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Tensile and creep performance of a novel mullite fiber at high temperatures

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ABSTRACT

The novel fiber CeraFib75 with a composition near to pure mullite was analyzed with respect to its potential for high-temperature applications. This mullite fiber free of glass phase was aimed to overcome the strength of commercial oxide fibers at high-temperatures. Tensile tests at room and high temperatures ranging from 900 to 1400 °C and creep tests were performed. Nextel™720, another crystalline mullite-alumina fiber, was tested as a reference. Microstructure and crystal phase analysis of the new fiber revealed mullite grains with traces of γ- and α-alumina in-between; it contains occasionally defects causing a reduced strength at room-temperature. Remarkably, at temperatures beyond 1200 °C, CeraFib75 presented a higher tensile strength than Nextel™720. During tensile tests at 1400 °C, an extended region of inelastic deformation was observed for CeraFib fibers only, which was related to a grain boundary sliding mechanism. Creep rates were of the same order of magnitude for both fibers.

1. Introduction

Advances in the aerospace field promoted a development of structural materials capable of withstanding mechanical loads at aggressive environments. Ceramic-matrix composites (CMC) proved to be good candidates for this field mainly because of their high strength, thermal and chemical stability, and considerable toughness [1]. Due to their thermo-mechanical resistance, early developments were initiated on systems based on either carbon or silicon carbide [2]. However, a crescent demand for applications at corrosive environments made these types of materials less suitable due to oxidation and consequent loss of mechanical properties [3]. More attention was then given to CMCs based on oxide ceramics [4], which are commonly categorized as all-oxide-CMCs.

Being stable oxides by nature, this class of material is resistant against oxidation induced degradation even at high temperatures. Therefore, they gained space on applications such as gas turbine combustors, heat shields and heat exchangers [5,6]. Nevertheless, the lower strength of oxide fibers, in comparison to non-oxide materials, limited their relevance in the past. The first attempt on producing oxide fibers is dated in the 70s with the production of fibers based on alumina–silica [7]. At that stage, oxide fibers presented two main problems related to low mechanical resistance at room temperature and low thermal stability. The development of all-oxide-CMCs was strongly driven in the 90s by the commercialization of fully crystalline oxide fibers [8]. Nowadays, the usage of oxide fibers is mainly related to the ones developed by the American company Minnesota Mining and Manufacturing (3M). With the trademark of Nextel 610, this 99% alumina fiber presents the highest tensile strength of available oxide fibers of around 3 GPa [9]. The high strength of this fiber is achieved by its fine microstructure of sub-micrometer α-alumina grains and the additions of dopants to reduce grain growth effects. In the further course, several other fibers were produced with the addition of mullite or zirconia phases to increase the thermal stability. Another fiber which is widely commercially used is the Nextel 720, which is an alumina–mullite fiber presenting a higher tensile strength and creep resistance than Nextel 610 at temperatures above 1100 °C. This better performance is credited to the morphology and size of the mullite grains [9], and makes Nextel 720 currently to be estimated as the preferred fiber for long-term applications.

Nonetheless, temperature is still a limiting factor for the appliance of oxide-CMCs. They are prone to degradation of their strength and embrittlement when subjected to high-thermal loads, which is normally associated with the degradation of the fibers [10–12]. Early studies by Wilson [13] showed that the

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aforementioned fibers present strength loss when tested at temperatures above 1000 °C. The strength retention of the fibers has also been studied by other authors, and the results obtained are somewhat conflicting. Deléglise [14] reported that Nextel 720 fibers tested at 1200 °C retained only 20% of its room temperature strength while Wilson [13] stated a strength retention of 80% at the same temperature. It is also possible to find creep studies in the literature showing that these fibers are also susceptible to creep at those temperatures [14–16]. Additionally, Schmücker [17,18] noticed in his works that the sub-micrometer fiber grains are prone to coarsening at high temperatures, and the phenomenon can be aggravated when the fibers are embedded in a matrix [19]. At this point, it is important to highlight that such temperatures can occur during the processing and application of oxide composites.

Hence, there is a need of development of oxide fibers capable of maintaining their mechanical strength at high-temperatures. In this matter, the German company CeraFib GmbH, based on research of the Institute of Textile Chemistry and Chemical Fibers (Denkendorf), is developing oxide fibers which are able to operate at higher temperatures [20]. This work aims to analyze the performance of the recently developed CeraFib 75 fiber when subjected to tensile loads at critical temperatures. This fiber presents a composition similar to pure mullite and aims applications at high temperatures. For that, several tensile tests at different temperatures were performed. The authors also take into account that the results found in the literature for Nextel 720 can be rather different depending on the source, and therefore, the same characterization methodology was applied to test this commercial fiber for comparison. Additional tests were conducted at the temperature of 1400 °C, at which the fibers started presenting an unexpected non-linear deformation. Creep properties were also analyzed and the deformation phenomena were interpreted with respect to the results obtained from the analysis of microstructure and phase content.

