

Lifetime prediction with acoustic emission during static fatigue tests on ceramic matrix composite at intermediate temperature under air

Emmanuel Maillet^a, Nathalie Godin^{*a}, Mohamed R'Mili^a, Pascal Reynaud^a, Gilbert Fantozzi^a, Jacques Lamon^b

^a INSA-Lyon, Laboratoire MATEIS, F-69621 Villeurbanne, France

^b LMT-CNRS, F-94230 Cachan, France

*Nathalie.godin@insa-lyon.fr

Keywords: Ceramic matrix composite, lifetime prediction, acoustic emission

Abstract

A main purpose of this paper is to consider the possibility of predicting rupture time of CMC from damage evolution recorded by AE technique. The energy of acoustic emission signals recorded at two sensors is used to evaluate in real-time energy attenuation due to damage accumulation. In this study SiC_f/[Si-B-C] composites are studied under static fatigue at 450 °C under air. During the static fatigue test, attenuation coefficient B, obtained from AE measurement; increases significantly during the first half of tests and reaches a plateau value at approximately 50% of the rupture time. The increase of attenuation coefficient B may be related to matrix crack opening. The plateau observed on the evolution of attenuation coefficient B indicates that matrix crack opening gets to an equilibrium state around 50% of the rupture time. Beyond that point, the imposed oxygen flux controls the speed at which fibers break by subcritical crack growth. Indeed, previous work demonstrated that during static fatigue tests, the AE energy release has a critical aspect at a local scale beyond 50% of the rupture time. This critical aspect corresponds to a second damage phase where subcritical crack growth in fibers is predominant, leading to ultimate failure of the composite.

1. Introduction

Ceramic Matrix Composites (CMCs) are candidates for use in aeronautical engines owing to their low density and good mechanical properties at high temperatures. CMCs can experience large deformations ($\approx 1\%$) as a result of energy dissipation through multiple matrix cracking and deflection of cracks at fiber/matrix interfaces. Several approaches to describe the mechanical behavior of CMCs under tensile loading [1-2] have been proposed. Moreover, the Acoustic Emission (AE) technique has been considered in order to monitor microstructural changes and damage evolution in CMCs at ambient and intermediate temperatures [3-5].

In static fatigue at high temperature under low stresses, composite ultimate failure results from oxidation-activated crack growth in fibers and associated stress redistributions when fibers fail [6]. The development of the [Si-B-C] self-healing matrix allowed protection against oxidation. Above 550°C, under oxidizing atmosphere, boron trioxide and silicon carbide react producing a borosilicate glass that fills up the cracks. Expected lifetimes in

service conditions are tens of thousands of hours, which can hardly be checked out using laboratory tests for practical reasons. Therefore, a real-time prediction of the remaining lifetime during tests is necessary. It requires the monitoring of damage evolution for which AE measurement is a suitable technique. In fact, the AE technique is based on the recording and analysis of transient elastic waves that are generated during material damage. It provides real-time data on initiation and evolution of damage in terms of location and mode.

The energy of an AE signal corresponds to a part of the energy released by the source. Therefore, it is a relevant measure of damage evolution. Momon et. Al [7]. studied the release of AE signals energy during static fatigue tests on woven SiCf/[Si-B-C] and Cf/[Si-B-C] composites at intermediate temperatures. The coefficient of emission R_{AE} was defined. It allowed the identification of a characteristic time around 60% of rupture time. Moreover, the Benioff law [8], introduced at first in seismology, was applied. Experimental data recorded after the characteristic time fitted well this power law, indicating critical features of energy release prior to rupture. Recently [9], determination of the coefficient of emission was improved to allow real-time determination.

The present paper proposes a method for the evaluation of energy attenuation that uses the energy recorded from AE sources generated during material damage. The method is based on the calculation of the ratio of AE energy recorded for each source at both ends of the specimen using two sensors. Attenuation is evaluated from thousands of local attenuation measurements obtained for the AE sources which are detected. Finally, the analysis of the critical aspect of AE sources energy release prior to rupture is discussed for 7 static fatigue tests.

