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► **To cite this version:**

Martin Genet, Pierre Ladevèze. A new approach to the subcritical propagation of cracks in ceramics. 14th European Conference on Composites Materials (ECCM14), Jun 2010, Budapest, Hungary. hal-01274841

HAL Id: hal-01274841

<https://hal.science/hal-01274841>

Submitted on 18 Feb 2016

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A NEW APPROACH TO THE SUBCRITICAL PROPAGATION OF CRACKS IN CERAMICS

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Abstract

A new modeling approach to subcritical crack propagation (i.e. static fatigue) in ceramics has recently been introduced [9] and applied to the case of ceramic fibers. This approach relies on classical fracture mechanics, in which the zone near the crack’s tip is modified by the environment (particularly through oxidation, corrosion, etc). In some cases, it comes down to the widely-used three-parameters Paris-like subcritical crack propagation law. For the general case, a two-parameter propagation law is derived that only depends on the total oxygen flux reacting at the crack’s tip. Consequently, this model is valid for any temperature, oxygen or water pressure, etc, which can vary along time, as long as the environment is modelled properly. For Hi-Nicalon fibers up to 1000°C in dry atmosphere, the chemical phenomena observed are introduced through simple diffusion/reaction models in order to define the true oxygen flux reacting at the crack’s tip. The resulting identified model is validated on experimental data.

1 Introduction

Woven Ceramic Matrix Composites (CMCs) reinforced with long fibers are potential candidates for high-temperature thermal/structural applications [4]. Their structural performance is controlled mainly by the fibers. Indeed, the fibers within the composite are protected from the surrounding cracks thanks to the mechanical fuse concept [3]. However, once the surrounding matrix has cracked, the fibers are no longer protected from the aggressive environment: air (actually, oxygen, water, etc.) can penetrate the crack network and damage the fibers through oxidation or corrosion [3]. There have been attempts to introduce an environmental protection of the fibers within the composite (using, e.g., self-healing multilayered ceramic matrices or fiber treatments, referred to as chemical fuses), but even then the environmental protection is not complete and the fibers remain one of the key factors in the material’s lifetime [3, 7].

The study of fibers lifetime under given environmental conditions is denoted *static fatigue analysis*, or *subcritical cracking*. Subcritical propagation, also known as *slow crack growth*, has been widely studied in the literature, especially for ceramics [11, 13, 12, 6]. These works describe the crack’s propagation using a Paris-like law, also called the Evans-Wiederhorn law, relating the crack’s velocity to the crack’s stress intensity factor calculated under the assumption of elastic behavior. This law, which depends on temperature, agrees fairly well with experimental results [13, 12], but cannot be applied directly to varying environmental conditions such as oxygen or water concentration around the fibers. Moreover, it was found [11, 12, 6, 10] that as the temperature increases two different propagation stages take place. In the first stage, called the *reaction-controlled stage*, the crack’s tip is damaged directly. The second stage, called the *diffusion-controlled stage*, is different in the sense that oxygen and water, prior to damaging



the crack's tip, must first diffuse through an oxide layer formed on the surface of the fiber. Corresponding models can be found in [11] for the fibers and in [10] for the multifilament tow.

An alternative approach to subcritical propagation has recently been introduced [9]. It relies on classical fracture mechanics, in which the environment modifies the damage zone only at the crack's tip, and then drives the progressive propagation of the crack. Pure elastic behavior without creep or mechanical damage is assumed, which is what is observed within the restricted scope of this study, as will be seen further on. The proposed model for subcritical propagation does not depend explicitly on temperature, oxygen pressure, etc, but depends only on the total oxygen flux reacting at the crack's tip (which, in turn, depends on temperature, oxygen pressure, etc). Consequently, the model is valid for any temperature or oxygen pressure, which can be functions of time, and the only difficulty consists in modeling the oxidizing environment of the crack properly. Only two parameters are needed in order to use the model under varying environmental conditions.

This paper first emphasizes a fundamental aspect of the new theory: if the crack's growth is relatively slow, one can achieve a very good approximation which, in the case of constant environmental conditions, leads to the widely-used Paris-like law for the lifetime. Afterwards, for Hi-Nicalon fibers up to 1,000°C in dry environment, the chemical phenomena observed are introduced through simple models in order to define the true oxygen flux reacting at the crack's tip. The resulting calibrated model is validated using the experimental data reported in [7, 8].

