

Approach to microstructure-behavior relationships for ceramic matrix composites reinforced by continuous fibers

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Abstract. Ceramic matrix composites (CMCs) reinforced with continuous fibers exhibit several features that differentiate them from homogeneous unreinforced materials. The microstructure consists of various distinct constituents: fibres, matrix, and fiber/matrix interfaces or interphases. Several entities at micro- and mesoscopic length scales can be defined depending on fiber arrangement. Furthermore, the CMCs contain flaw populations that govern matrix cracking and fiber failures. The paper describes the microstructure-behavior relations for ceramic matrix composites reinforced with continuous fibers. It focuses on matrix damage by multiple cracking, on ultimate fracture, on delayed fracture at high temperatures, and on stochastic features induced by flaw populations. Models of damage and ultimate failure are based on micromechanics and fracture probabilities. They provide a basis for a multiscale approach to composite and component design.

1. Introduction

The microstructure of a material can influence strongly physical properties such as strength, toughness, ductility, high temperature behavior, which govern the application of these materials in industrial practice. Because microstructures are complex ensembles of materials, few researchers have attempted to correlate microstructure with properties [1]. Instead, average material data are used to produce approximate models for material behaviour. In some cases, mean-field models work well, but we can't expect them to be predictive for cases when properties exhibit statistical distributions, or depend on microstructural features or environment.

Several entities at micro- and mesoscopic length scales can be defined depending on fiber arrangement: single filament-interface-matrix assemblies (represented by microcomposite test specimens), tow-reinforced matrix in woven composites (represented by minicomposites), unidirectional plies in laminated composites (Fig. 1).

CMCs are highly heterogeneous as they are made of constituents with distinct shapes and properties: fibres, matrix, fiber/matrix interfaces and interphases. They also contain various flaws. Being damage tolerant, they can include cracks in the matrix, in the interfaces and in the fibers. They are versatile, which implies that some properties can be tailored by selecting appropriate constituents, which is made possible by the broad spectrum of potential materials. The stress-strain response to load is dependent

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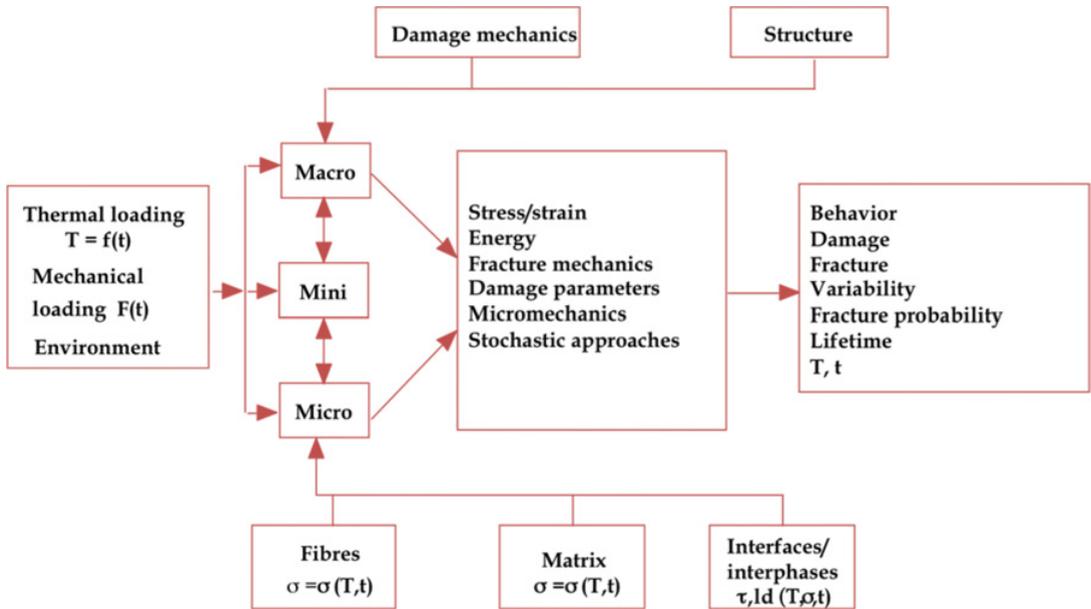


Figure 1.

on constituent properties: it is linear or non linear depending on contrast in elastic properties and fracture characteristics of constituents. The constituents are ceramic materials. As such, they exhibit statistical distributions of strength data, as a result of inherent sensitivity to microstructural flaws with random distribution. The flaw populations govern matrix cracking and fiber failures. Thus, description of microstructure-behavior relations using a bottom-up multiscale approach to the mechanical behavior is essential to understand the phenomena at low length scale that influence the behavior at macroscopic scale. It can be the basis of an approach to composite and component design.

