# IDENTIFICATION AND VALIDATION OF A MULTIPHYSICS MACROMODEL FOR THE LIFETIME PREDICTION OF SELF-HEALING CERAMIC MATRIX COMPOSITES

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## ABSTRACT

The objective of this paper is to present the identification strategy and the validation of a model used to predict the behavior and lifetime of self-healing ceramic matrix composites. This model is divided into three parts: a mechanical part which describes the evolution of the cracks, a physicochemical part which describes the degradation of the fibers and the healing process, and a crack opening indicator which links these two parts. The identification and the validation of the mechanical part are carried out on the macroscale using stress/strain curves from tensile tests. The crack opening indicator is identified using a micromodel which is identified and compared to macroscopic experimental results. The physicochemical part is identified from tests and observations on the microscale, then updated according to lifetimes obtained from creep tests on composites. This model shows good agreement with thermomechanical loading experiments in a medium range of temperatures.

## 1. INTRODUCTION

This paper focuses on SiC/SiC ceramic matrix composites known as self-healing materials [1]. The self-healing process consists in filling active cracks with an oxide, which limits the diffusion of oxygen toward fibers that might suffer from subcritical cracking [2]. Therefore, this process leads to a considerable extension of the material's lifetime. The challenge associated with the use of such materials resides in the development of robust models of their mechanical behavior up to failure, even for lifetimes up to 50,000 hours for civil applications. The model used in this paper is defined on the macroscopic scale and is capable of taking into account the various degradation/healing mechanisms involved in complex multiaxial thermomechanical [3,4] and chemical [5] loading. It is built from an understanding of the degradation/healing mechanisms on the microscale. The model is divided into three parts. The first part concerns the mechanical modeling, i.e. the modeling of the evolution of the different crack networks in the material using the anisotropic damage theory [6]. The second part concerns the physicochemical modeling, i.e. the modeling of the degradation of fibers in the presence of oxygen and of the healing mechanism. The third part concerns the link between the other two, i.e. the description of the opening of the intra-yarn transverse cracks which allows or prevents diffusion of the oxygen toward the fibers. The identification of this macromodel was carried out on several scales, from the macroscopic scale for mechanics to the microscopic scale for physicochemical mechanisms. The model, which has been implemented into an in-house analysis code, has been validated on several examples with respect to both the lifetime prediction and the physical understanding of the degradation/healing scenarios. The main objective of this paper is to describe the model's identification procedure, to determine its range of validity and to pinpoint the parts of the model which need to be improved.

With regard to mechanical damage, the model was identified, for the most part, on the macroscopic scale through macro composite tests. This was the case of matrix and yarn damage and of fiber breakage, using the work of [7]. For the fatigue part of the model associated with the wear of the interface between the fibers and the matrix, a micromodel was used. This micromodel, which was also used to set up a crack opening indicator, was based on the work of [7,8,9]. This indicator was used to introduce the physicochemical model of the fiber's degradation and self-healing process in an oxidizing environment. The physicochemical model was identified mainly on the microscale using the work of LCTS [2,9], except for some parameters related to oxide ejection which remained macro. A sensitivity analysis was performed for various parameters of the model.
In order to validate this model, a numerical tool capable of taking into account complex multiaxial mechanical and chemical loading was developed. This tool enabled us to demonstrate the capabilities of the model in predicting the lifetime of self-healing ceramic composites and can be used to compare numerical and experimental results. Several examples were calculated to validate the influence of the thermomechanical load path on the composite's lifetime.

## 1. THE MECHANICAL MODEL

The mechanical behavior was identified and validated against tests performed at INSA Lyon and Snecma Propulsion Solide (SAFRAN group) and presented in [10,11].
The mechanical macromodel is based on the work of [6,3,9] and is presented in [4]. In this model, the damage kinematics is not defined *a priori*, but through the evolution laws. In addition, the total damage is partitioned according to the various degradation mechanisms involved:

- inter-yarn matrix cracking
- intra-yarn longitudinal and transverse matrix cracking
- fiber breakage
- debonding and wear of the fiber/matrix interface

The damage and plastic evolution laws were identified based on 0° and 45° cyclic tensile tests or fatigue loading tests. One should note that no experimental data for multiaxial loading and only limited data for 45° tests is available. The main remaining difficulty consists in dividing the global damage observed from experiments into damage contributions associated with the different crack networks. In fact, this is easy in the case of low or high stresses: under low stresses, damage is assumed to correspond to inter-yarn matrix cracking, whereas high stresses in the fiber's direction lead to intra-yarn matrix cracking. In the intermediate zone, i.e. the beginning of intra-yarn damage and the saturation of inter-yarn matrix cracking, additional experimental data is required to distinguish between the two mechanisms. A technique worth exploring would be acoustic emission [13,14].