2 The proposed approach for subcritical propagation

2.1 Defects modeling

In this paper, classical Weibull theory and associated idealizations are used to describe the distribution of defects over the fiber's surface [7, 9].

In Weibull theory, the strength of a fiber of length L , denoted σ_R , follows the cumulative density function:

$$p(\sigma_R < \sigma) = 1 - \exp\left(-\frac{L}{L_0} \left(\frac{\sigma}{\sigma_0}\right)^{m_\sigma}\right) \quad (1)$$

where L_0 , σ_0 and m_σ are classical Weibull coefficients.

Moreover, the growth of surface defects is assumed to be a simple one-dimensional problem in which the size of a defect is described by a scalar parameter (Figure 1) [7, 9]. Thus, the Stress Intensity Factor (SIF) induced by a surface defect of size a in a fiber subjected to a stress σ can be calculated using classical elasticity theory:

$$K = \sigma Y \sqrt{a} \quad (2)$$

where $Y = 2/\sqrt{\pi}$ is the shape coefficient associated with the crack assumed to be constant throughout the propagation.

This modeling framework introduces two important quantities for a fiber with Critical Stress Intensity Factor (CSIF) denoted K_c (see Figure 1):

- The initial size of the largest surface defect, denoted a_0 :

$$\sigma_R Y \sqrt{a_0} = K_c \quad \implies \quad a_0 = \left(\frac{K_c}{\sigma_R Y}\right)^2 \quad (3)$$



- The critical size of the defects, denoted a_c :

$$\sigma Y \sqrt{a_c} = K_c \implies a_c = \left(\frac{K_c}{\sigma Y} \right)^2 \quad (4)$$

Thus, the subcritical cracking problem is to derive a set of equations defining the evolution of the crack's size $a(t)$ from a_0 to a_c .

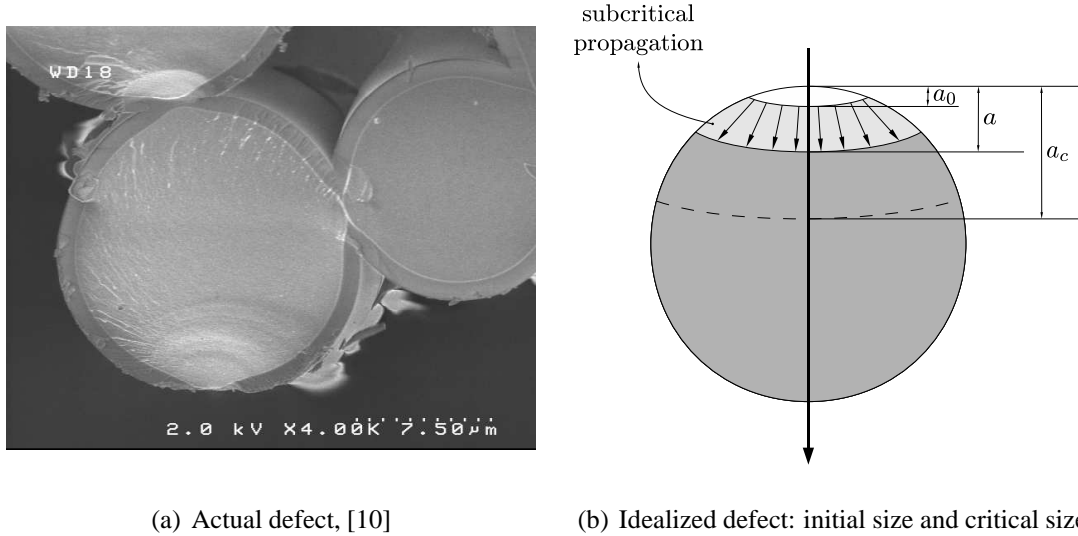


Figure 1: Surface defect in a cross section of a fiber

2.2 The classical model of subcritical propagation

The approaches found in the literature [13, 12, 7] are based on a Paris-like law for the crack's velocity:

$$\frac{da}{dt} = v_0 \cdot K^n \quad (5)$$

where v_0 and n are parameters which must be calibrated from experimental data. However, this law has several limitations: i) the crack's propagation does not follow the principles of fracture mechanics; ii) the temperature dependence induced by chemical reactions is generally handled through an exponential law (or Arrhenius law) for v_0 , which requires the introduction of an additional parameter which must be calibrated; iii) the introduction of external environmental conditions, such as the oxygen concentration, and their variations is not straightforward.

