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On the dimensional analysis for the creep rate prediction of ceramic fibers

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A B S T R A C T

A novel semi-empirical equation for the steady-state creep rate description of ceramic fibers is here proposed. Dimensional analysis was applied to identify the relationship between the variables related to the creep rate. Besides the variables known from the Arrhenius creep equation, a temperature factor that accounts for changes with the temperature was also taken into consideration. The resultant equation shows that the creep rate is proportional to the temperature, applied stress and two material constants, as well as inversely proportional to the fiber grain size. The two material constants k and C described in the equation can be easily determined by creep tests at different temperatures under the same applied stress. Therefore, this equation can be more efficiently applied to predict the creep rate of ceramic fibers under different applied stresses and initial grain sizes. To confirm this, the proposed equation was used to fit the creep rate data set of two ceramic fibers, Nextel 720 and CeraFib 75. In summary, the equation provides a good fit (adjusted R-squared up to 0.97) and prediction for the experimental data of tests with different temperatures, applied stresses and initial grain sizes.

1. Introduction

Ceramic matrix composites (CMCs) have gained more and more attention over the last decades. The concept of enhancing the fracture toughness of ceramics by reinforcing them with ceramic fibers relies on several crack deflection mechanisms [1]. CMCs show a pseudo-plastic mechanical behavior, as well as other interesting properties of ceramic materials like high strength and high thermal and chemical stability. Hence, CMCs have gained space in several applications that require high-strength structural materials at elevated temperatures. From these, it can be listed: components for gas turbines, heat shield of space vehicles and hot gas filters. Considering the temperatures achieved in these applications, the creep performance of CMCs is of particular importance. In this sense, several authors have studied the creep deformation of different CMC systems above 1273 K (1000 °C) [2–5].

It is generally agreed that when the load is applied in the direction of the fibers, the creep resistance of the composite will depend mostly on the properties of the fibers [3,4,6]. Here lies a problem as most of the polycrystalline fibers rely on small grains to achieve high strength, and the creep resistance is proportional to the grain size [7]. This is even more preoccupying for oxide fibers, since they are less creep resistant than non-oxide materials [8]. Therefore, several works on the creep performance of the commercially available ceramic fibers can also be found in the literature [9–13]. Most of these works are based on empirical research. Normally, the primary objective is to determine the steady-state creep rate under certain test conditions. The results are then fitted with creep equations and models proposed for bulky ceramics and other materials. The most used is the Arrhenius creep rate equation:

$$\dot{\varepsilon} = \frac{ADGb}{k_B\theta}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^n;\ D = D_0 \exp\left(\frac{-Q}{R\theta}\right) \tag{1}$$

where $\dot{\varepsilon}$ is the steady-state creep strain rate, A is a dimensionless constant, D is the diffusion coefficient, G is the shear modulus, b is the Burger's vector, k_B is the Boltzmann's constant, θ is the testing temperature, d is the grain size, p is the inverse grain size exponent, σ is the applied stress, n is the creep stress exponent, D_0 is the maximal diffusion coefficient, Q is the creep activation energy and R is the universal gas constant.

In summary, creep tests with different applied stresses and temperatures are used to determine n and Q, respectively. The Arrhenius equation provides a good fit for the creep data, but it can be rarely used for the prediction of the fiber creep rate under other conditions. This goes by the fact that most of the material constants/properties in the equation cannot be accurately determined with experiments. For instance, the activation energy Q is normally determined by the plot $\ln(\dot{\varepsilon})$ *vs.* 1/θ, considering tests at different temperatures and the same applied stress. By doing so, the resultant coefficient should be regarded only as

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an apparent activation energy, since it does not consider the term $1/k_B\theta$ and the other variations of the material properties due to the different test temperatures [3].

Given the problematic above, the objective of this work is to apply dimensional analysis to formulate an equation to predict the creep rate of ceramic fibers. Dimensional analysis is a very effective tool to indicate the relevant data and how they are related. This analysis consists on mathematically expressing a relationship between the variables involved in a physical situation [14–16]. The resulting expression can be then used to effectively obtain any unknown factor from experimental results. In order to validate the proposed equation, creep data of two commercial ceramic fibers, Nextel 720 and CeraFib 75, were used. The data consisted of tests with creep stresses of 100–300 MPa and temperatures of 1373–1573 K (1100–1300 °C) [11]. In addition, the experimental data of fibers with different initial grain sizes were fitted to analyze the effect of the microstructure on the creep rate [17].