The paper describes the microstructure-behavior relations for woven composites. Woven SiC-based CMCs are promising materials and they are used in systems running at high temperatures. They provide excellent examples to discuss the microstructure-behavior relationships in CMCs. 1D composites suffer several limitations as a result of high anisotropy.

2. Multiscale approach

Figure 1 presents a chart of the multiscale approach that is underlying research work on the behaviour of CMCs reinforced by continuous fibers that has been initiated in 1990. It summarizes the loading cases, the concepts and approaches on which were founded the models of composite response that were developed, and the characteristics of mechanical behaviour that were determined or predicted.

3. Composite mechanical behavior

3.1 Tensile stress-strain behavior of composites reinforced by continuous fibers

Ceramic matrix composites exhibit an elastic damageable behavior (Fig. 2). This means that the response of damaged composite is elastic, as indicated by the initial linear deformations on reloading. The typical stress-strain curves for 2D CVI SiC/SiC composites shown on Fig. 2 summarize trends in the mechanical behavior. This composite behaves linearly to a strain of about 0.03%, and then it exhibits a curved

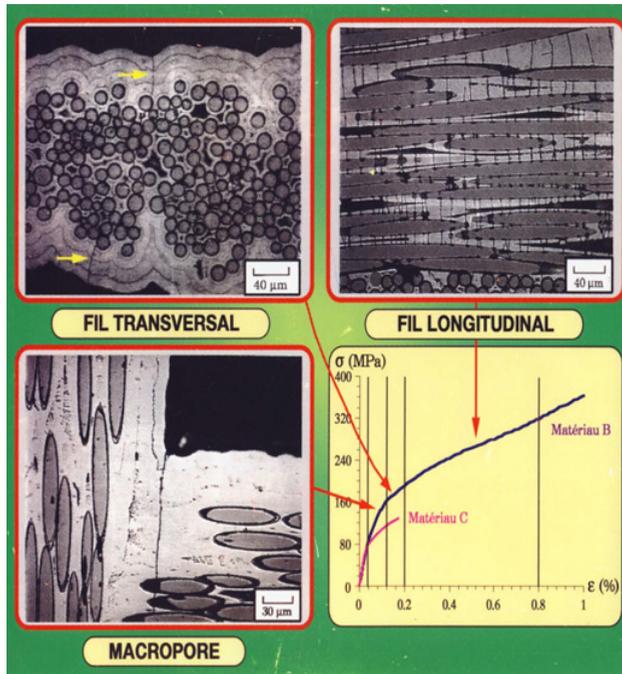


Figure 2.

domain as a result of matrix cracking. Saturation of matrix damage is indicated by a point of inflection (Fig. 2). Then the ultimate portion of the curve reflects the deformation of fibers and individual fiber breaks.

Matrix damage reflected by nonlinear deformations involves multiple microcracks or cracks, perpendicular to fiber direction, and that are arrested by the fibers at fiber/matrix interfaces. In the composites reinforced with fabrics of fiber bundles, matrix damage is influenced by multi length scale structure [3, 4]. In 2D SiC/SiC and 2D C/SiC, damage consists of transverse cracks located, in a first step, between the longitudinal tows and deflected by the tows; in a second step, within the longitudinal tows and deflected by the filaments. In 2D C/SiC, the first step of damage initiates during cooling down from the processing temperature as a result of large Carbon fiber coefficient of thermal expansion when compared to SiC matrix. Damage modes in CVI SiC matrix composites have been identified using in-situ microscopy during tensile tests and analysis of acoustic emission signals.

In dense 2D composites without macropores, the first cracks initiate in the transverse tows. The directions of principal stresses are dictated by fiber orientations. Under axial tension, all the matrix cracks are perpendicular to loading direction. Under off-axis tension, those matrix cracks located in the tows are perpendicular to fiber direction, whereas those located between the tows are perpendicular to the load direction.