2.3 The proposed model of subcritical propagation

2.3.1 Fundamentals

The basic idea is very simple. The propagation of defects follows classical fracture mechanics:

$$\begin{cases} K \leq K_{sc}(a(t), t) \\ \dot{a}(t) \geq 0 \\ \dot{a}(t) \cdot (K_{sc}(a(t), t) - K) = 0 \end{cases} \quad (6)$$



where K_{sc} is the SubCritical Stress Intensity Factor (SCSIF), which is smaller than the CSIF K_c because of the oxidation. Let us emphasize that K_{sc} is a function of time and space.

Now let us consider the dependence of K_{sc} on the environment. The progression of the oxidation reaction which is responsible for the material's damage is described by the reaction rate ϕ_r , which is the most suitable parameter to represent the oxidation-induced degradation of the local mechanical properties (Young's modulus, strength, etc.). For the example of the degradation of the CSIF into the SCSIF, one writes:

$$\begin{cases} K_{sc}(x, t = 0) = K_c \\ \dot{K}_{sc}(x, t) = -f(K_{sc}(x, t), \phi_r(x, t)) \end{cases} \quad (7)$$

where f is a simple function, which leads to the following damage law:

$$K_{sc}(x, t) = K_c \left(1 - g \left(\underbrace{\int_0^t \phi_r(x, \tau) d\tau}_{r(x,t)} \right) \right) \quad (8)$$

where g is another simple function.

2.3.2 Calculation of r

The calculation of r is carried out through the resolution of a diffusion-reaction problem within the fiber with prescribed boundary conditions. In a first analysis, we consider the following assumptions (see Figure 2):

1. The diffusion-reaction phenomenon is localized near the oxygen input. Thus, the problem can be written over a semi-infinite unidirectional space.
2. The velocity of the diffusion-reaction process is significantly higher than that of the crack's growth. Thus, the evolution can be considered quasi-stationary.
3. The mechanical and chemical fields are uncoupled. Thus, the reaction and diffusion coefficients are independent of the strain or stress state.

Thus, the concentration and flux of oxygen within the fiber can be written [9]:

$$\begin{cases} c(x, t) = \frac{\phi_0(t)}{\sqrt{kD}} \cdot \exp\left(-\sqrt{\frac{k}{D}}(x - a(t))\right) \\ \phi(x, t) = \phi_0(t) \cdot \exp\left(-\sqrt{\frac{k}{D}}(x - a(t))\right) \end{cases} \quad (9)$$

Where D is the diffusion coefficient of O_2 in SiC , k the reaction coefficient of O_2 with SiC , ϕ_0 a given boundary function, and v_0 a given velocity function.

Considering that the diffusion-reaction process zone is small compared to the size of the crack, r can be written [9]:

$$r(x, t) \approx \begin{cases} \frac{\phi_0(t)}{\dot{a}(t)} & \text{when } x = a(t) \\ 0 & \text{when } x > a(t) \end{cases} \quad (10)$$

Therefore, from now on, we will consider only the surface term:

$$r(t) = r(a(t), t) = \frac{\phi_0(t)}{\dot{a}(t)} \quad (11)$$



Finally, the damage law writes:

$$K_{sc}(t) = K_c \left(1 - g \left(\frac{\phi_0(t)}{\dot{a}(t)} \right) \right) \quad (12)$$

where $\phi_0(t)$ is the incoming oxygen flux at the crack's tip, *i.e.* the reaction rate of the oxidizing surface reaction, and $\dot{a}(t)$ is the crack's velocity.