2. Material and experimental procedure

2.1 Material

The composite material was manufactured by SAFRAN Snecma Propulsion Solide. It was made of woven PyC coated SiC fibers (Hi-Nicalon, Nippon Carbon Ltd, Japan) and a multilayered [Si-B-C] matrix produced via chemical vapor infiltration. A seal coat protected the material. The fiber volume fraction was 35 to 40% and porosity was about 12 v%. Dog-bone shaped specimens were machined.

2.2 Mechanical testing

Fatigue tests were performed under a constant load. A pneumatic tensile machine was used. It has been designed to ensure high stability for long duration tests while reducing environment noise. Before loading, each specimen was heated up to the test temperature (450°C or 500°C) at a rate of 20°C/min. Then, after one hour at the test temperature, it was loaded up at a rate of 600 N/min to the test load selected in the range of 40 to 95% of the rupture load (indicated for each test in terms of stress as the ratio σ/σ_r). The rupture load (and rupture stress σ_r) had been determined during tensile tests under monotonous loading at room temperature. An extensometer was used for elongation measurement. The machine was equipped with a 25 kN load cell.

2.3 Acoustic Emission monitoring

Two sensors (micro80, Physical Acoustics Corporation) were positioned, 190 mm apart, on specimen. The sensors were placed in housings machined in the grips. A holding system with spring was used in order to maintain constant contact pressure between sensors and material throughout test. Medium viscosity vacuum grease was used as a coupling agent. Each sensor was connected to a preamplifier (gain: 40 dB, frequency range: 20-1200 kHz), which was connected to the data acquisition system (two-channel MISTRAS 2001, Physical Acoustics Corporation). The threshold was set to 32 dB in order to filter out signals from ambient noise. The acquisition parameters were set as follows: peak definition time 50 μ s, hit definition time 100 μ s and hit lockout time 1000 μ s. These values were optimized before test so as to obtain consistent AE signal parameters (rise time, duration, energy, ...) for similar artificial sources (Hsu-Nielsen sources). For each AE signal, the following data were recorded: arrival time (first threshold crossing), stress and strain values, as well as signal energy. Wave velocity (9500 m/s) was determined prior to testing using a pencil lead break procedure. The location of sources $x(n)$ was determined from the difference in event time detection by sensors. Since two sensors were used, location was determined with respect to specimen longitudinal axis. Only those signals located in the gauge length (60 mm) were kept. Each source n was therefore described by the time at which the closest sensor was triggered $t(n)$, its abscissa $x(n)$ and the recorded energy by two sensors $E1(n)$ and $E2(n)$.

3. Energy of acoustic emission sources and evaluation of attenuation B

3.1 Recorded AE signal energy vs. source energy

It is generally accepted that the energy of an AE signal includes the energy released by the source at crack initiation. Various parameters affect recorded energy: distance of wave propagation, energy attenuation due to damage, coupling between sensor and material surface and sensor frequency response. Wave theory states that the energy of an acoustic wave decreases exponentially with the increase of propagation distance. Therefore, the following equation was proposed to describe the energy of recorded AE signals (for instance, at sensor 1) received from the source n :

$$E_1(n) = E_s(n) \cdot A_1 \cdot e^{-B(L+x(n))} \quad (1)$$

$E_s(n)$ is the energy released at source n in the form of elastic waves. Due to differences in coupling between sensor and material surface or in sensor frequency response, for a source located at equal distance, the sensors may record significantly different amounts of energy. Thus, A_1 is the proportion of source energy that is recorded by sensor 1. It is a constant characteristic of sensor. $L + x(n)$ is the distance of propagation from source n to sensor 1 ($2L$ being the distance between sensors). The attenuation coefficient B is related to the propagation medium, which is subjected to changes due to damage evolution.