The damage phenomenon is a top down (from large to small volumes of material), multi length scale, and sequential process. The fibers in the loading direction are loaded progressively, towards a tow-controlled behavior.

3.2 Ultimate failure

Matrix damage and ultimate failure are successive phenomena. Ultimate failure generally occurs after saturation of matrix cracking, when the load is carried totally by the longitudinal tows. In that case,

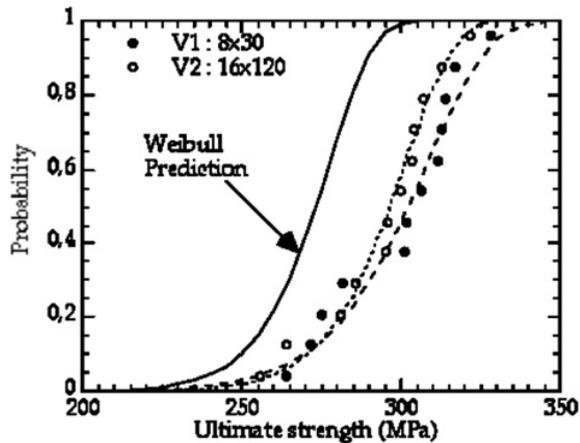


Figure 3.

the strains-to-failure of composite and tows are very close: $>0.8\%$ for most SiC-based tows (Nicalon and Hi-Nicalon at least [3]). This shows that fiber weaving has a negligible influence. The analysis of acoustic emission signals indicate that fracture of filaments starts at strains $>0.6\%$. Lower composite strain-to-failures indicate premature failure as a result of filament interactions (like contacts or too strong interfaces).

The *ultimate failure of a tow* of parallel fibers involves two steps:

- a first step of stable failure, and
- a second step of unstable failure.

During the first step, the fibers fail individually under increasing load. In the absence of fiber interactions, the load is carried by the surviving fibers only (equal load sharing). Fiber interactions cause tow weakening leading to premature failures. The ultimate failure of a tow (second step) occurs when a surviving fiber cannot tolerate the load increment resulting from a fiber failure. This particular filament that initiates instability is designated the critical fiber. It fails after the failure of 12–17% of the filaments present in a tow, for Nicalon and H-Nicalon fibers and more generally for those fibers with Weibull modulus $5 < m < 8$ [3, 4]. The statistical distributions of SiC Nicalon or Hi-Nicalon filament failure data show that the lowest strain-to-failure is about 0.5%.

The *ultimate failure of a longitudinal tow coated with matrix* results from the same two-step mechanism and involves global load sharing when a fiber fails. In the presence of multiple cracks across the matrix and associated interface cracks, the load carrying capacity of the matrix is tremendously reduced or annihilated. The matrix-coated tows behave like dry tows under the stress state including shear lags induced by matrix cracks. The ultimate failure of a matrix-coated tow occurs when a critical number of fibers have failed. This mechanism operates in the tows within textile CVI SiC/SiC composites. The ultimate failure of composite is caused by the failure of a critical number of tows (≥ 1), depending on the stress state: ~ 1 under uni-axial tension, > 1 in bending.

As a result of the process of fibre flaw population truncation during stable fracture, the population of critical flaws get reduced [5]. As a consequence, composite strength data exhibit a limited scatter and an insignificant dependence on stressed volume and loading conditions (Fig. 3). Thus, the flexural strength is 1.15 times as large as the tensile strength [5, 6].

The Weibull model does not describe the volume dependence of strength data [5, 7], since the weakest link concept is violated due to the presence of cumulative damage process. However, the Weibull modulus (m) that can be extracted from the statistical distribution of strength data can be

regarded as an indicator of scatter. m values in the range 20–29 have been obtained for 2D woven SiC/SiC composite. Such values reflect a small scatter.

The stochastic nature of the stress-state, as a result of the presence of flaws, heterogeneities and cracks, is also a feature that needs to be taken into account at macroscopic scale. Detail on the model that predicted satisfactorily bending strengths from tensile data can be found in [7].

4. Modelling of stress-strain behavior

4.1 Stochastic model of matrix fragmentation in 1D composite and minicomposite

Modelling of fragmentation is critical to the determination of composite non-linear stress-strain behaviour and ultimate failure, the development of optimized composites and the design of structural components.