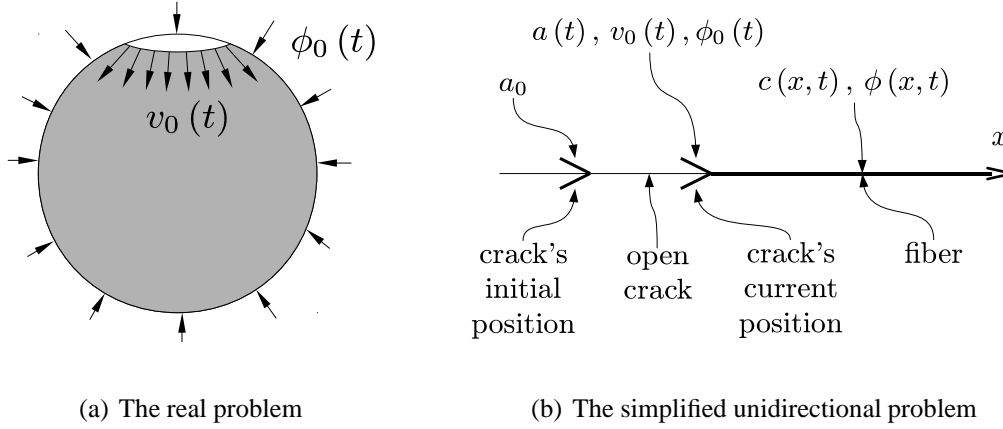


Figure 2: The diffusion-reaction problem for the calculation of r

2.3.3 Choice of g

Among the several possible choices for g [9], we consider the following one:

$$g(r) = 1 - \lambda/r^n \quad (13)$$

where λ and n are positive scalars, which is a good compromise between simplicity and physical meaning [9].

In this case, the damage law writes:

$$\frac{K_{sc}(t)}{K_c} = \lambda \left(\frac{\dot{a}(t)}{\phi_0(t)} \right)^n \quad (14)$$

2.3.4 Summary

Finally, each part of the model has a clear physical meaning:

- Equation (6) is the standard equation of fracture mechanics. Since the damaging reaction is a surface reaction (see Equation (11)), the subcritical propagation is stable and continuous. Thus, during the whole propagation ($\dot{a}(t) > 0$), Equation (6) reduces to:

$$K(a(t), t) = K_{sc}(t) \quad (15)$$

- The damage law (14) also have a clear interpretation: the lower the velocity and/or the higher the reaction rate, the smaller the SCSIF and, conversely, the higher the velocity and/or the lower the reaction rate, the larger the SCSIF. The power law defined by λ and n characterizes the degradation of the CSIF for a given amount of oxygen arriving at the crack's tip. This is an intrinsic material law which depends neither on temperature nor on time.



Grouping Equations (2) for the crack's intensity, (15) for the crack's propagation and (14) for the material's degradation through oxidation, this theory produces a differential problem in $a(t)$ which describes the crack's subcritical propagation under any mechanical loading and any chemical environment:

$$\dot{a}(t) = \frac{\phi_0(t)}{\lambda^{1/n} K_c^{1/n}} \cdot K^{1/n} \quad (16)$$

This can lead to several models depending on the complexity of the description of the chemical environment surrounding the crack.

It is worth mentioning that if the reaction rate is assumed to be constant over time, one reverts to the framework of the Paris-like law (see Equation 5).

3 Application to Hi-Nicalon fibers lifetime

3.1 Environment modeling

Among the several possible models for the fiber oxidizing environment [9], the one studied in this paper is a variation of the framework introduced in [5], customized for the $SiC_{(s)} + O_{2(g)} \rightarrow SiO_{2(s)}$ reaction on the surface of the fiber. This enables us to introduce the effect of the oxide layer generated around the fiber which delays the arrival of oxygen at the crack's tip.

The calculation of the reaction rate is carried out through the resolution of a diffusion-reaction problem within the oxide layer generated around the fiber. In a first analysis we consider the following assumptions (see Figure 3):

1. The problem is axisymmetrical and the thickness of the oxide layer is small in relation to the fiber, which allows to consider a unidirectional model.
2. The velocity of the diffusion-reaction process is significantly higher than that of the growth of the oxide layer, which allow to consider a quasi-stationary evolution.
3. The mechanical and chemical fields are uncoupled.

The concentration and flux of oxygen within the oxide layer can then be written [9]:

$$\begin{cases} c(x, t) = \left(1 + \frac{x}{e(t)}\right) \cdot c_0(t) - \left(\frac{x}{e(t)}\right) \cdot \frac{c_0(t)}{1 + ke(t)/D} \\ \phi(x, t) = \frac{kc_0(t)}{1 + ke(t)/D} \end{cases} \quad (17)$$

where k is the reaction coefficient between O_2 and SiC , whose temperature dependence is assumed to be of Arrhenius type (k_0 being the prefactor and E_a^k the activation energy):

$$k = k_0 \cdot \exp\left(-\frac{E_a^k}{RT}\right) \quad (18)$$

c_0 is the O_2 concentration in the surrounding air, related to the partial O_2 pressure through the ideal gas law:

$$c_0 = \frac{p_{O_2}}{RT} \quad (19)$$

D is the diffusion coefficient of O_2 in SiO_2 , whose temperature dependence is assumed to be of Arrhenius type (D_0 being the prefactor and E_a^D the activation energy):

$$D = D_0 \cdot \exp\left(-\frac{E_a^D}{RT}\right) \quad (20)$$



and where e is the thickness of the oxide layer, given by:

$$\begin{cases} \frac{de}{dt}(t) = \frac{\rho}{M} \cdot \frac{kc_0(t)}{1 + ke(t)/D} \\ e(t=0) = 0 \end{cases} \quad (21)$$

where M and ρ are respectively the volume mass and the molar mass of SiO_2 .

Thus, the reaction rate at the crack's tip writes [9]:

$$\phi_0(t) = \frac{kc_0(t)}{1 + ke(t)/D} \quad (22)$$

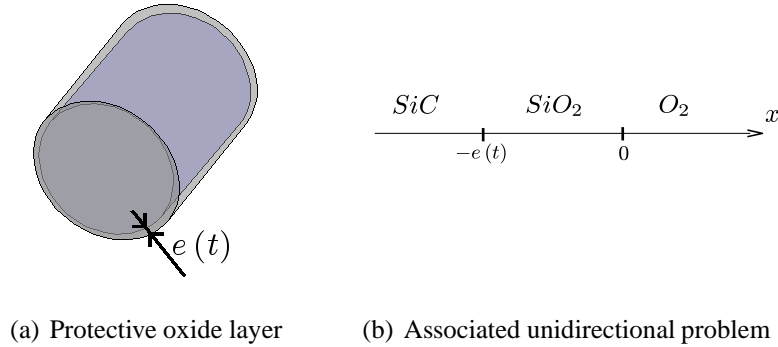


Figure 3: The fiber's self-protection problem

3.2 Lifetime models

Finally, the fiber's time-to-rupture, denoted t_R , is the solution of the differential problem:

$$\begin{cases} a(t=0) = a_0(\sigma_R); e(t=0) = 0 \\ \dot{a}(t) = \frac{1}{\lambda^{1/n}} \cdot \frac{kc_0}{1 + ke(t)/D} \sigma^{1/n} Y^{1/n} \sqrt{a(t)}^{1/n} \\ \frac{de}{dt}(t) = \frac{\rho}{M} \cdot \frac{kc_0(t)}{1 + ke(t)/D} \\ a(t=t_R) = a_c(\sigma) \end{cases} \quad (23)$$

3.3 Calibration

3.3.1 Summary of published data

For Hi-Nicalon fibers, the toughness, previously presented in [2, 7], as well as the Weibull coefficients, previously presented in [7], are given in Table 1.

Moreover, the silica-related coefficients, as well as the diffusion coefficient of oxygen in silica, previously presented in [1], are given in Table 2.

3.3.2 Fitting of experimental data

We use [7]'s experimental data of fiber lifetime probability law at several mechanical and environmental conditions. It can be shown [9] that the calibrated values presented Table 3 are the unique ones to minimize the model-experiments distance.

Figure 4 shows the comparison of the prediction given by the calibrated model with the experimental data. It is clear that the model is compatible with experimental data at low and high temperature, and then naturally deals with reaction- and diffusion-controlled propagation.



K_c ($MPa \cdot \sqrt{\mu m}$)	L_0 (mm)	σ_0 (MPa)	m_σ
1250	1	3640	8.47

Table 1: Toughness [2, 7] and Weibull coefficients [7] of Hi-Nicalon fibers

ρ_{SiO_2} ($Kg \cdot mol^{-1}$)	M_{SiO_2} ($Kg \cdot m^{-3}$)	D_0 ($m^2 \cdot s^{-1}$)	E_a^D ($J \cdot mol^{-1}$)
$60.0843 \cdot 10^{-3}$	2200	$2 \cdot 10^{-8}$	177,000

Table 2: Silica properties and diffusion coefficient of oxygen in silica [1]

k_0 ($m \cdot s^{-1}$)	E_a^k ($J \cdot mol^{-1}$)	λ ($mol \cdot \mu m^{-3}$)	n
$1.47 \cdot 10^2$	180,000	0.2	0.13