2. Dimensional analysis

It is a known fact that all engineering quantities can be defined in terms of the basic dimensions, *e.g.* mass (M), length (L), time (T), temperature (θ), mole (N). Hence, any equation describing a physical situation must be dimensionally homogeneous. In order to apply dimensional analysis to the creep of ceramic fibers, the main parameters involved should be first listed. Most of these parameters were already listed above. Note that k_B is not included since it is equal to R divided by the Avogadro constant. Here, it is also introduced a temperature factor θ_0 to account for the changes with the temperature. In this sense, the strain rate $\dot{\varepsilon}$ can be expressed as a function of these physical properties as follows:

$$\dot{\varepsilon} = f(\sigma, G, \theta, \theta_0, Q, R, d, b, D_0) \tag{2}$$

Normally, Buckingham π Theorem gives a good generalized strategy for achieving a solution for the problem [14]. According to this theorem, Eq. (2) can be rewritten as a set of dimensionless groups:

$$\varphi(\pi_1, \pi_2, \cdots, \pi_{m-n}) = 0 \tag{3}$$

where each π group is a function of the governing (repeating) variables plus one of the remaining variables. In other words, the repeating variables are those that appear in one or most of the π groups, which influences directly the strain rate. For the present case, the chosen repeating variables are the following: Q, R, θ_0, G, b, D_0. It is worth mentioning that the repeating variables may contain one or all the independent dimensions involved (M, L, T, θ, N).

The number of π groups involved can be determined from the following equation:

$$number \text{ of } \pi \text{ } groups = m - n \tag{4}$$

where m is the number of variables of the creep problem, in this case 10, and n is the number of independent dimensions, five. Hence, the number of π groups is equal to five, so Eq. (3) becomes:

$$\varphi(\pi_1, \pi_2, \pi_3, \pi_4, \pi_5) = 0 \tag{5}$$

It is now possible to form the five π groups given in Eq. (5). Needless to say that all π groups are dimensionless, *i.e.*, they have dimensions of $M^\circ L^\circ T^\circ \theta^\circ N^\circ$, and form dimensional homogeneity. Thus, to determine the variables involved in the first π_1 group, it can be proceeded as follows:

$$\pi_1 = \varphi_1(\dot{\varepsilon}, Q, R, \theta_0, G, b, D_0) \tag{6}$$

From the dimensional homogeneity, Eq. (6) can be expressed as:

$$M^0L^0T^0\theta^0N^0 = [T^{-1}][ML^2T^{-2}N^{-1}]^u[ML^2T^{-2}\theta^{-1}N^{-1}]^v[\theta]^w[ML^{-1}T^{-2}]^x[L]^y[L^2T^{-1}]^z \tag{7}$$

By equating the dimensions of the above equation from both sides, the exponents can be found as: $u = 0$, $v = 0$, $w = 0$, $x = 0$, $y = 2$, $z = -1$. With these exponents, π_1 can be rewritten as:

$$\pi_1 = \frac{\dot{\varepsilon}b^2}{D_0} \tag{8}$$

In a similar manner, π_2 can be expressed as:

$$\pi_2 = \varphi_2(\sigma, Q, R, \theta_0, G, b, D_0) \tag{9}$$

Repeating the same procedure, then π_2 is given by:

$$\pi_2 = \frac{\sigma Q}{R\theta_0 G} \tag{10}$$

By similarity of the dimensional factors, the other groups π_3, π_4, π_5 can be derived as dimensionless functions:

$$\pi_3 = \frac{dQ}{bR\theta_0}, \ \pi_4 = \frac{\theta}{\theta_0}, \ \pi_5 = \frac{R\theta}{Q} \tag{11}$$

It must be pointed out that the dimensional analysis permits a number of manipulations, as long as the π groups remain dimensionless. In general, the defining equation could be expressed, for example, as:

$$\varphi(\pi_1, \pi_2, (\pi_3)^i, \cdots, \exp(\pi_{m-n})) = 0 \tag{12}$$

Hence, π_5 can be expressed as:

$$\pi_5 = \frac{R\theta}{Q} \rightarrow \exp(C\theta) \tag{13}$$

where C is a materials constant that is proportional to R/Q.