In stress-based approaches, the stress on fragmenting constituent is compared to the strength of elements (chain-of-segment model [8]), or fragments (fragment dichotomy model [8]). These models are based on extreme value theory. They can be integrated into 2D or 3D architectures. The chain-of-element model is based on Bayesian statistics and flaw strength density function. It has been developed recently [9]. It can be extended to 2D and 3D reinforced composite.

a) The chain-of-segment model

The chain-of-segment model has been widely used in the Monte Carlo simulation of fragmentation of fibers in a polymer or metal matrix. The distribution of element strengths is constructed from the weakest link failure of independent fibers. Fiber elements as well as a single fiber tension tested at arbitrary gauge length follow a similar Weibull distribution (extreme value statistics):

$$P(\sigma, L) = 1 - \exp \left[-\frac{L}{L_o} \left(\frac{\sigma}{\sigma_o} \right)^m \right] \quad (1)$$

where L designates the length of either the element or the fiber.

In Monte Carlo simulations, a fiber length L is subdivided into N_e segments, each of length $L/N_e \ll L$. Each segment is randomly assigned strength according to the Weibull cumulative probability distribution derived from (1):

$$P \left(\sigma, \frac{L}{N_e} \right) = 1 - \exp \left[-\frac{L}{N_e L_o} \left(\frac{\sigma}{\sigma_o} \right)^m \right] \quad (2)$$

A uniform stress σ_f is gradually applied along the entire fiber length, and a break is formed at a segment when the stress on the segment equals the assigned strength of that segment. Comparison with available experimental fragmentation results is very sensitive to the statistical parameters introduced in Eq. (2). Agreement was not obtained with those parameters derived from the failure of independent fibers with gauge length L [10, 11]. Drawbacks associated to fiber strength distribution dependence on length and statistical fiber strength characteristics have been addressed in [12]. Other experimental results showed that predictions are satisfactory only for low stresses quite far from the saturation of fragmentation [10].

The process of fragmentation is not addressed correctly: an in-parallel arrangement is implicitly considered instead of an in-series one, weakest link statistics are used instead of Bayesian statistics and the underlying flaw strength distribution refers to the weakest flaws instead of the whole population of flaws present in the material.

b) The fragment dichotomy model [4, 8, 9]

The fragment dichotomy model considers fragments (instead of identical segments) of decreasing size as fragmentation proceeds. It reproduces the process of fragment generation. Each fragment results from the failure of a parent fragment. Failure is caused by the most severe flaw in the parent fragment. The fracture inducing flaw corresponds to the low extreme of the flaw strength distribution pertaining to the parent fragment. This model predicted satisfactorily the distributions of fragment strengths for the SiC matrix in SiC or C fiber reinforced minicomposites, as well as the tensile stress-strain behaviour of minicomposites [4, 8, 13].

c) The Bayesian model

The Bayesian model of fragmentation is the exact solution when fragmentation is assimilated to progressive multiple failure of a chain of elements. Failure of a link in a chain which can experience successive failures involves conditional probabilities since failure of a link depends on the response of the other links. Probability of the k^{th} failure is derived from the following basic equation of conditional probabilities for the k^{th} failure:

$$P(\bar{k} - 1 \cap k) = \bar{P}_{k-1} \cdot P(k/\bar{k} - 1) = P^{\bar{k}} - P^{\bar{k}-1} \quad (3)$$

The subscript $\bar{k} - 1$ designates the event of non failure for the $(k - 1)^{\text{th}}$ link, k the event of failure for link k . \bar{P}_{k-1} is the probability of no failure of link $k - 1$, P_k is the probability of k^{th} failure.

From (3) it comes:

$$P(k/\bar{k} - 1) = 1 - \frac{1 - P_k}{1 - P_{k-1}} \quad (4)$$

Note that Eq. (4) represents the operation of truncating the flaw strength distribution at probability P_{k-1} , to obtain the distribution of unbroken links after the $(k-1)^{\text{th}}$ failure. This is the pertinent distribution for the surviving links that will experience failure under increased load. P_{k-1} and P_k pertain to the non truncated flaw strength distribution. $P(k/\bar{k} - 1)$ corresponds to the truncated distribution after the $(k-1)^{\text{th}}$ failure. For simplicity, $P(k/\bar{k} - 1)$ will be referred to as P^k .

