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J. C. Sangleboeuf, Th. Bretheau, O. Sudre, D. Caldemaison. A quantitative mesoscale characterization of the mechanical behaviour of Ceramic Matrix Composites. *Key Engineering Materials*, 1997, 127-131, pp.721-728. 10.4028/www.scientific.net/KEM.127-131.721 . hal-00111579

HAL Id: hal-00111579

<https://hal.science/hal-00111579>

Submitted on 26 Aug 2019

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A Quantitative Mesoscale Characterization of the Mechanical Behavior of Ceramic Matrix Composites

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Keywords: CMC, In Situ Tension Tests, Micromechanics, Matrix Cracking

ABSTRACT

An experimental micro-macro kinematic description of matrix cracking in unidirectional ceramic matrix composites is proposed. It has been enlightened by observations performed during an *in situ* tensile test in a Scanning Electron Microscope. The characterization of matrix crack nucleation, propagation and coalescence has been done with new parameters and related to the macroscopic behaviour.

1. INTRODUCTION.

The different damage and failure mechanisms in ceramic matrix composites are now well known, and the critical role of interfaces has been demonstrated by numerous authors. Two classes of theoretical models have been proposed to predict the macroscopic mechanical behaviour of such materials but no model can encompass the complexity of the problem.

The first class of models combines a phenomenological description of the different matrix damage modes with a global approach by damage mechanics. For any given composite and after a relatively straightforward phase of parameter identification, these models find some use in structural designing [1]. However, the parameters in the models have seldom relation with physical composite characteristics [2]. Thus, no feed-back information is given to the material designer for further improvement of the composite properties.

The second starts with a fracture mechanics description of the physical events that have been well identified and characterised at the fibre scale and is pursued by extending this analytical or semi-analytical micromechanical model to a more macroscopic description [3]. This approach is meaningful for the material designer but gets useless for the structural designer due to the complexity induced by multidirectional fibre architectures and multiaxial loading.

In the present study we try to bridge the gap between these two descriptions by performing experiments at a mesoscale to quantify damage kinetics in the composite and identify some physical parameters meaningful for both classes of models. The mechanical testing technique consists in *insitu* tensile tests in a Scanning Electron Microscope (SEM). Some new parameters have been defined so as to explain the matrix cracking evolution.

2. EXPERIMENTAL PROCEDURE.

2.1. Material.

All experiments were conducted on a unidirectional laminate of SIC:MAS provided by ONERA. This material is made of 27% Nicalon NLM202 fibre in a MLAS matrix (ref : MLAS SG-P2 Saint-Gobain Recherche).

2.2. Specimens.

Tensile specimens 100 x 2 x 4,8 mm³ were prepared by machining 100 x 100 x 4,8 mm³ plates with a diamond blade. The matrix layer at the plate faces was removed by polishing; the two other faces were as well polished. An Au/Pd thin layer was sputtered on the gauge to facilitate SEM observations.

2.3. Testing.

Ex situ tensile tests, monotonous and repeated, were performed on a servomechanic machine at a $2.4 \cdot 10^{-5} \text{s}^{-1}$ nominal strain rate. Special attention was given to avoid any bending. The specimens were directly glued and clamped into the grips what leaves an effective 20mm gauge length for testing. Strain was measured by means of an extensometer attached to the gauge section for accuracy. Stress/strain curves allow to identify macroscopic parameters such as : initial modulus E_0 , tangent modulus E_t , permanent strain ϵ_p , maximum opening of hysteresis loop ϵ_{max} . (fig1).