Test results leading to partial identification of the model, respectively a 0° tensile test and a 45° tensile test, are shown in Figures 1 and 2.

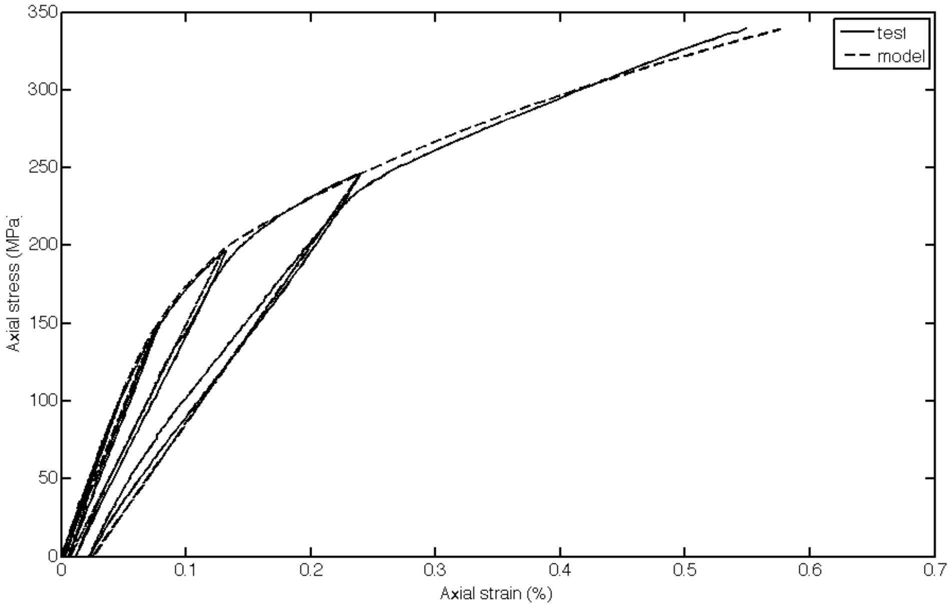


**Figure 1: Identification of the mechanical behavior of the composite from a 0° tensile test**

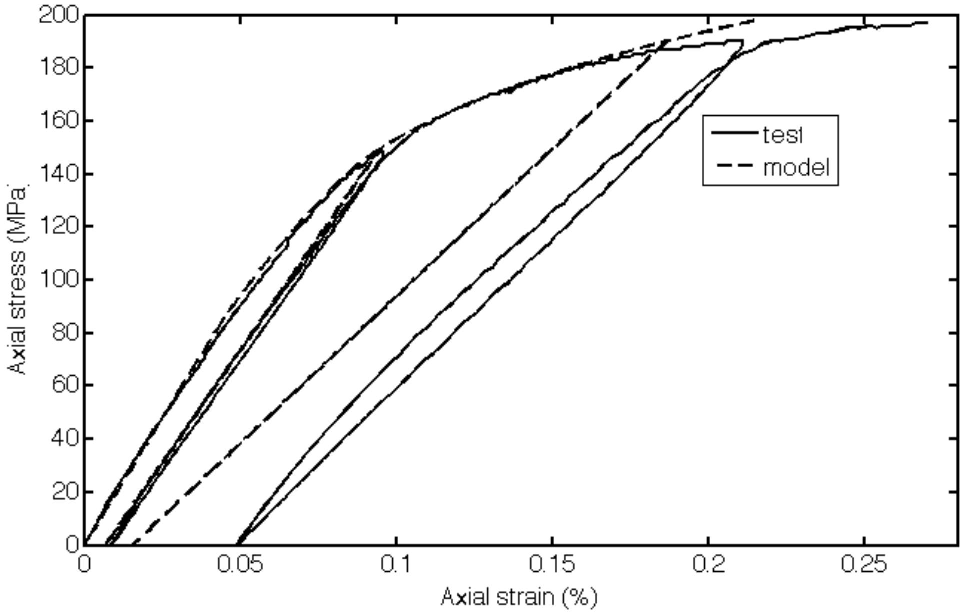


**Figure 2: Identification of the mechanical behavior of the composite from a 45° tensile test**

The part of the model associated with fatigue was identified in the same way.