The following equation for the stress at formation of the k^{th} crack was established in [8]:

$$S_k = S_0 k^{1/p} \left(\frac{V_o}{V} \right)^{1/p} [-Ln(1 - P_z)]^{1/p} \quad (5)$$

Where p and S_0 are statistical parameters. P_z may be taken to be 1%, 0.1% or less, depending on the flaw population size. P_z may be also selected randomly, or k dependent, but it must remain smaller than a certain limit. To some extent, this limit depends on the number of flaws in the distribution. It is assumed that the number of remaining flaws is much larger than 1 so that statistics may apply.

Good predictions of fragmentation strengths (Fig. 4) were obtained with the fragment dichotomy model and the Bayesian model [15]. By contrast, the fragmentation stresses were either overestimated (low stresses) or overestimated (high stresses) by the Monte Carlo simulation method. The upper and lower bounds show that predictions exhibit a significant uncertainty. Although it presents flaws in its foundation, the Monte Carlo simulation method is generally used for the treatment of multiple failures in a chain of links. It may provide a rough approximation of the low fragmentation stresses when the number of elements is similar to the number of fragments at saturation. However, this requirement cannot be met for prediction purposes, since the number of fragments is one of the outputs of analysis. Using larger amounts of elements leads to high variability in predictions, overestimation of most of fragmentation strengths, and misestimation of fragmentation stresses (Fig. 4).

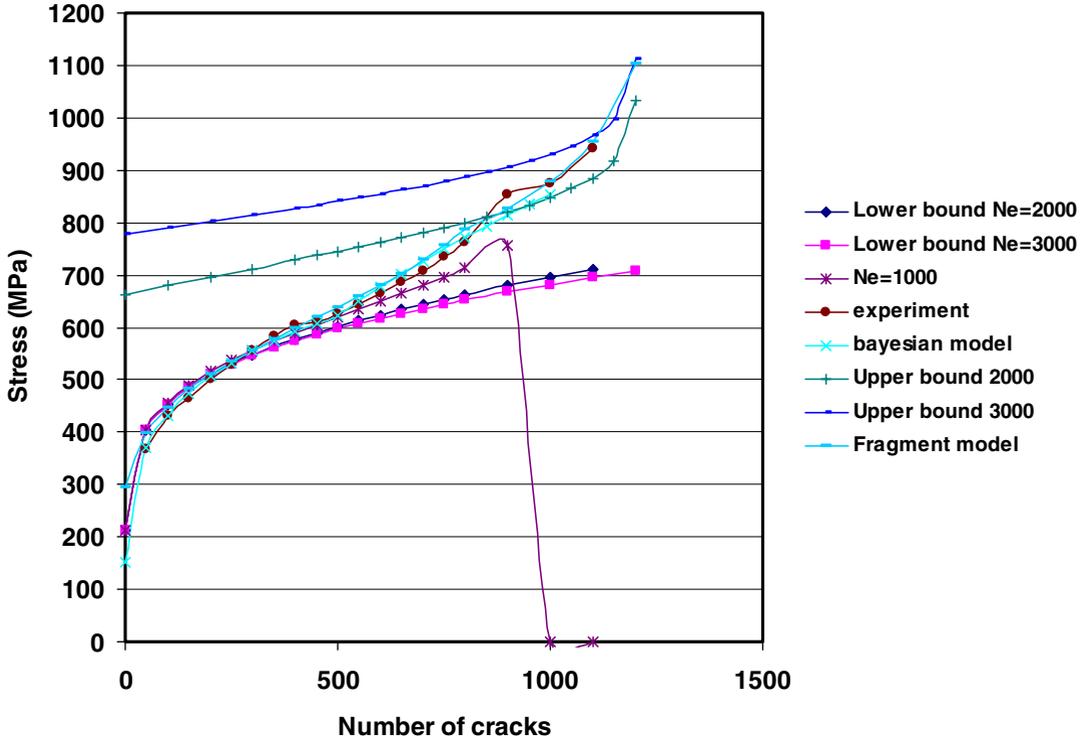


Figure 4.

In [9] the Bayesian model was extended to non-uniform and multiaxial stress-states and applied to 2D reinforced composites by using the multiaxial elemental strength.

