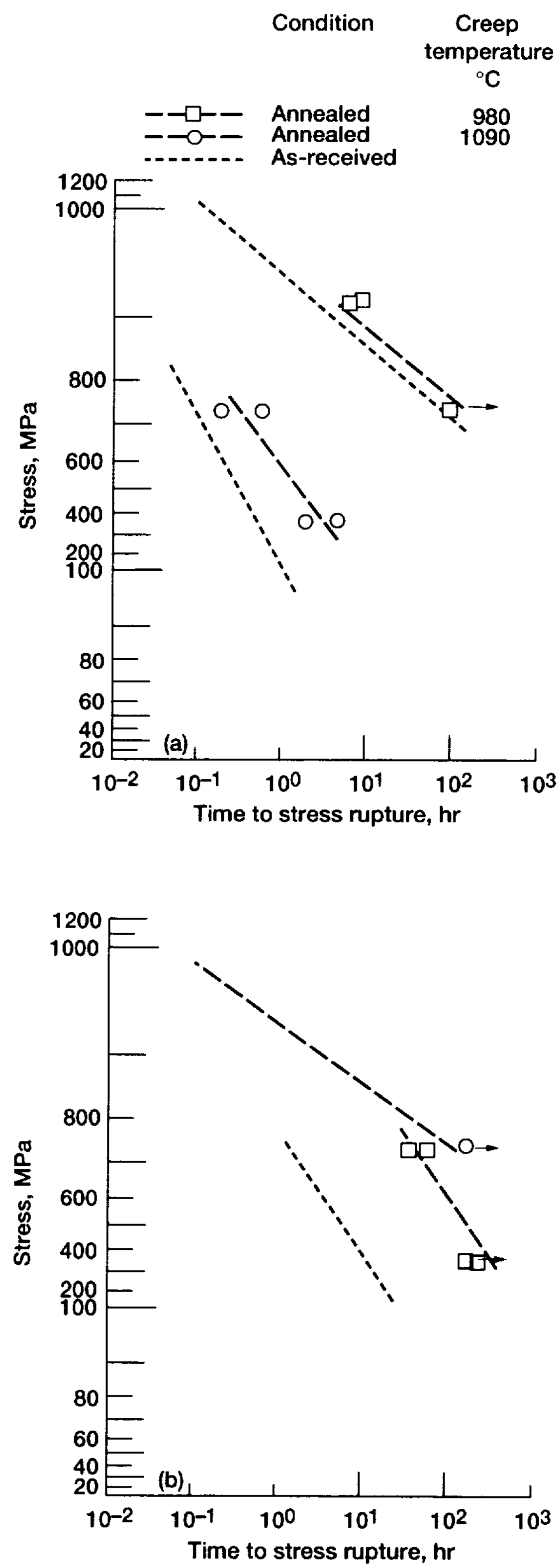

Figure 6.—Annealing increases rupture time of as-received oxide fibers (arrows indicate interrupted tests). (a) Nextel 610. (b) Altex.

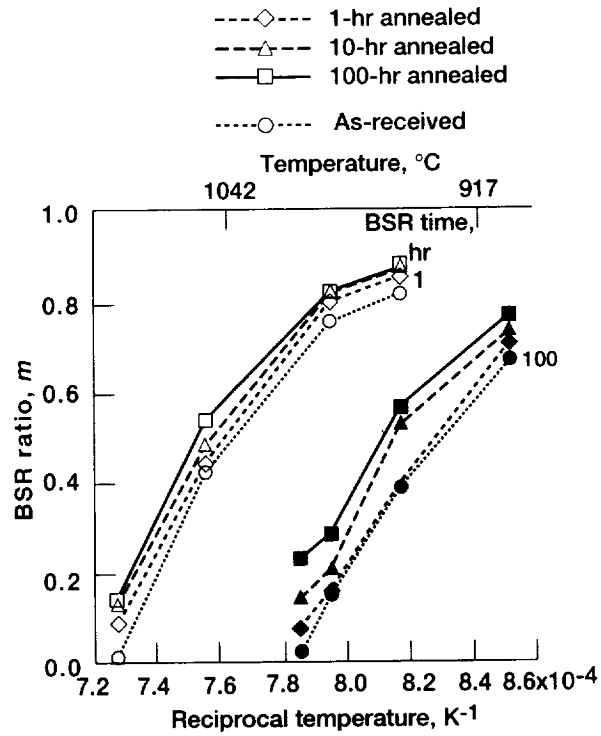

Figure 7.—BSR test results (*m*-ratio data) for Nextel 610 fiber after 1-, 10-, and 100-hr anneals at 1100 °C in air indicate an increase in creep resistance with annealing time.