By additional manipulation of the π groups, Eq. (5) becomes:

$$\frac{\dot{\varepsilon}b^2}{D_0} = \left(\frac{\sigma Q}{R\theta_0 G}\right)^3 \left(\frac{dQ}{bR\theta_0}\right)^{-3} \left(\frac{\theta}{\theta_0}\right) \exp(C\theta) \tag{14}$$

Reorganizing the terms of the equation above, it is then possible to obtain the following relation:

$$\dot{\varepsilon} = \frac{D_0 b}{G^3\theta_0} \frac{\sigma^3}{d^3} \theta \exp(C\theta) \tag{15}$$

The equation can be further simplified by considering a constant $k = \frac{D_0 b}{G^3\theta_0}$, which can be regarded as a material constant. The final equation for the creep rate is then:

$$\dot{\varepsilon} = k\frac{\sigma^3\theta}{d^3}\exp(C\theta) \tag{16}$$

On close examination of the above expression, it can be seen that the creep rate is a proportional function of the stress (σ), temperature (θ) and properties of the material (k and C). On the other hand, it is inversely proportional to the grain size (d). In fact, these findings are coherent with previous experimental findings on ceramic fibers [11,17]. Furthermore, the equation can also be further adjusted using dimensional analysis to fit other types of ceramic materials.

3. Data fitting

Eq. (16) was used to fit the creep rate data of two ceramic fibers tested under various conditions. The selected fibers were a mullite-alumina fiber, Nextel 720, and a mullite fiber, CeraFib 75. Nextel 720 is a fiber that consists of several slightly miss-oriented mullite grains of 160 nm [11], forming 500 nm mullite mosaics with some smaller and elongated α-alumina grains in between [18]. As a result, Nextel 720 shows the highest creep resistance amongst the currently available polycrystalline oxide fibers [9]. On the other hand, CeraFib 75 has a microstructure of several equiaxial mullite grains of around 175 nm and traces of γ-alumina [17].

The fiber filament tests were performed according to the standard DIN EN 15365. The testing rig consisted of a dead-load system for the control of the applied load and a two SiC heating element oven. For the tests, single filaments were attached to the testing rig and heated up to the testing temperature with a heating rate of 1 K/s. After achieving the

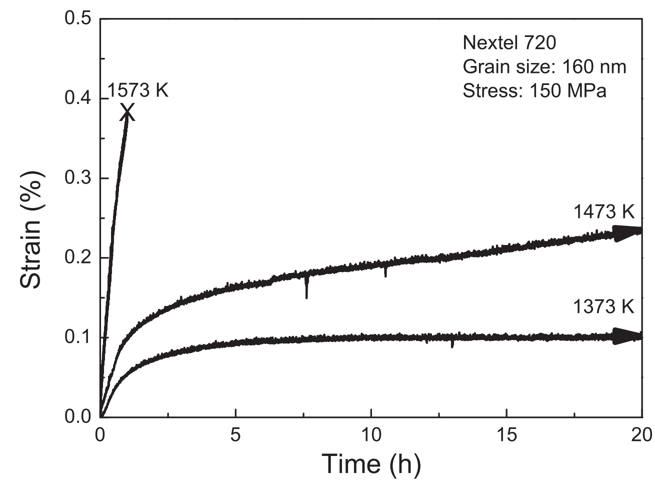

Fig. 1. Examples of creep deformation *vs.* time curves for Nextel 720 under 150 MPa at temperatures of 1373–1573 K. Arrows indicate that the sample did not fail at the given time.

testing temperature, the load, *i.e.*, weight attached to the lower end of the fiber, was slowly released. The tests were performed until the fiber failed or until the run-out time of 50 h was achieved. The test conditions (temperature and stress) were chosen in accordance to ranges relevant to the application of the ceramic fibers. Fig. 1 shows examples of creep curves for Nextel 720 tested at different temperatures. Note that the graphs are plotted until 20 h for better visualization of the test at 1573 K. For the analysis, the steady-state creep rate was measured. After the test, samples were preserved for the measurement of the cross-section area and calculation of applied stress. Further details of the tests were published in Almeida et al. [11,17].