## 2. THE CRACK OPENING INDICATOR

The link between the mechanical macromodel and the physicochemical macromodel is achieved through an intra-yarn transverse matrix crack opening macroindicator.

Because such an opening (about 1 micrometer for a 160 MPa axial tensile load) was not observed experimentally (only crack densities were observed, see Figure 3), the identification of this part of the macromodel was carried out using a micromodel. This micromodel is based on [8,15,16,17] and is described in [9,4]. It was identified in order to give results which are consistent with the macro tensile tests stress/strain curves of composites. This work was done by [18]. Additional work to improve this initialization and make it more robust is being considered.

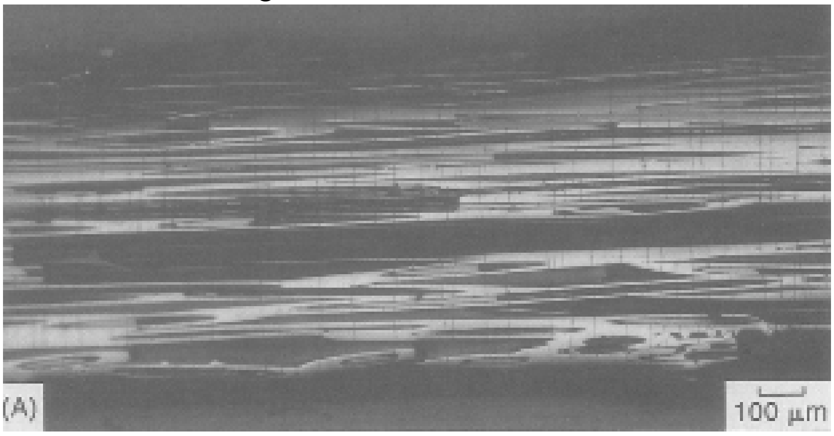


**Figure 3: Distribution of intra-yarn cracks in a minicomposite (from [19])**

Because this indicator is of major importance for the prediction of lifetime, an experimental study is underway in order to determine the actual crack opening either on microcomposites or on laminated composites. A complementary approach consists in constructing a refined finite element model in order to determine this opening numerically.

## 3. THE PHYSICO-CHEMICAL MODEL

The physicochemical part of the model involves two mechanisms:

- fiber degradation in the presence of oxygen.
- healing (oxidation reaction of the matrix layers and diffusion of the oxygen).

### The fiber degradation model

Fiber degradation in the presence of oxygen is described in [2,20] as a subcritical cracking mechanism. The available data consisted of lifetime creep tests on Hi-Nicalon fibers and yarns. Different stress levels were applied at a wide range of temperatures (from 450°C to 850°C). Different partial oxygen pressures (from 5% to 80% of the atmospheric pressure) were also studied in [2]. Let us note that throughout the healing process the oxygen level behind the healing oxide plug near the fibers was much less than the levels indicated by the tests.
An evolution model was proposed in [5] based on the work done at LCTS [2, 20].
This failure stress evolution law is controlled by a weighted cumulated oxygen concentration denoted Θ. This kind of control was validated and a comparison with experimental data from [2] is shown in Figure 4. In this figure, $\theta\sigma_r^{\ n} = B(T)$ is related to the model proposed by Cluzel (in an integrated form for constant stress, concentration and temperature) and $t\sigma_r^{\ n} = A(T)$ is related to an earlier model developed and identified by Gauthier.