3.2 Attenuation B

To evaluate energy attenuation, the ratio of AE signal energies recorded at both sensors is calculated for each source n . For an easier identification of attenuation coefficient B , $X(n)$ is defined as the natural logarithm of this ratio. From Equations (1), it comes:

$$X(n) = \log \frac{E_1(n)}{E_2(n)} = \log \frac{A_1}{A_2} - 2.B.x(n) \quad (2)$$

A_1/A_2 represents the relative effect of acquisition that is for source n the fraction of source energy recorded by sensor 1 with respect to that recorded by sensor 2. The second term indicates the effect of propagation, which depends on source location $x(n)$ and on attenuation coefficient B . Both parameters can be estimated from $X(n)$ for various sources (1, 2, ..., n , ...) since X is a linear function of x . Because attenuation is expected to vary with damage, parameters estimation is performed at successive time intervals. For a given time interval, values of $X(n)$ are observed in various space intervals in order to take into account uncertainties in AE sources localization.

Estimation of attenuation constants was performed as follows. Attenuation is evaluated for a given time interval using the median values of $X(n)$ in every space interval (width: 10 mm, overlapping: 5 mm). Each median value of $X(n)$ corresponds to a few hundreds AE sources located in the same space-time interval. The median values of $X(n)$ corresponding to the space intervals at both ends of the gauge length are discarded and a linear approximation is carried out. In both cases, the data points fit well a linear function (coefficient of determination R^2 greater than 0.98). Both attenuation parameters can therefore be determined, the coefficient of attenuation B from the slope of the linear fit. Each time interval corresponds to 2000 AE sources. At every increment of 500 sources, a new time interval is considered. The overlapping is set in order to accurately monitor evolution of both attenuation parameters.

4. Towards lifetime prediction

4.1 Release of AE sources energy under static fatigue loading

Figure 1 represents the cumulative AE sources energy under static fatigue for the test at 500°C - $\sigma/\sigma_r = 0.67$. The energy released during initial loading was not plotted. It accounts for more than 90% of the total energy and was attributed to matrix cracking. A significant energy release is observed at the beginning of static fatigue. The activity then decreased until approximately 50% of rupture time. Finally, the energy release exhibited acceleration prior to rupture. Consistent observations were performed on the 7 studied fatigue tests.

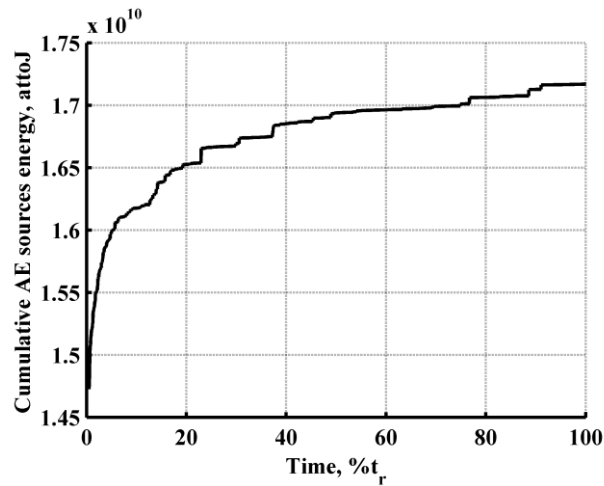


Figure 1. AE sources energy release under static fatigue. Test at 500°C - $\sigma/\sigma_r = 0.67$

4.2 Identification of attenuation parameters B

Attenuation was characterized using AE data recorded during 7 fatigue tests on woven SiCf/[Si-B-C] composites carried out 450°C and 500°C. Attenuation constants were estimated as indicated above. To ensure the relevance of every value of attenuation coefficient B and ratio of A_i , the coefficient of determination R^2 was calculated for every approximation. When a linear approximation had a value of coefficient of determination R^2 lower than 0.95, the associated values of both parameters were discarded. Values of R^2 lower than 0.95 were not observed in every test, and they appeared only for time intervals either at the beginning or at the end of tests. They were attributed to heterogeneous acoustic activity throughout the gauge length.