The two material constants of Eq. (16), k and C, were determined from the creep tests performed at the temperatures of 1373–1573 K with the constant creep stress of 150 MPa, as well as considering the initial fiber grain size. Fig. 2a shows the creep data for Nextel 720 under such conditions. For each temperature, two tests were performed. k and C were obtained by performing a best fit of the experimental data using Eq. (16), and resulted as: $k = 1.81 \times 10^{-39}$ $nm^3/(MPa^3\ K\,s)$ and $C = 0.0432\ K^{-1}$. To verify the quality of the fit, the adjusted R-squared (R^2) was also calculated. In this case, R^2 of 0.967 was measured, which is considerably high for a non-linear fit.

Using the parameters obtained by the fitting of the tests at different temperatures (Fig. 2a), the creep rates of Nextel 720 at 1473 K and stresses of 100–330 MPa were calculated and compared to the actual creep data under similar conditions, cf. Fig. 2b. As it can be seen, Eq. (16) predicts the data fairly good using the parameters obtained before. The only significant outliners are found with the creep tests under 287 MPa. Thus, the fit showed lower R^2 of 0.791. Nevertheless, this discrepancy can be attributed to differences between the different fibers tested, as seen by the scatter of the experimental data with the same applied stress.

The same methodology was applied for the data of CeraFib 75. In this sense, Fig. 3a shows the determination of the material constants using the data of creep tests at different temperatures. Again, two tests were performed for each temperature. The calculated material constants were: $k = 2.87 \times 10^{-47}$ $nm^3/(MPa^3\ K\,s)$ and $C = 0.0483\ K^{-1}$. Fig. 3b shows the prediction, using the aforementioned parameters, of the data obtained with different applied stresses at 1473 K. Once more, Eq. (16) proves to be a good fit for the creep data at different temperatures and to predict the creep rate under different conditions. R^2 of 0.960 and 0.901 were measured for the fit regarding different temperatures and stresses, respectively.

The calculated constants can also give interesting input regarding the creep resistance of the tested materials. The effect of the testing

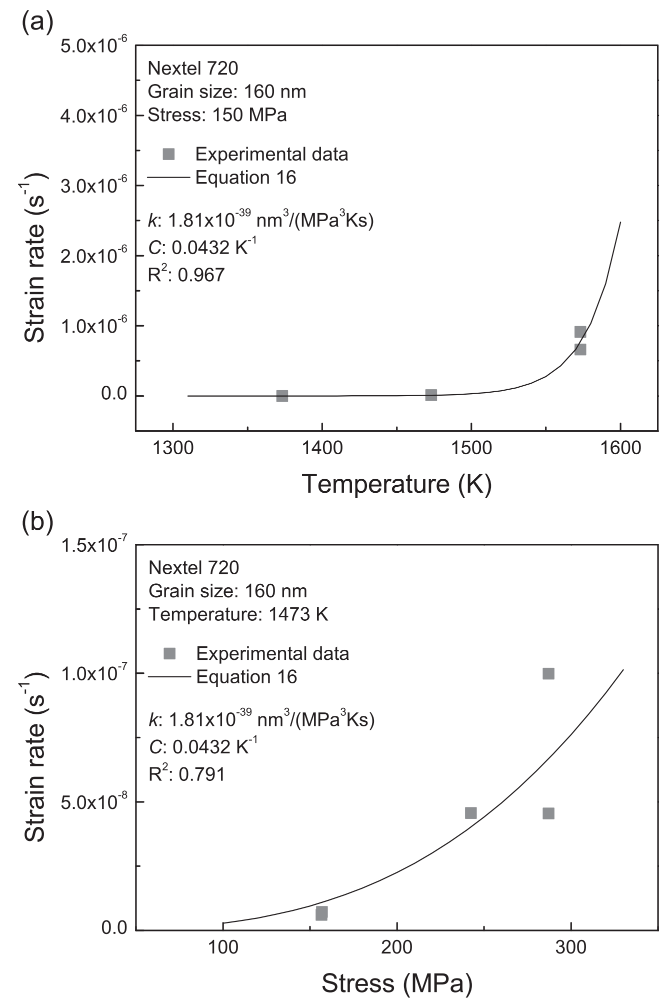

Fig. 2. Fitting of Nextel 720 creep rates using Eq. (16): (a) determination of material constants using the creep rate data of tests at different temperatures, (b) prediction of the creep rates of tests under different stresses. Creep rate data [11] were obtained from tests on as-received Nextel 720 fibers.