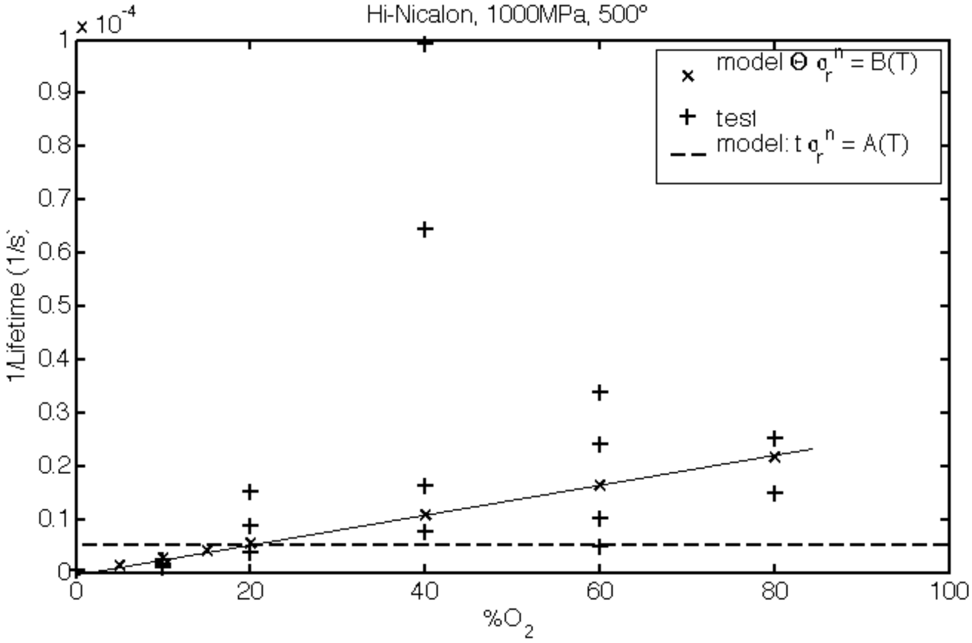


**Figure 4: Evolution of the inverse of the lifetime as a function of the oxygen percentage for Hi-Nicalon yarns under 1000MPa tensile stress at 500°C.**

These data available for yarns were scaled up for composites using a macro parameter, identified using a tensile test failure stress, which is very close to the fiber rate in the composite. A more refined approach taking into account the waviness of the fabric and the damage level was studied in [21] using a 3D finite element micromodel. Genet and Ladevèze also proposed a law, inspired by the work of [2,22], for this mechanism. This law is capable of taking into account the influence of the stress level applied to the fiber.

**The healing model**

The healing mechanism consists in filling the cracks with an oxide plug in order to limit (by diffusion) the access of the oxygen from the composite pores to the fibers. This oxide plug is created by oxidation of specific matrix layers by the oxygen.
The associated macromodel is based on the description of the evolution of an oxide plug in a reference crack [5]. This reference crack is partially defined using the macro crack opening indicator constructed above. This model tends to take into account the various chemical reactions between the oxygen and the different matrix layers as well as volatilization in the presence of water vapor or ejection by cyclic mechanical loading.
Most of the parameters involved in this model were initialized from values extracted either from micro SEM observations or from models on the microscale [12, 23]. In fact, very few of these parameters have a significant influence on the final lifetime. This lifetime is the only result on the macroscopic scale which is available to quantify the healing mechanism experimentally. In other words, the whole healing process is summarized by only a single macroscopic experimental scalar value. The remaining influential parameters are identified in order to get good agreement with the lifetime test results at 500°C for one level of applied stress (160MPa). In addition, these parameters must stay within a reasonable range determined by micro considerations. Two of these influential parameters are:

- The crack opening indicator.
- The oxide plug partial ejection parameter (in the case of cyclic fatigue loading).

For example, for a tensile test at high constant stress (300MPa) and at a constant medium-range temperature of 600°C, even a factor 2 in the crack opening indicator can lead to a factor 1000 in the lifetime prediction. In this particular case, if the opening indicator is too small, rupture can occur before the healing process is completed, which is terrible for the lifetime prediction.

The model of the healing process was validated against lifetime predictions. A wide range of creep tensile tests on composites were performed [10, 11] at different temperatures (from 450°C to 750°C) under 20 kPa controlled partial oxygen pressure and at various stress levels (from 80 MPa to 310 MPa). This lifetime validation also validated the crack opening indicator and the damage macromodels. One should note that this validation was not done in the lifetime range of a civil application of this type of material (about 50,000 h). Figure 5 shows lifetime comparisons for 500°C and 600°C tests, i.e. at medium temperatures. Figure 6 shows similar comparisons at 450°C and 750°C.