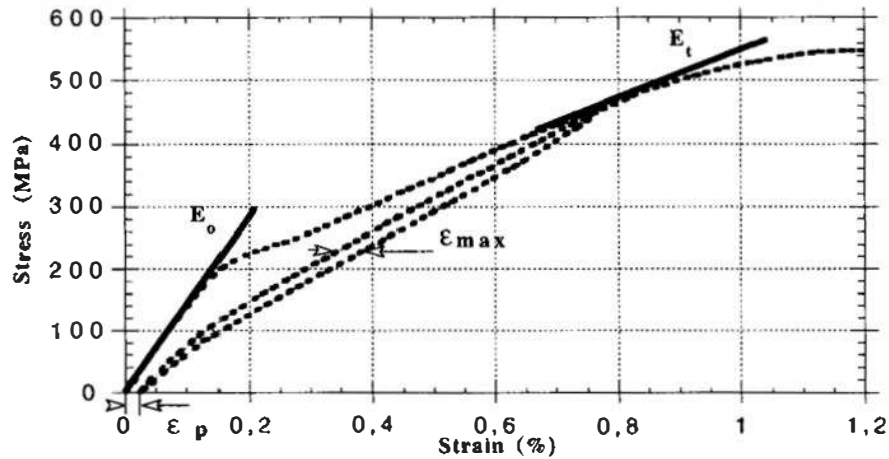


fig 1 :Typical stress/strain curve and definition of macroscopic parameters.

The choice of these parameters will be enlightened during meso/macro correlation.

In situ tensile test under in the SEM were performed at a nominal strain rate of $2,5 \cdot 10^{-5} \text{s}^{-1}$. *Ex situ* and *in situ* strain rates are very close so as to ensure a time independent interpretation. Panoramas of the whole sample surface, $2 \times 20 \text{ mm}^2$, and enlargement of some typical areas were worked out at several stress level so as to study the onset and the evolution of matrix cracks, and also to get the evolution of the crack opening in connection with its neighbourhood.

Mesoscopic observations can be related to macroscopic informations with reference to the corresponding stress level.

3. EXPERIMENTAL RESULTS.

This study exhibits three scales :

- macroscale : the Representative Volume Element (RVE), characterised by the evolution of global parameters such as the tangent modulus.
- mesoscale : several fibres inside the matrix, characterised by matrix cracking.
- microscale : a single fibre inside the matrix, characterised by interfacial decohesion, friction, and fibre failure.

A central point for the correlation between two scales, is the choice of an indicator at a scale that is representative of the kinetic of evolution of the pertinent mechanisms active at a lower scale. The evolution of several parameters will be studied in the following.

3.1. Macroscale.

The composite presents a non-linear behaviour before failure, with a "plateau" at the beginning of the non linear stage. Permanent strains and quite large hysteresis loops characteristic of dissipation phenomena can be noted. (fig 2).

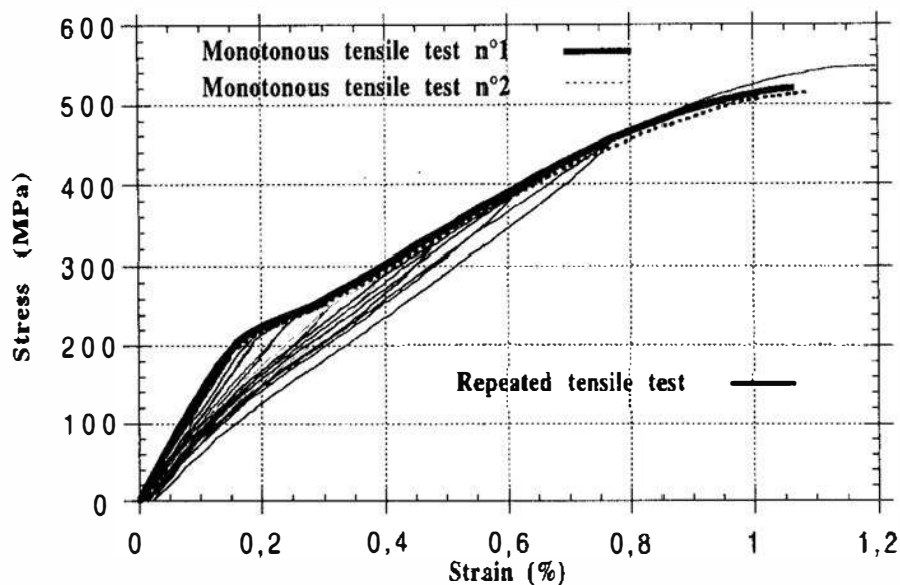


fig 2 : Stress/strain curves.