temperature on the creep rate is related to the constant C. In other words, the higher value of C from the CeraFib 75 fibers means a higher sensitivity of the fiber to the testing temperature. The constant k is rather complex, given by the fact that it depends on several other material constants. Therefore, a proper meaning for this constant cannot be defined, apart from adjusting the creep rate under the given conditions. However, it should also be highlighted that the "impact" of k on the creep rate will also depend on its product with $\exp(C\theta)$. For instance, the product of both terms for CeraFib 75 at 1473 K is 2.29×10^{-11} $nm^3/(MPa^3\ K\,s)$, whereas for Nextel 720 is only 7.89×10^{-12} $nm^3/(MPa^3\ K\,s)$. Hence, CeraFib 75 shows higher creep rates than Nextel 720 with similar grain size under such conditions, although the difference gets smaller at lower temperatures and bigger at higher temperatures.

Naturally, the creep rate will also depend on the microstructure of the fiber as bigger grains have a higher creep resistance. Here it should be highlighted that the constants C and k do not depend on the grain size of the fibers. Therefore, the calculated constants for CeraFib 75 (Fig. 3a) can also be used for the creep data of the same material with different grain sizes. To attest that, the creep data of heat treated CeraFib 75 fibers were also analyzed. Prior to the creep tests, heat treatments of 25 h at temperatures ranging from 1473 to 1673 K (1200–1400 °C) were done on the fibers, which resulted in grain growth [17]. Fig. 4a presents the creep rate data of as-received and heat treated CeraFib 75 fibers, showing different grain sizes, tested at 1473 K and

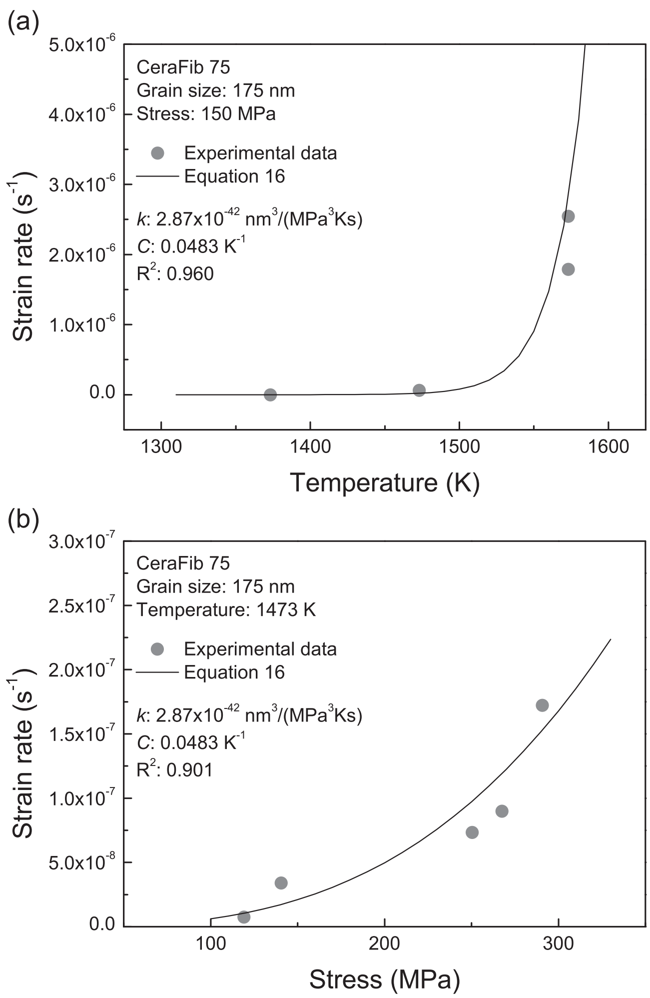

Fig. 3. Fitting of CeraFib 75 creep rates using Eq. (16): (a) determination of material constants using the creep rate data of tests at different temperatures, (b) prediction of the creep rates of tests under different stresses. Creep rate data [11] obtained from tests on as-received CeraFib 75 fibers.