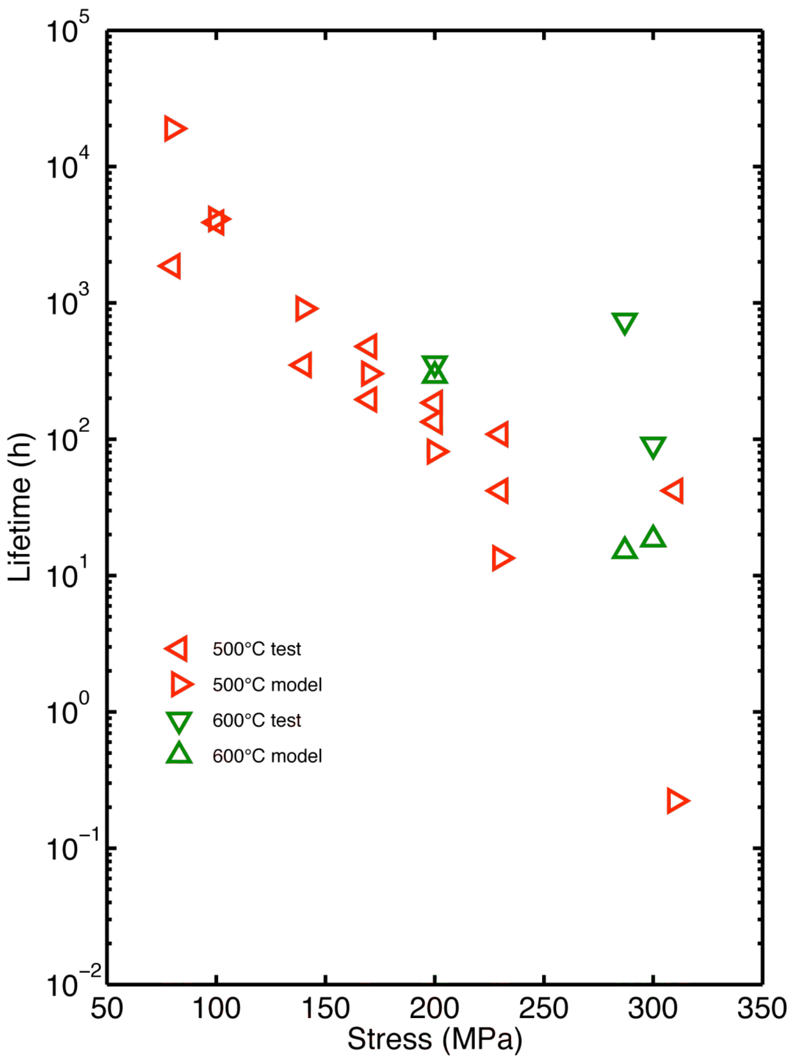


**Figure 5: Lifetime comparison between the model and experimental results (500°C, 600°C)**

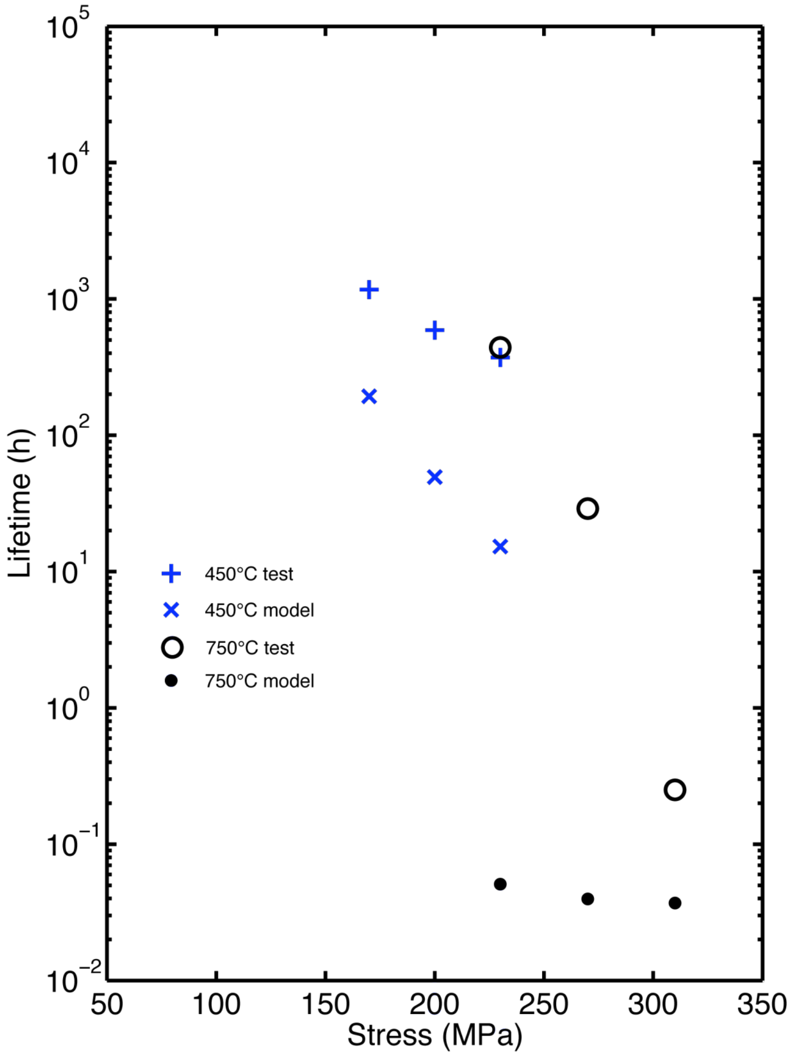


**Figure 6: Lifetime comparison between the model and experimental results (450°C and 750°C)**

First, one should note that validation is not an easy task because the experimental dispersion is not known very well. As shown in Figures 5 and 6, the model is valid in a medium stress range (between 100 MPa and 200 MPa) and in a wide temperature range (from 500°C to 700°C). This is quite remarkable because, as mentioned before, the physicochemical part of the model was identified on the microscopic scale and only one piece of information on the macroscopic scale (i.e. one point for one temperature and one stress level) was needed to complete the identification procedure.
At low and high stress levels, the lifetime prediction is respectively too long and too short. At both high and low temperatures, the lifetime prediction is too short.

With regard to improving the lifetime prediction, the influence of stress is due to the crack opening indicator, whose form is not suitable for high stress levels. In the case of low stress levels, the presence of a preexisting crack would have to be assumed. This would lead to an extension of the stress validity range of the model.
Concerning the temperature validity range, new healing scenarios for high temperatures should be taken into account. In fact, at temperatures greater than 700°C, other matrix layers react with the oxygen and protect the fibers. At low temperatures, the identification of the micro law should be improved.