The behaviour is highly reproducible either under monotonous or repeated tensile conditions; nevertheless the strain to failure is always higher for repeated tests.

A special attention was paid to the tangent modulus E_t since its evolution is characteristic of the rate of damage and dissipation mechanisms but not of the present state of these mechanisms. Thus we have access to informations concerning the rate of the mechanisms through the absolute value of $dE_t/d\sigma$, the sign resulting from the competition between these mechanisms (fig 3).

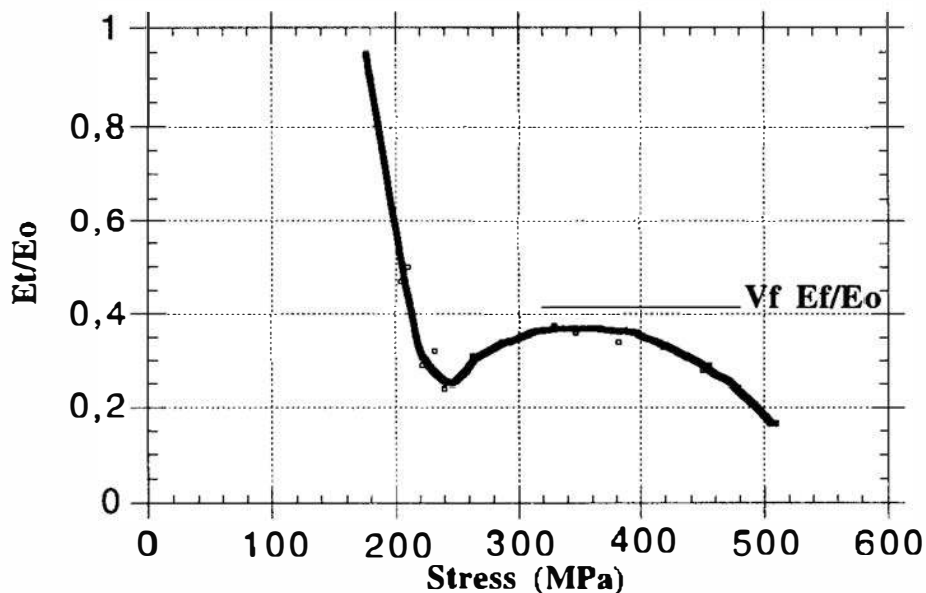


fig 3 : Evolution of E_t/E_0 vs. the applied stress.

This particular shape has already been reported by Spearing and Zok [4].

The behaviour can be divided in four stages : a rapid decrease between 0 and 180MPa, then a minimum between 180 and 240MPa, an increase passing by a maximum between 240 and 380MPa and a slower decrease between 380 and 520MPa.

These stages can also be observed in the evolution of two other macroscopic parameters ; the maximum opening of hysteresis loops (ϵ_{max}) for the dissipative aspect of the behaviour, and the permanent strain (ϵ_p) for the irreversible aspect (fig 4).

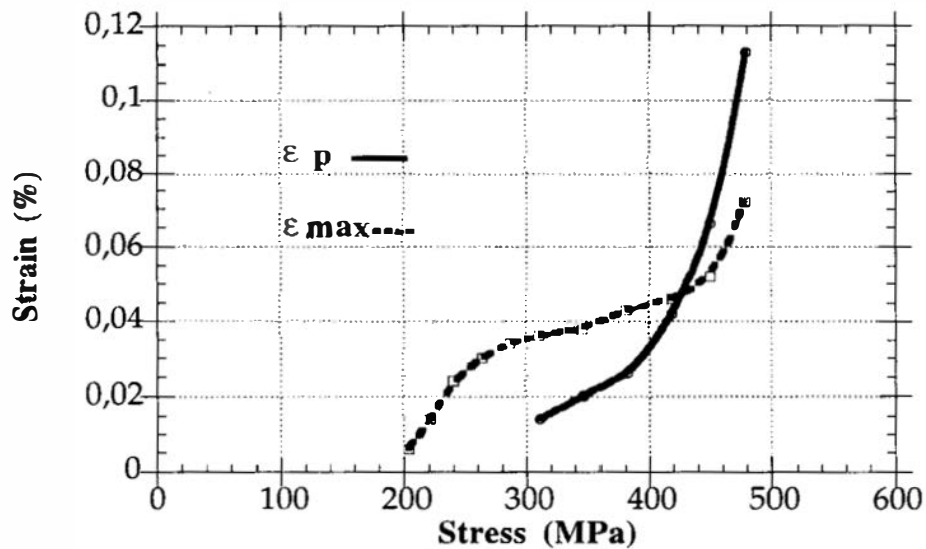


fig 4 : Evolution of ϵ_p and ϵ_{max} .