Figure 2 shows the evolution of attenuation coefficient B during the test carried out at 500°C - $\sigma/\sigma_r = 0.95$. The initial value corresponding to the first 2000 AE sources is around $1.7 \cdot 10^{-2} \text{ mm}^{-1}$. A slight increase is observed during initial loading up to maximum load at 0.2 hour. The specimen was then unloaded in order to monitor damage caused by initial loading. It results in a decrease of attenuation coefficient B down to nearly its initial value. Static fatigue loading began around 0.5 hour and a steep increase of attenuation coefficient B is observed up to 15 hours when the value reaches a plateau ($3.6 \cdot 10^{-2} \text{ mm}^{-1}$). The 6 other (Figure 3) tests exhibited a reproducible evolution of attenuation coefficient B with a significant increase between 10 and 50% of the rupture time (up to a factor of 2) and a plateau value beyond.

Attenuation coefficient B increases significantly during the first half of tests and reaches a plateau value at approximately 50% of the rupture time. If the growth of attenuation coefficient B is related to matrix crack opening, the plateau observed on the evolution of attenuation coefficient B indicates that matrix crack opening gets to an equilibrium state around 50% of the rupture time. The significant increase of matrix crack opening observed before 50% of the rupture time is attributed to oxidation of carbon in the interphase causing an increase in length of the debonded region of fibers in the vicinity of matrix cracks. Beyond 50% of the rupture time, the oxygen flux, determined by the degree of matrix crack opening, controls the rate at which fibers break by subcritical crack growth. Previous work showed that during static fatigue tests on SiC/SiC composites, the AE energy associated with sources

generated in the rupture zone exhibits critical features beyond 50% of the rupture time. This critical aspect corresponds to a second damage phase where subcritical crack growth in fibers is predominant, leading to ultimate failure of the composite.

Therefore, the monitoring of attenuation coefficient B provides a new indicator for damage monitoring of ceramic matrix composites. The characteristic evolution of attenuation coefficient B also allows considering the detection of the plateau as an indicator for lifetime prediction. The present methodology was applied here to AE data recorded during static fatigue tests carried out at 450°C and 500°C. However, it is worth noting that the methodology is directly applicable to tests conducted at higher temperatures. The only restrictive factor is the temperature to which the AE sensors are subjected and this is limited in the present acquisition set-up by placing the sensors away from the specimen's hot zone.

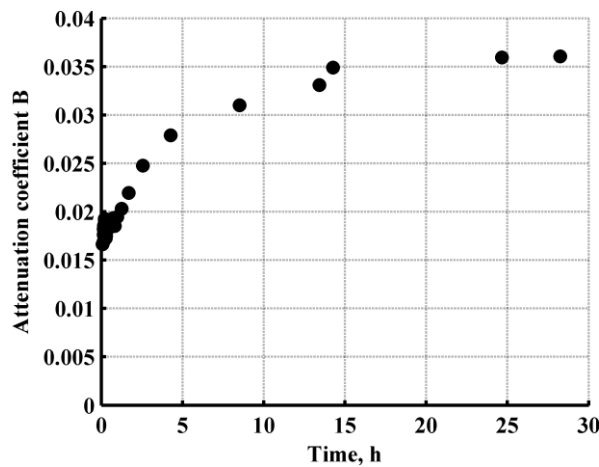


Figure 2. Test at 500°C - $\sigma/\sigma_r = 0.95$. Attenuation coefficient B vs. time throughout the fatigue test

