

Creep mechanisms and microstructure evolution of NextelTM 610 fiber in air and steam[☆]

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Abstract

Creep rates of NextelTM 610 alumina fibers were measured at 1100 °C and 100–500 MPa in air and steam. Steam increased creep rates and reduced fiber lifetimes. Fiber microstructures were characterized by TEM. The small amounts of grain growth, fiber-axis grain elongation, and pore growth that occur during creep were quantified. To separate the effects of stress and temperature on microstructural evolution, grain growth and elongation were also quantified for fibers heat-treated for 1–100 h in air at 1100–1500 °C. Grain growth laws were determined. The contributions of pore growth and grain elongation to creep strain were quantified. Grain elongation accounts for a large fraction of the strain during creep in air, but little in steam. Pore growth was more pronounced in steam, but does not create significant creep strain. Creep and failure mechanisms consistent with the observed microstructural changes are discussed.

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1. Introduction

Oxide–oxide ceramic matrix composites (CMCs) are damage tolerant and oxidation resistant, and have good high-temperature mechanical properties in air.^{1–10} They are attractive in comparison to SiC–SiC CMCs when low tensile and interlaminar stress, long times in oxidizing environments at <1200 °C, low thermal conductivity, and low cost drive material selection. Creep rates can be temperature and/or stress limiting for CMCs,^{11,12} particularly for oxide–oxide CMCs. Fiber creep almost always governs creep of CMCs in tension. CMC applications often involve exposure to water, either as a combustion product or as atmospheric humidity. Tensile creep rates of oxide–oxide CMCs with NextelTM 610 or NextelTM 720 fibers can be more than an order of magnitude higher in steam than in air.^{13–22} Recently, an order of magnitude enhancement of creep by steam was observed for NextelTM

610 and NextelTM 720 fibers alone.^{23–25} The mechanisms that promote higher creep rates in steam are therefore of great interest, both for modeling CMC mechanical behavior and for developing new materials systems that are more creep resistant.

Many studies of tensile creep of various alumina-based fibers in air have been conducted,^{26–34} and a lot of research has been done on compressive creep of polycrystalline alumina, with and without various impurities, in air.^{35–48} Most of the work up to 2003 has been thoroughly reviewed.⁴⁷ Only a few studies have been done in environments containing water.^{49–51} These studies report higher creep rates in steam than in air, which is sometimes referred to as hydrolytic weakening. In silicate minerals hydrolytic weakening is well known, and mechanisms involving climb-controlled dislocation creep or enhancement of diffusion-creep have been proposed.⁵²

Creep measurements on polycrystalline alumina fibers usually differ from those on bulk polycrystalline alumina in several ways. Fiber measurements are typically done in tension, while bulk studies are in compression. Fiber grain sizes are usually less than 0.1 μm,^{25,53,54} but bulk polycrystalline grain sizes are usually greater than 1 μm. Fiber diameters are less than 15 μm, so equilibration with the test environment at relatively low temperatures can be achieved faster than in bulk material.

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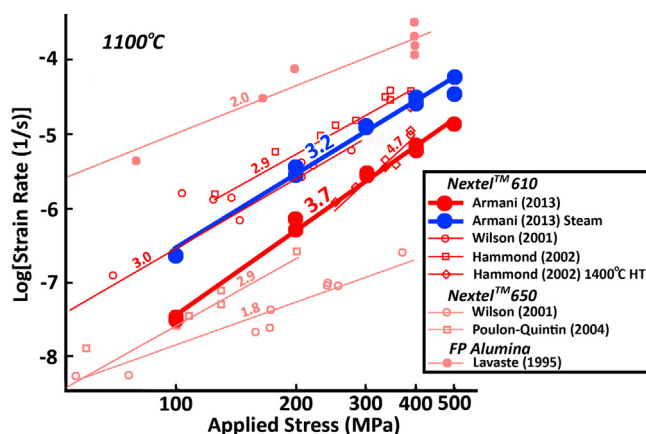


Fig. 1. Steady-state strain rate vs. applied stress of Nextel™ 610 fibers in air and steam. Other alumina fibers are included for comparison.

Nextel™ 610 is a high-purity alumina fiber (>99% Al_2O_3) manufactured by 3M™ Corporation (Minneapolis, MN). The fiber tow contains 400 filaments with an average filament diameter of 12 μm . Trace Fe and Si are added for α -alumina nucleation and grain growth suppression, respectively. Fiber properties, microstructure, and composition were reported elsewhere.^{28,33,54–56} In previous on Nextel™ 610, creep tests were done at 1100 °C in air and in steam for stresses from 100 to 500 MPa.^{23,24} The creep rates in steam were nearly one order of magnitude higher than those in air, and creep lifetimes were lower than those in air (Fig. 1). Flow stress exponents of $n \approx 3.2$ and 3.7 were found in air and in steam, respectively. Tensile strengths were lower at higher loading rates in steam, suggesting operation of an environmentally assisted subcritical

crack growth mechanism. The experiments, creep rates, and microstructural parameters are listed in Table 1.