3.2. Mesoscale.

Matrix cracks can be observed at low magnification; they are quite tortuous due to microstructural heterogeneities (variation in the local fibre volume fraction) and cracks interaction. This interaction shows two characteristic distances: the first one is lower than tortuosity (short range screen effect); the second one has a magnitude equivalent to the average distance between cracks (large range screen effect).

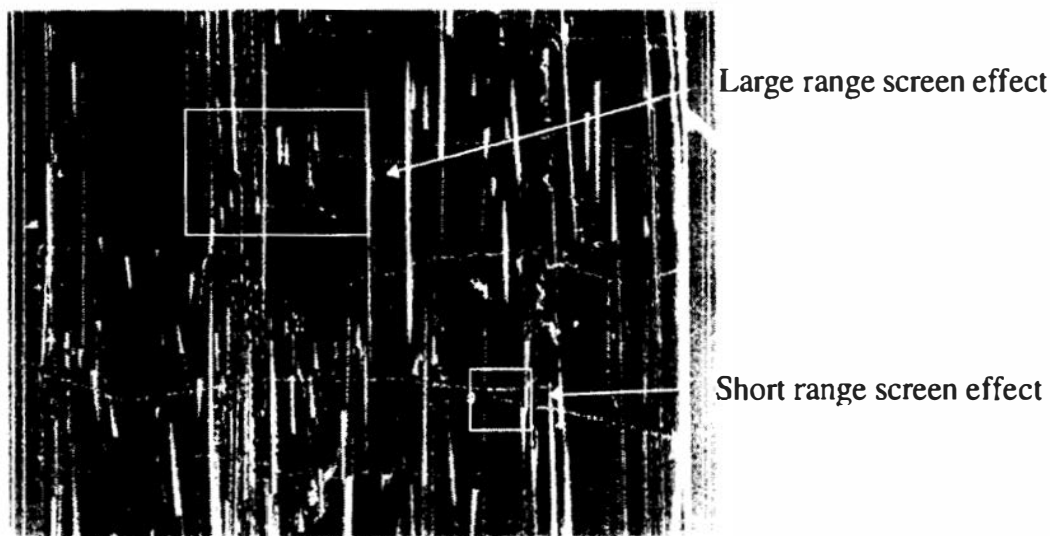


fig 5 : Matrix cracks observed at mesoscale (x55) under 255 MPa stress.

It is very important to take into account these screen effect phenomena if we want to define what is the dimension of a crack at such a scale. When cracks are not in the same plane, their identification is easy. But, when cracks are roughly in the same plane and show a short range screen effect, we will consider that two cracks coalesce when they are physically connected (when there is a real crack path) and when secondary branches are deactivated. For example, on figure 5, the two cracks presenting a short range screen effect have not coalesced; they are connected but the secondary branch is still open (otherwise it could not be observed).

With this definition of a crack, it is interesting to follow the evolution, in relation with the applied stress, of three parameters representative of matrix cracking (fig 6) :

- the linear density D_{lin} , measured by the intercept method and classically used,
- the total density D_{tot} (total number of cracks/specimen length),
- the average length of crack, L_{av} .