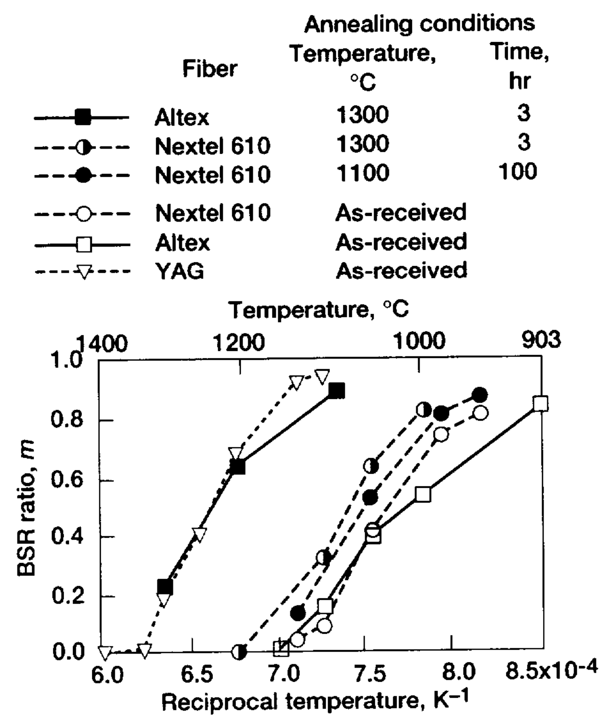

Figure 8.—Results of 1-hr BSR test on polycrystalline oxide fibers in their as-received condition and on Nextel 610 and Altex fibers before and after annealing at 1100 and 1300 °C [8,9].