The previous study of the effects of steam on creep of Nextel™ 610 fiber tows is expanded to include extensive TEM characterization and quantification of microstructure evolution during creep.^{23,24} Small amounts of grain growth, fiber-axis grain elongation, and pore growth are quantified. Some microstructural changes are subtle and require a large amount of data for quantification. To separate the effects of temperature and stress on microstructure evolution, grain growth and fiber-axis grain elongation are also characterized and quantified after heat-treatment under no load, without creep. Creep mechanisms consistent with creep test data and microstructural evolution are discussed. Creep rates in air were lower than those reported elsewhere (Fig. 1).^{33,34} Possible reasons for these differences are discussed. A detailed description of fiber microstructure evolution in air with heat-treatment between 1100 °C and 1500 °C will be published in a forthcoming paper.

2. Experiments

Detailed descriptions of the tensile creep test apparatus and methods used for Nextel™ 610 in air and steam have previously been published.^{23,24} All tests were performed at 1100 °C with a 1 °C/s heat-up rate and a 45 min hold prior to testing. A fiber tow area of $4.52 \times 10^{-8} \text{ m}^2$ was used to calculate engineering stress for the 400 filament tows. Strain was calculated from the specimen elongation measurements using published methods.^{57–59} The cold grip method was used, so fiber elongation was measured outside the furnace. The total fiber elongation was the sum of contributions from different temperature zones: the hot zone with a uniform temperature of 1100 °C, the temperature gradient

Table 1
Summary of creep-rupture results for Nextel™ 610 fiber tows at 1100 °C in laboratory air and in steam.

Environment	Creep stress (MPa)	Creep strain – ϵ (%)	Steady-state creep rate (10^{-8} s^{-1})	Time to rupture (h)	$\log[X \pm \delta]$ (nm)]	$\log[\text{pore diameter} \pm \text{SD}]$ (nm)]	Pore volume (%)	Grain elongation: $\epsilon - \epsilon_0$ (%)	Grain elongation ϵ_C (%)
As received	δ	δ	δ	δ	1.87 ± 0.15	1.06 ± 0.25	2.1	12	δ
Air	100	1.8 ^a	3.6	>100	1.91 ± 0.17	1.05 ± 0.21	1.1	19	6
Air	100	1.5 ^a	3.2	>100	1.91 ± 0.15	0.99 ± 0.27	1.5	13	2
Air	200	16.9	54.5	48.90	1.93 ± 0.17	1.05 ± 0.29	2.6	31	17
Air	200	14.2	74.2	31.60	1.89 ± 0.17	1.12 ± 0.26	3.1	17	8
Air	300	10.8	296.0	6.67	1.91 ± 0.15	1.05 ± 0.27	2.1	20	7
Air	300	7.9	318.0	4.08	1.89 ± 0.16	1.07 ± 0.26	2.2	21	9
Air	400	5.0	739.0	1.30	1.87 ± 0.16	1.05 ± 0.26	2.2	21	13
Air	400	4.2	643.0	1.33	1.88 ± 0.15	1.10 ± 0.26	2.9	16	6
Air	500	4.4	1430.0	0.55	–	–	–	–	–
Air	500	3.0	1400.0	0.42	–	–	–	–	–
Steam	100	9.9 ^a	22.3	>100	1.96 ± 0.14	1.04 ± 0.29	3.8	20	5
Steam	200	15.1	363.0	6.70	1.91 ± 0.16	1.11 ± 0.33	4.8	17	5
Steam	200	14.9	285.0	8.52	1.96 ± 0.15	1.13 ± 0.28	2.9	19	3
Steam	300	6.5	1330.0	0.92	1.91 ± 0.14	1.11 ± 0.24	2.1	7	-5
Steam	300	6.8	1240.0	0.95	1.91 ± 0.14	1.12 ± 0.25	3.3	14	3
Steam	400	3.8	3090.0	0.25	1.90 ± 0.13	1.11 ± 0.26	3.1	13	2
Steam	400	2.1	2480.0	0.20	1.85 ± 0.12	1.03 ± 0.28	2.4	7	1
Steam	500	3.2	5800.0	0.10	1.88 ± 0.14	1.15 ± 0.25	3.5	8	1
Steam	500	3.9	3400.0	0.23	1.90 ± 0.14	1.00 ± 0.28	3.1	17	2

^a Run-out, defined as 100 h at creep stress. Failure of specimen did not occur when the test was terminated.

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