Table 3: Calibrated reaction coefficient of oxygen with silicon carbide and subcritical propagation coefficients

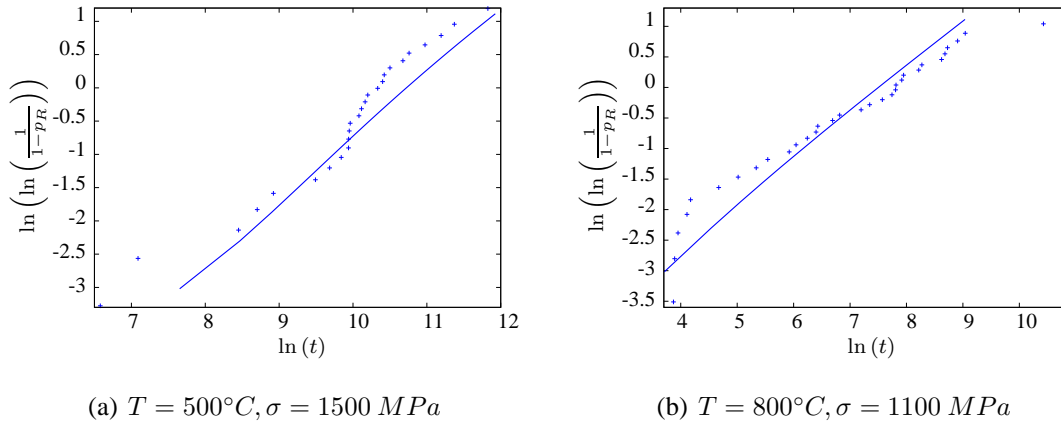


Figure 4: Fiber lifetime cumulative probability distributions: calibrated second model vs. experiment



3.4 Elements of validation

A first element of validation can be given. However, not enough data is available on individual Hi-Nicalon fibers. Therefore, one must use data on Hi-Nicalon fiber bundles. Indeed, observing that a fiber bundle consists of a large number of fibers (≈ 500), one can show that its lifetime is subject to only small variations and is close to that of a particular fiber, called the critical fiber, characterized by a given probability of failure [7, 8]. The comparison of the predicted and experimentally observed [8] fiber bundle lifetimes as functions of the partial oxygen pressure (Figure 5) shows that the orders of magnitude predicted using the model are correct. Thus, the model may be used in high oxygen pressure environment (e.g. for accelerated test), and in low oxygen pressure environment (e.g. behind the protection of the self-healing matrix).

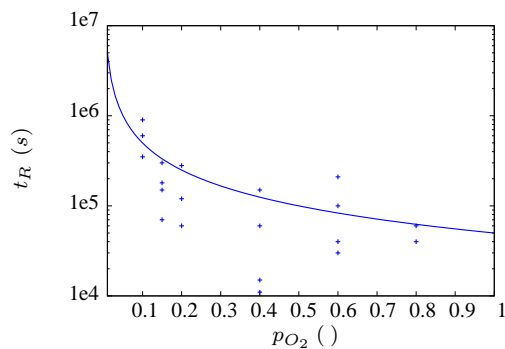


Figure 5: Fiber bundle lifetime vs. partial oxygen pressure, at $T = 500^{\circ}C$ and $\sigma = 1,000 MPa$: predicted lifetime of the bundle's critical fiber (*i.e.* the fiber with 5 % failure probability) vs. experiment [8]

4 Conclusion

This paper has presented some aspects of a recent work [9] that reexamined the well-established modeling framework for the subcritical propagation of cracks in ceramics. A more general theory for the description of the degradation of the material due to oxidation was introduced and applied to the case of surface defects on ceramic fibers. The analysis showed that the classical Paris-like law belongs to this framework.

A lifetime prediction tool for ceramic fibers was derived from the new framework, which takes into account the oxide layer generated around the fibers, thus delaying the oxidation process. After calibration, the predictions given by the model are correct at both low and high temperatures because this model handles reaction-controlled propagation and diffusion-controlled propagation naturally. Some validation was presented, based on [7, 8]'s experimental data, and work is currently underway in order to extend this validation.

A first interesting application of this approach would be the analysis of accelerated tests (for example using high partial oxygen pressure) in order to reduce the cost of testing campaigns. A second one would be the introduction of matrix self-healing mechanisms, in order to extend this prediction tool from fibers to complete composite tows.



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