4.2 Ultimate failure in 1D composite and minicomposite

Ultimate failure is derived from the survival probabilities of fibre volume elements delineated by matrix fragments [4]. Global load sharing is a realistic assumption, as discussed in a previous section on ultimate failure. Equivalent fibre length (L_{equi}) is defined as the length subjected uniformly to the peak stress at the failure probability for the actual stress state induced by cracks in the matrix and in interface. Overlapping of interface cracks is taken into account ($l_i < l_{di}$). Failure probability of a fibre within minicomposite is given by the following equation:

$$P_f = 1 - \exp \left[- \left(\frac{\sigma_{f \max}}{\sigma_{of}} \right)^{m_f} A_f L_{\text{equi}} \right] \quad (6)$$

$$\text{With } \sigma_{f \max} = \frac{F}{A_f^t (1 - \alpha)} \quad (7)$$

F is the applied force, A_f is the cross sectional area of a single filament, A_f^t is the tow cross sectional area, α is the fraction of individual fiber breaks (the probability of the N^{th} filament). Ultimate failure of minicomposite results from instability in the evolution of individual fibre failures. It is characterized by $\alpha = \alpha_c$ or $\delta F / \delta \alpha = 0$. Solving Eq. (6) requires a numerical analysis to determine the spatial distribution of matrix cracks and the associated stress state [4]. Simple closed form expressions can be obtained

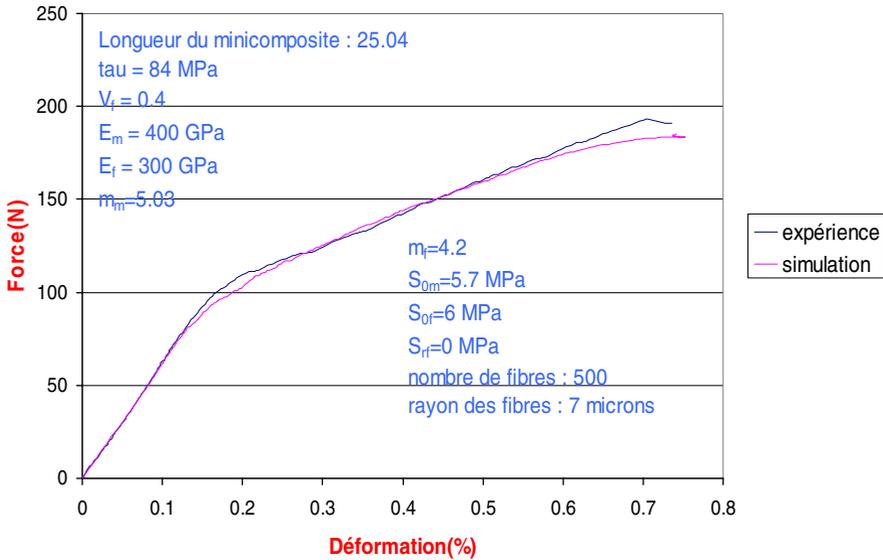


Figure 5.

when there is no overlapping of interface cracks. These equations allow detailed understanding of fiber fracture.

4.3 The stress-strain behaviour of 1D composites and minicomposites

The tensile stress-strain behaviour of composites is dictated by fiber deformations and failure. Deformations are determined from the stress-state in fibers induced by the number of fragments:

$$\varepsilon = \frac{1}{LE_f} \int_L \sigma_f(x) dx \tag{8}$$

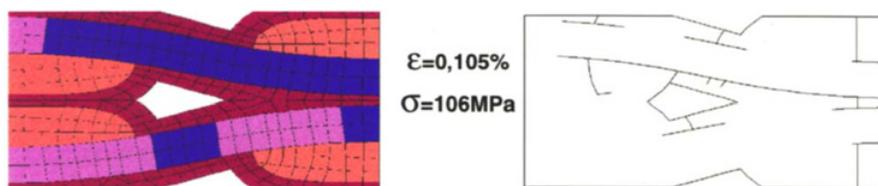
with L = fiber length.

Figure 5 exemplifies the satisfactory predictions of stress-strain behaviour which have been obtained on various minicomposites, using the fragment dichotomy model [4, 8, 13, 14]. The characteristics of constituents required for computations include intrinsic properties (the thermoelastic constants) and fracture parameters (debonded interface characteristics and statistical parameters). They were measured independently. Thermally induced stresses arise during cooling down from the processing temperature in the C/SiC minicomposites shown here. The resulting multiple cracking is responsible for the initial non-linear part of the stress-strain curve.