## 4. CONCLUSIONS

This paper focuses on the identification and validation of a macromodel dedicated to the prediction of the behavior and lifetime of self-healing ceramic matrix SiC/SiC composites. The model is divided into three parts: the mechanical part, the physicochemical part and a link (a crack opening indicator) between the two. The mechanical part is identified on the macro-scale while the crack opening indicator is identified using a micromodel. The physicochemical part is initialized using micro observations and updated in order to yield good lifetime predictions. The whole model was validated on the macroscale based on damage predictions as well as lifetime predictions.

The model is capable of achieving quite good predictions in a medium range of thermomechanical loading cases. One should be aware that experimental dispersion is unknown and can be significant. The validity range of the model could be extended by addressing several points:

- improving the crack opening indicator and developing a better understanding of the associated physical mechanisms at low and high stress levels.
- adding new healing scenarios for high temperatures.
- taking variability into account.

## REFERENCES


[1]Dambrine, B,. “Which composite materials in turbojets of Snecma Moteurs?”, *JNC14, Quatorzièmes journées nationales sur les composites*, pp 3-10, 2006, in french.

[2]Gauthier W., Lamon J., Pailler R., “Static fatigue of monofilaments and of SiC Hi-Nicalon yarns at 500°C and 800°C”, *Revue des Composites et des Matériaux Avancés*, Vol. 16(2), pp 221-241, 2006, in french.

[3]Ladevèze P.,Letombe S., Cluzel C., “A CMCs damage model based on micro and macro-mechanics for high temperatures and complex loadings”, *HTCMC 4 - 4th Int. Conf. on High Temperature Ceramic Matrix Composites*, pp 578-583, 2001.

[4]Baranger E., Cluzel C., Ladevèze P., Mouret A., “Prediction of the lifetime of self-healing ceramic matrix composites: I- Macroscopic mechanical modelling of cacking”, *JNC15, Quinzièmes journées nationales sur les composites*, 2007, in french.