2. Experimental

2.1. Materials

The main object of the studies in this work is the mullite fiber CeraFib 75. This small diameter fiber (10–12 μm) produced by dry-spinning has an initial composition close to stoichiometric 3/2 mullite (72 wt% alumina–28 wt% silica). During processing, the silica content is decreased due to the formation of volatile Si species, which leads to an end composition of 75–25 wt%. The final microstructure is expected to be of mullite grains with traces of smaller alumina grains dispersed. This represents a great advance since the Si is stable in the alumina-rich mullite phase and not free as a glass phase. For comparison, another commercial fiber was tested. Nextel 720 is an 85–15 wt% alumina–silica fiber produced by a *sol–gel* route. Its microstructure is known to be of a mosaic of mullite grains with elongated alumina grains [14]. It is important to highlight that the fibers do not present the same chemical composition, but the comparison is justified since both are the only fully crystalline mullite fibers available. Table 1 summarizes the main information regarding the fibers, and presents the density measurement here performed according to norm ASTM D 3800.

Table 1
Summary of tested fibers.

Fiber	Manufacturer	Composition (wt%)[a]	Density (g/cm^3)
Nextel 720	3M	85 Al_2O_3–15 SiO_2	3.17 ± 0.09
CeraFib 75	CeraFib	75 Al_2O_3–25 SiO_2	3.26 ± 0.19

[a] Information by manufacturer.

2.2. Characterization methods

Before the mechanical tests, other characterization methods were performed in regard to microstructure and phase investigation. X-ray diffraction (XRD) analysis was conducted to identify and quantify the phases present on each fiber. For that, fibers were crushed into a powder and then analyzed using a SEIFFERT XRD 3003 research edition XRD.

Grain size measurement was realized by the analysis of the microstructure using the line-intercept method with the help of the image analysis software Lince (TU Darmstadt, Germany). To do so, fibers were embedded in epoxy resin, and then ground, polished, and followed by thermal extraction of the resin at 800 °C. The microstructure was revealed by a thermal etching process at 1300 °C for 40 min. Finally, pictures of the microstructure were taken from five different fibers using a scanning electron microscope (SEM), Zeiss SUPRA 40, with an acceleration voltage of 0.5 kV.

2.3. Mechanical tests

Single filament tensile tests were performed according to norms DIN EN 1007-4 and 1007-6. Sample preparation was done with surgical gloves to avoid contaminations. Various testing temperatures were used starting at room temperature and then ranging from 900 to 1400 °C. The fibers were tested in a tensile testing machine equipped with a 1 N load cell, model Interface ULC-1N-535, and a linear variable differential transformer (LVDT) sensor of ±1 mm. A two SiC heating element oven was used for the tests at high temperature. System compliance was measured prior testing. 30 samples were tested with a traveling speed of 1 mm/min until failure for statistical meaning. The cross-section area of every fiber tested was determined after testing with an optical microscope, SENSOFAR PLμ 2300. Thus, the stress–strain relation of each individual fiber could be determined taking into account the gauge length of 25 mm.

Creep tests were also carried out to determine the creep parameters at the mentioned temperatures. The stress and temperature effects on creep were assumed to be described by the standard power law creep equation:

$$\dot{\varepsilon} = A_0 \sigma^n \exp\left(\frac{-Q}{RT}\right)$$

where $\dot{\varepsilon}$ is the steady-state creep rate, A_0 is a constant, σ is the applied stress, n is the stress exponent, Q is the activation energy, R is the universal gas constant and T is the absolute temperature. The stress exponent was determined from tests done at 1200 °C with three different loads applied. The determination of the apparent activation energy for creep was performed at temperatures of 1100–1300 °C and a constant stress of 150 MPa. For the later test, the diameter measurement was carried out before the test in order to assure the applied stress. A total of two specimens were tested for each testing condition. Tests were conducted until the fiber failure or until the run-out time of 100 h was achieved. A dead load system was used to achieve a better control of the applied load, and the same oven used for tensile tests was applied for the creep tests. The effective gauge length was calculated based on the relation reported earlier [21] and determined as 11.0 and 11.17 mm for CeraFib 75 and Nextel 720, respectively.

3. Results

3.1. Fibers overview

The thermal etch procedure was proved to be efficient to reveal the microstructure of CeraFib 75 fibers. SEM pictures of the