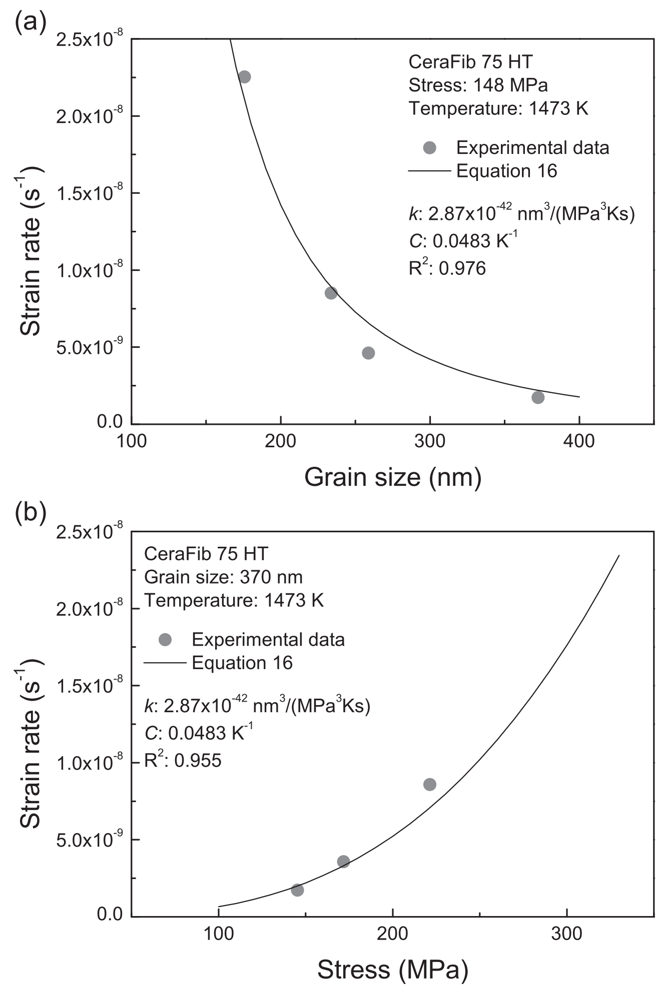

Fig. 4. Fitting of CeraFib 75 creep rates using Eq. (16): (a) prediction of the creep rates of fibers with different initial grain size, (b) prediction of the creep rates of tests under different stresses. Creep rate data [17] obtained from tests on heat-treated CeraFib 75 fibers.

creep stress of 148 MPa. For the plot, the average grain size was used; although the scatter on the measured grain size was not taken into account. In addition, Fig. 4b shows the creep data of CeraFib 75 heat treated at 1673 K (1400 °C), resulting in 370 nm mullite grains, tested at 1473 K under different creep stresses. In both cases, Eq. (16) presents a good prediction of the experimental data using the constants previously determined. Very high R^2 of 0.976 and 0.955 were measured for the fits considering fibers with different grain sizes and applied stresses, respectively.

4. Conclusions

Using dimensional analysis, a semi-empirical equation for the creep rate of ceramic fibers is here proposed: $\dot{\varepsilon} = k\frac{\sigma^3\theta}{d^3}\exp(C\theta)$. This metrology proved to be a very useful tool to identify the relationship between the variables related to the creep of ceramic fibers. In the end, an equation similar to the Arrhenius equation was found. Still, the proposed equation is more simple as it relates only two material constants, k and C, with the other testing variables: temperature, creep stress and material grain size. The constant C can be associated with the material sensitivity to the testing temperature. On the other hand, k cannot be properly interpreted since it is a more complex constant that will depend on the material's diffusion coefficient, Burger's vector, shear modulus and temperature factor θ_0. Nevertheless, both constants can be easily calculated with creep tests at different temperatures and constant stress. Therefore, the proposed equation is very useful for the calculation/prediction of the creep rate under other conditions, *e.g.*, different applied stresses or different fiber grain sizes.

The resultant equation was then used to fit the creep data of two as-received ceramic fibers, Nextel 720 and CeraFib 75, tested under different temperature and stress conditions. In general, CeraFib 75 showed lower creep resistance and higher sensitivity to the testing temperature. The material constants were determined from the creep tests at different temperatures, and used to calculate the creep rate under different applied stresses. Temperatures and stresses used were in accordance to the range of interest for ceramic fibers and composites. In all cases, the proposed equation showed a good fit for the creep data with adjusted R-squared coefficients up to 0.97. Furthermore, the constants obtained with the data of the as-received fibers were also used to predict the creep rate of heat treated fibers, *i.e.*, different grain size, and yet again, a good fit to the experimental data was found. For other ceramic materials, the equation can be also adjusted using dimensional analysis.

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