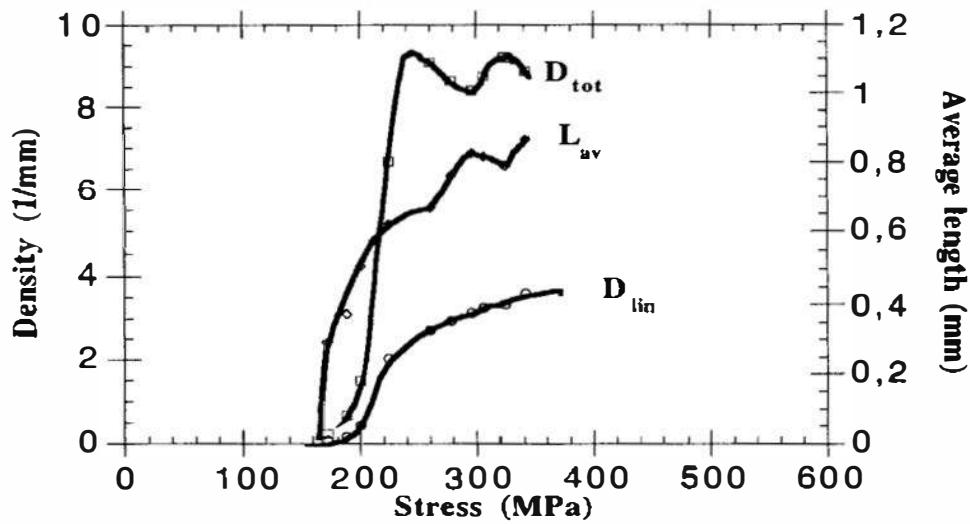


fig 6 :Evolution of D_{lin} , D_{tot} and L_{av} .

These three curves (fig 6) show unambiguously that the matrix crack initiation stress is about 150Mpa; the stress corresponding to the saturation of D_{lin} can be considered as the matrix crack saturation stress and evaluated at 400Mpa; D_{lin} increases monotonously with the applied stress; this evolution has induced some authors [5-7] to propose that matrix cracking develops in two phases: first the unstable creation of cracks gives rise to a regular pattern; then, these cracks propagate in a stable manner throughout the entire section of the specimen.

Nevertheless, the evolution of D_{tot} leads to moderate the previous interpretation, by accounting with the oscillatory behaviour of matrix cracking. The rapid evolution of the number of cracks for stresses between 150 and 240MPa corresponds to the nucleation of the first generation of cracks, just followed by a coalescence stage; then the evolution is made of nucleation and coalescence steps with a lower amplitude compared to the initial burst. This decrease in amplitude occurs because less and less defects can nucleate new cracks due to the stress relaxation induced by the cracks already present in the composite. Moreover, the evolution rate decreases too; this could be a consequence, at a local scale, of the short range screen effects (fig 7). Thus, for a particular crack, the plot of the crack opening displacement vs. stress shows the effect of its neighbourhood on its propagation: a secondary crack, nucleated around 180Mpa, induces a deceleration of the principal crack; the arrest of the secondary around 210Mpa allows a re-acceleration of the principal one (fig 8).