[5]Cluzel C., Baranger E., Ladevèze P., Mouret A., “Prediction of the lifetime of self-healing ceramic matrix composites: II- cracks and oxidation mechanisms análisis”, *JNC15, Quinzièmes journées nationales sur les composites*, 2007, in french.

[6]Ladevèze P., 2002, "An anisotropic damage theory with unilateral effects: applications to laminate and three- and four-dimensional composites", *Continuum damage mechanics of materials and structures*, O. Allix and F. Hild editors, Elsevier.

[7]Penas O., Reynaud P., Rouby D., Fantozzi G., “Self-healing SiCf/SiC composite behavior under high temperature cyclic fatigue in air “, *HTCMC 4 - 4th Int. Conf. on High Temperature Ceramic Matrix Composites*, pp 480-485, 2001.

[8]Evans A.G., Zok F.W., McMeeking R.M., “Fatigue of ceramic matrix composites”, *Acta. Metal. Mater,.* Volume 43, Issue 3, Pages 859-875, 1995.

[9]Letombe S., Cluzel C., Ladevèze P., “A macroscopic model coupling oxidation and damage for CMCs”, *JNC13, Treizièmes journées nationales sur les composites*, pp. 713-722, 2003, in french.

[10] Penas O., "Etude de composites SiC/SiBC à matrice multiséquencée en fatigue cyclique à hautes températures sous air", Thesis INSA Lyon, 2002.

[11] Moevus M., "Mécanismes d'endommagement, émission acoustique et durées de vie en fatigue statique du composite SiC/(Si-B-C) aux températures intermédiaires (<800°C)", Thesis INSA Lyon, 2007.

[12] Rebillat F., Martin X., Guette A., “Kinetic oxidation laws of boron carbide in dry and wet environnements”, *HTCMC 5 - 5th Int. Conf. on High Temperature Ceramic Matrix Composites*, pp 321-326, 2004.

[13] Moevus M., Rouby D., Godin N., R’Mili M., Reynaud P., Fantozzi G. and Farizy G., “Analysis of damage mechanisms and associated acoustic emission in two SiC/[Si–B–C] composites exhibiting different tensile behaviors. Part I: Damage patterns and acoustic emission activity”, *Composites Science and Technology*, Volume 68, Issue 6, May 2008, Pages 1250-1257.

[14] Moevus M., Rouby D., Godin N., R’Mili M., Reynaud P., Fantozzi G. and Farizy G., “Analysis of damage mechanisms and associated acoustic emission in two SiC/[Si–B–C] composites exhibiting different tensile behaviors. Part II: Unsupervised acoustic emission data clustering”, *Composites Science and Technology*, Volume 68, Issue 6, May 2008, Pages 1258-1265.

[15] Aveston J., Cooper G.A., Kelly A., "Single and multiple fracture", *Proceedings of conference on the properties of fibre composites of the national physical laboratory*, IPC Sci. Technol. Press, vol.4, pp.15-26, 1971.

[16] Rouby D., Reynaud P., "Fatigue behavior related to interface modification during load cycling in ceramic-matrix fibre composites", *Composites Science and Technology*, v. 48, pp. 109–118, 1993.

[17] Reynaud P., "Cycle fatigue of ceramic-matrix composites at ambient and elevated temperatures", *Composites Science and Technology*, v. 56, pp. 809-814, 1996.

[18] Letombe S., "Modélisation du couplage oxydation/endommagement des Composites à Matrice Céramique Autocicatrisante", *Thesis ENS-Cachan*, 2005.

[19] Lissart N., Lamon J., 1997, "Damage and failure in ceramic matrix minicomposites: experimental study and model", Acta material, v. 45, no. 3, pp. 1025-1044.

[20] Pailler F., Lamon J., "Micromechanics based model of fatigue/oxidation for ceramic matrix composites", *Composites Science and Technology*, Volume 65, Issues 3-4, March 2005, Pages 369-374

[21] Genet M., Ladevèze P., Lubineau G., Mouret A., "Toward a virtual material for lifetime prediction of CMCs ", *ECCM13*, Stockholm, 2008.

[22] Wachtman J. B., "Mehanical properties of ceramics", John Wiley and Sons, 1996.

[23] Garitte E., Rebillat F., Guette A., "B4C as the precursor of the healing in a SiC/SiC composite : behavior under wet atmosphere", *ECCM12*, Porto, 2006.