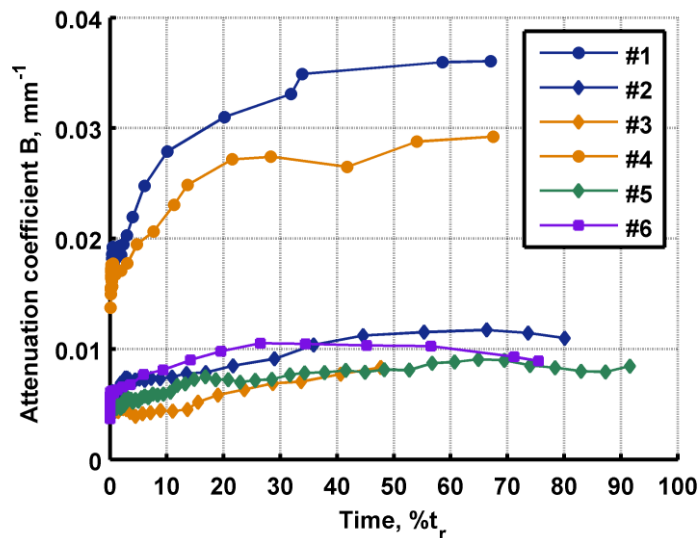


Figure 3: Attenuation coefficient B vs. time (in % rupture time)

5. CONCLUSION

This paper presents a method for the determination of AE energy attenuation based on AE energy of sources generated by material damage during fatigue tests. The effects of energy attenuation due to propagation distance, acquisition and damage accumulation are eliminated in order to accurately define the energy of AE sources. The energy release prior to rupture under static fatigue exhibits a critical evolution locally (± 15 mm around the rupture point) at 50% of rupture time regardless of the applied stress level. It is attributed to slow crack growth in the SiC fibers. Moreover, the characterization of energy attenuation will be studied with a view to damage monitoring. The method proposed in this paper can be used in real time and it allows the detection of a significant increase of attenuation during early phases of damage. Future work will aim at determining the contribution of each damage phenomenon on energy attenuation.

ACKNOWLEDGMENTS

The authors gratefully acknowledge SAFRAN Snecma Propulsion Solide and the CNRS for supporting this work as well as G. Fayolle for helpful comments.

REFERENCES

- [1] J. Lamon, A micromechanics-based approach to the mechanical behavior of brittle matrix composites, *Compos Sci Technol*, 61, 2259-72 (2001).
- [2] G. Fantozzi, P. Reynaud, Mechanical hysteresis in ceramic matrix composites, *Mat Sci Technol A*, 521-522, 18-23 (2009).
- [3] M. Moevus, D. Rouby, N. Godin, M. R'Mili, P. Reynaud, G. Fantozzi, G. Farizy, Analysis of damage mechanisms and associated acoustic emission in two SiC/[Si-B-C] composites exhibiting different tensile curves. Part I: Damage patterns and acoustic emission activity, *Compos Sci Technol*, 68, 1250-57 (2008).
- [4] M. Moevus, N. Godin, M. R'Mili, D. Rouby, P. Reynaud, G. Fantozzi, G. Farizy, Analysis of damage mechanisms and associated acoustic emission in two SiC/[Si-B-C] composites exhibiting different tensile curves. Part II: Unsupervised acoustic emission data clustering, *Compos Sci Technol*, 68, 1258-65 (2008).
- [5] S. Momon, N. Godin, P. Reynaud, M. R'Mili, G. Fantozzi, Unsupervised and supervised classification of AE data collected during fatigue test on CMC at high temperature, *Comp: Part A*, 43, 254-60 (2012).
- [6] W. Gauthier, J. Lamon, Delayed failure of Hi-Nicalon and Hi-Nicalon S multifilament tows and single filaments at intermediate temperatures, *J Am Ceram Soc*, 92, 702-709 (2009).
- [7] S. Momon, M. Moevus, N. Godin, M. R'Mili, P. Reynaud, G. Fantozzi, G. Fayolle, Acoustic emission and lifetime prediction during static fatigue tests on ceramic matrix composite at high temperature under air, *Comp: Part A*, 41, 913-18 (2010).
- [9] C. G. Bufe, D. G. Varnes, Predictive modeling of the seismic cycle of the Greater San Francisco Bay Region, *J Geophys Res*, 98, 9871-83 (1993).
- [10] E. Maillet, N. Godin, M. R'Mili, P. Reynaud, J. Lamon, G. Fantozzi, Analysis of Acoustic Emission energy release during static fatigue tests at intermediate temperatures on Ceramic Matrix Composites: towards rupture time prediction, *Comp Sci Technol*;72:1001-7, (2012).