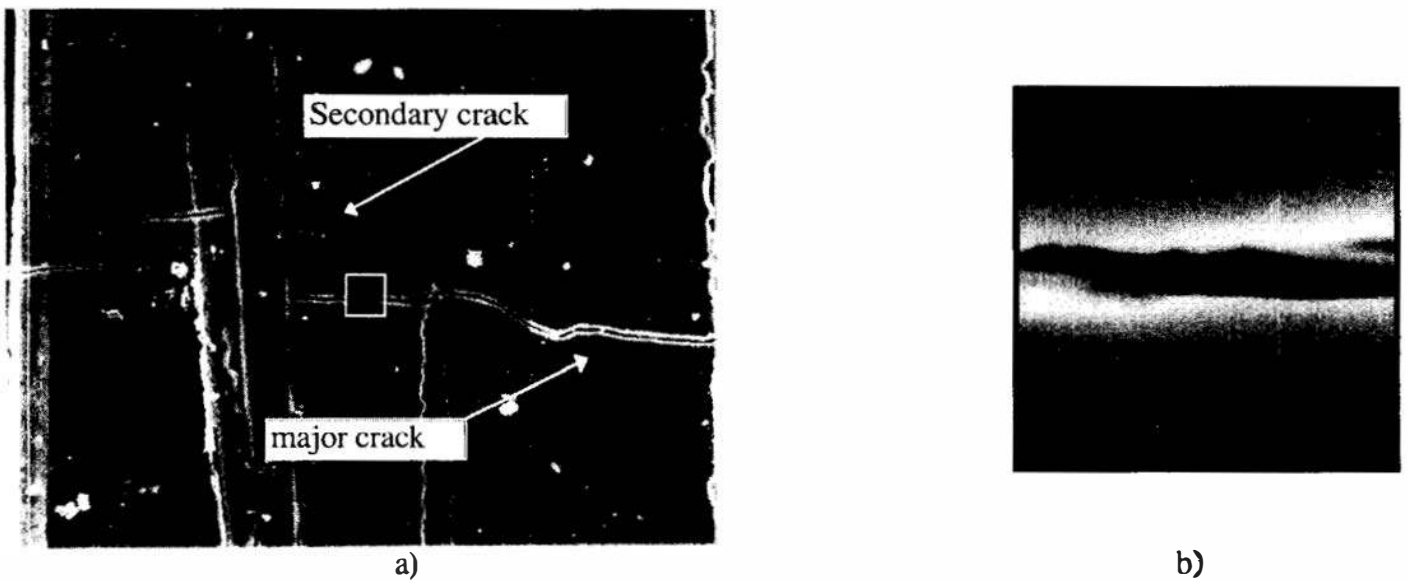


fig 7 : a) Local screen effect (x650).
b) Crack opening (x17000).

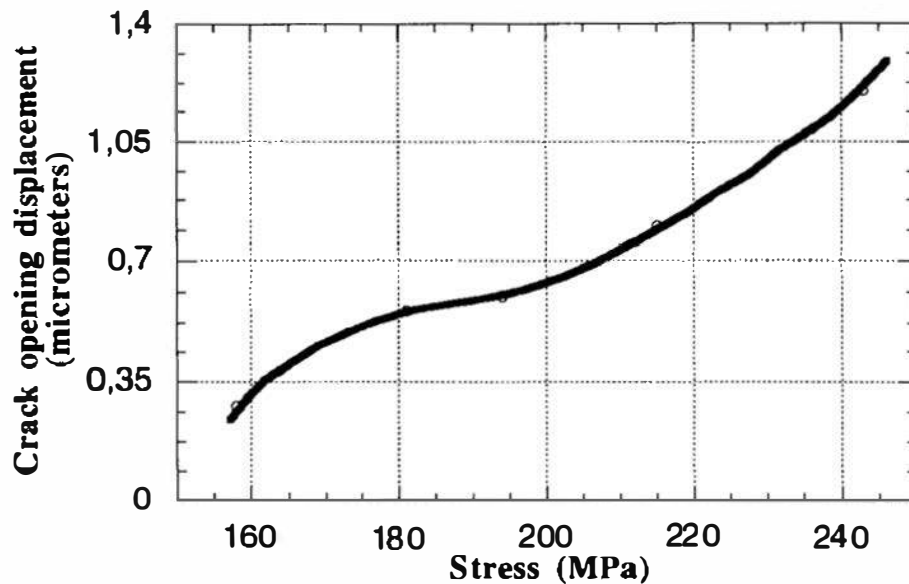


fig 8 : Evolution of the crack opening displacement for the principal crack of figure 7.

Moreover, this oscillatory behaviour of matrix cracking is confirmed by the evolution of L_{av} (fig 6). This parameter decreases with the creation of new short cracks, and increases during coalescence.

Therefore it can be concluded that matrix cracking begins by a nucleation stage (150 - 250MPa), followed by coalescence/nucleation stages until the applied stress reaches 400MPa. The control mechanism is crack propagation, but nucleation and coalescence disturb the regularity of the evolution.

4. MESOMACRO CORRELATIONS.

The decrease of E_r/E_0 between 180 and 240MPa (fig 3) seems to be clearly due to the initial nucleation stage ranging from 150 and 240MPa. The rapid nucleation of cracks bridged by fibres leads jointly to increase the friction surface at fibre/matrix interfaces; this is confirmed by the increase of ϵ_{max} between 190 and 260MPa (fig 4). The inflexion point at 240MPa corresponds to the transition between monotonous and oscillatory regimes in the evolution of the total density.