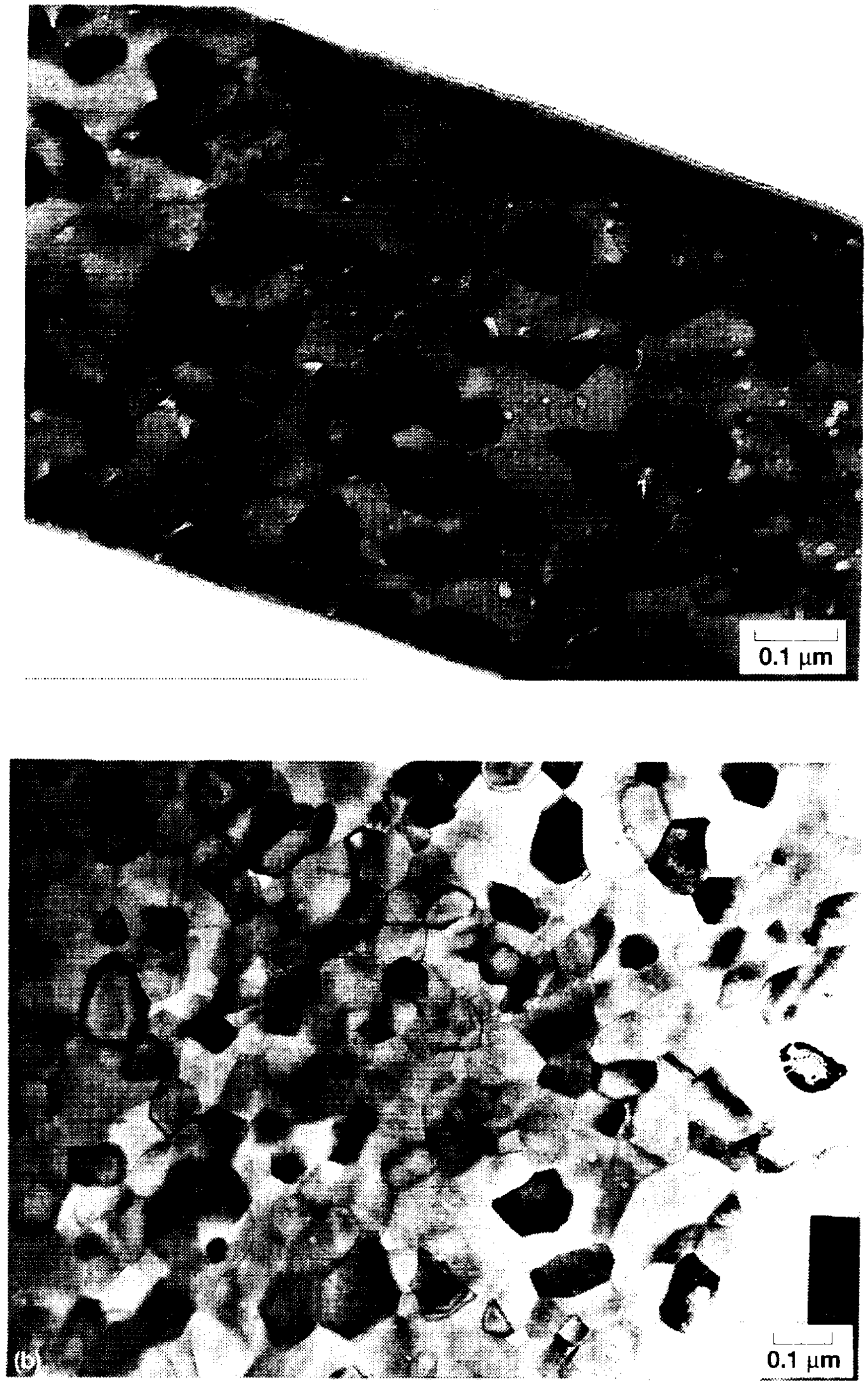

Figure 9.—Microstructure of Nextel 610 illustrates very little change in grain size after 100-hr anneal at 1100 °C. (a) As-received. (b) Annealed.

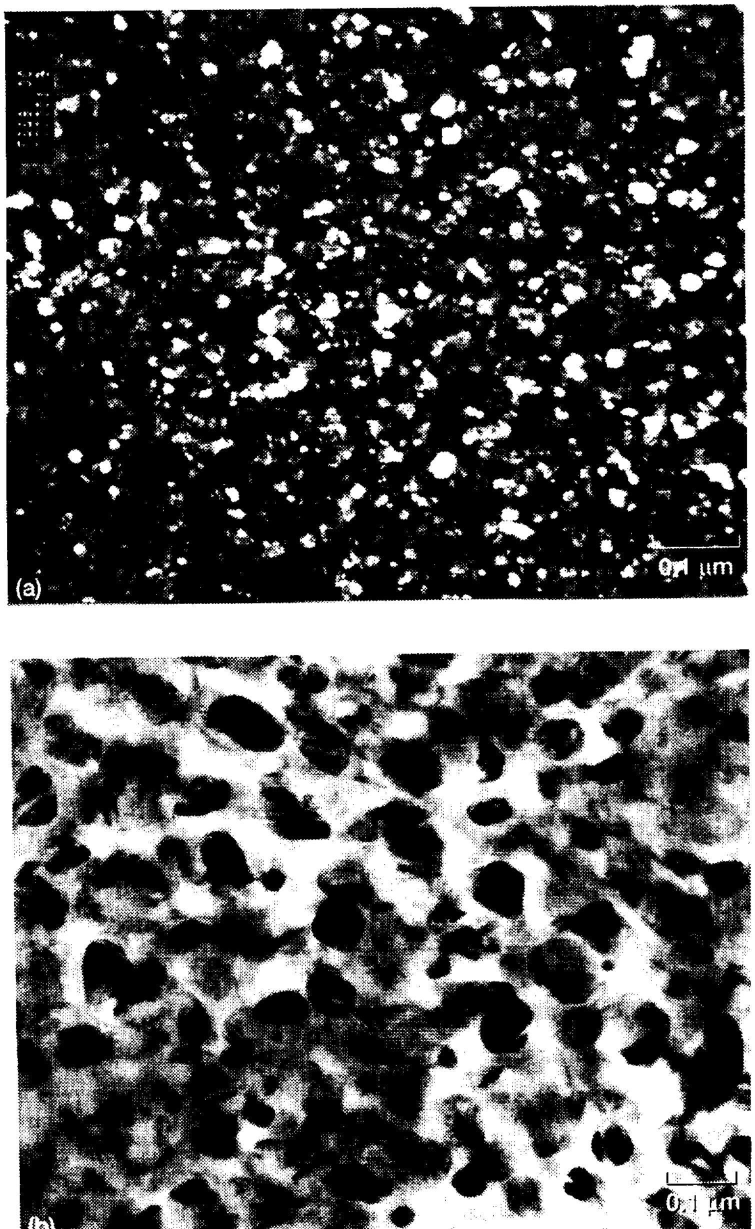

Figure 10.—Altex fiber grain size increased fourfold after 3-hr exposure at 1300 °C. (a) As-received grain size = 0.02 μm. (b) After annealing at 1300 °C.

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13. ABSTRACT *(Maximum 200 words)*

Continuous-length polycrystalline aluminum-oxide-based fibers are being considered as reinforcements for advanced high-temperature composite materials. For these fine-grained fibers, basic issues arise concerning grain growth and microstructural instability during composite fabrication and the resulting effects on the fiber's thermo-mechanical properties. To examine these issues, commercially available Nextel 610 (alumina) and Altex (alumina-silica) fibers were annealed at 1100 and 1300 °C for up to 100 hr in air. Changes in fiber microstructure, fiber tensile creep, stress rupture, and bend stress relaxation (BSR) that occurred with annealing were then determined. BSR tests were also used to compare as-received and annealed fibers to other polycrystalline oxide fibers. Annealing was shown to have a significant effect, particularly on the Altex fiber, and caused it to have increased creep resistance.

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