The increase of E_r/E_0 in the range 240 - 380MPa is correlated with the oscillatory regime where matrix cracking is controlled by crack propagation. Moreover, the evolution of ϵ_{max} between 280 and 400MPa seems to indicate that matrix cracking reaches saturation on a stable manner. For applied stress ranging from 340 to 380MPa, the curve representing the evolution of E_r/E_0 is under $\sqrt{f}E_r/E_0$, which corresponds to the stiffness of the only fibres of the composite (fig 3). In that stress range, matrix cracking is near saturation and it can be considered that load is supported by fibres only. Under the reasonable hypothesis of a total fibre/matrix decohesion of a weak interfacial shear stress, the proximity between the residual stiffness and the theoretical one induces that only a few fibres have failed at that stage.

The decreasing stage of E_r/E_0 between 380 and 500MPa seems to be correlated with fibre failure: this failure causes a decrease in the elastic energy stored in the bridged cracks; during unloading they consequently close less allowing an increasing permanent strain ϵ_{max} (fig 4). All these correlations are confirmed by acoustic emission during tensile tests; it begins to be very active around 350MPa.

The increase in strain to failure under repeated loading, compared to monotonous one, can be interpreted owing to the observation of the fibre/matrix interphase (fig 9); it shows, after only a few cycles, a damaged aspect with debris; that damage can weaken the interfacial shear stress and promote the extension of the interfacial cracks and then diffuse the stress concentrations associated with matrix cracks; a more homogeneous stress state could delay the specimen failure.

5. CONCLUSION.

In situ tensile tests inside a scanning electron microscope have allowed to give a more detailed description of matrix cracking by means of three predominant mechanisms: nucleation, propagation and coalescence. The classical linear density parameter has proved to be insufficient to give a precise enough description of the complex reality of matrix cracking kinetics; two new parameters have been proposed: the «total crack density» and the «crack average length». On the basis of these parameters and of an interfacial friction law, now in experimental identification, a micro-meso correlation is to be performed.

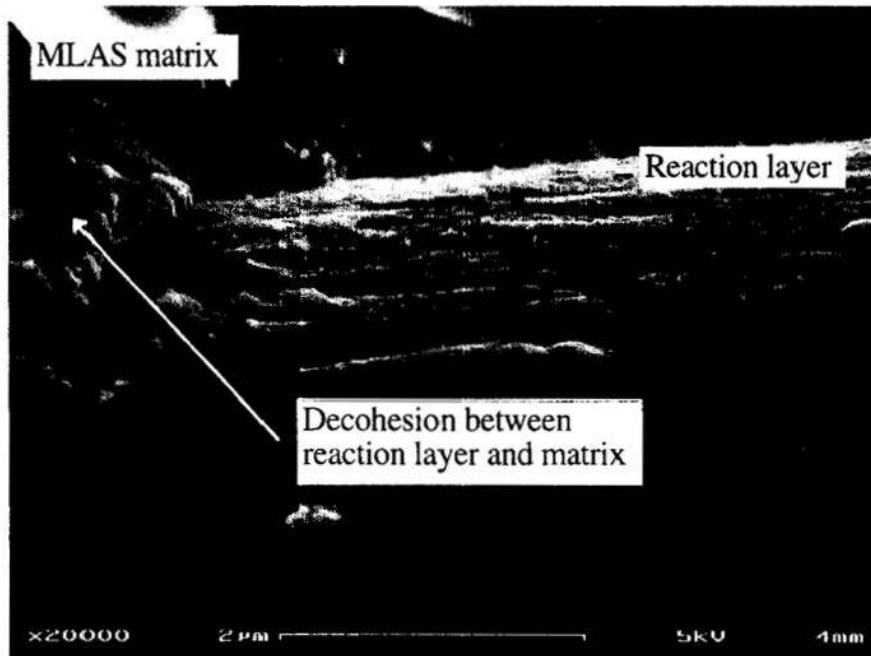


fig 9 : Fibre/matrix interphase observed near the fracture surface after seven tension-tension cycles.

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