The model can be used for composite design as well as for the investigation of microstructure/behavior relations. The influence of constituent properties and size can be predicted.

5. Prediction of matrix damage in 2D woven composites

Matrix damage can be simulated as a process of multiple failures in volume elements in the matrix and in the tows for a 1D composite, or in the longitudinal and the transverse tows in 2D composite. This approach suits a finite element mesh, assuming that the elements contain a sufficient amount of flaws. The process involves crack initiation from flaws and extension element by element. The criterion for


Figure 6.
Table 1. Flaw strength parameters for the matrix.

Matrix	Shape parameter	Scale factor
Interply matrix	4.9	1.6
In transverse tows	4.9	0.6
In longitudinal tows	6.2	7.3

cracking is based on failure probability. The 2D structure with different tow directions, interply matrix and macropores can be reproduced (Fig. 6).

In [15, 16], finite element analysis of failure probabilities has been developed for the computation of matrix damage evolution in 2D woven SiC/SiC and C/C composites. Stresses in the matrix were computed using a commercial computer code. Failure probabilities are determined using a post processor which includes the multiaxial elemental strength model [9, 17, 18] for multiaxial fracture.

The deformations were increased stepwise. At each step, stresses and failures probabilities were computed. Cracks were introduced in critical elements using node splitting technique. A critical element is characterized by the failure probability of 1. The load is increased as long as there is no critical element. When a critical element is obtained, successive computations of failure at constant deformation are done and the crack is increased element by element while failure probability at crack tip is 1.

Computations of crack locations and damage evolution under uniaxial tension compared quite well with in-situ observations on 2D SiC/SiC and C/C composites. An example of crack pattern obtained under bending conditions is shown on Fig. 6. The stress-strain curves derived from average stresses at cell boundaries compared satisfactorily with experimental results. This approach can be applied to 3D meshes.

6. Predictions of rupture time

Lifetime is governed by the failure of fibers. It is driven first by the damage induced by loading, which determines the stress state and allows ingress of aggressive species. The sequence of mechanisms that control lifetime may be summarized as follows:

- First, matrix cracking and associated fiber debonding depending on the magnitude of applied load.
- Second, environmentally driven phenomena that cause either weakening or overloading of fibers that carry the loads.
- Time to failure corresponds to the kinetics of decrease of the load carrying capacity of fibers.

At high temperatures in air, the following elementary phenomena affect fiber load carrying capacity, depending on temperature:

- The oxidation of interphases causes increases of stresses operating on fibers; this phenomenon is observed with carbon interphases from 450 °C.
- Creep of the matrix at temperatures > 1200 °C causes increase of stresses on fibers.
- Oxidation of fibers may cause fiber weakening. This phenomenon is observed on carbon fibers. Kinetics laws for carbon consumption are available. The strength degradation can be related to carbon mass loss rate [45].

- Creep of fibers is also responsible for weakening at temperatures $> 1200^{\circ}\text{C}$. Much work has been devoted to creep of fibers. But the rate in strength degradation has not been modeled.
- Slow crack growth in SiC fibers at temperatures $< 1000^{\circ}\text{C}$. This phenomenon is addressed in the chapter on fibers.
- It is worth noting that oxidation can slow down the phenomenon of weakening when a layer of oxide protects the fibers against degradation by environment. Oxide is produced in the matrix cracks of self healing matrices or at the surface of SiC fibers (at temperatures depending on fiber composition).

Additional structural phenomena take place in composite specimens:

- overloading of fibers when a fiber fails.
- catastrophic failure of isolated fibers which cannot withstand overloading or of groups of touching fibres. As a consequence, weak as well as strong fibers can be eliminated during this step.

Finally, various stochastic structural features need to be taken into account:

- The stresses on fibers depend on both the elementary force f operating on each fiber, and the section of fiber. Fiber diameters have been shown to follow a Gaussian distribution [46], so that fiber sections S_i within a tow are also described using a Gaussian distribution $P_G(S_i)$.
- Fiber strengths follow a Weibull distribution denoted $P_W(\sigma_f)$.
- The elementary forces f depend on microstructural factors which control load sharing such as the amount of matrix bonded to the fiber (the distance to neighbouring fibers), fiber arrangement, density of matrix cracks and the size of interface cracks. So, the elementary forces follow a statistical distribution.
- When a fiber breaks from slow crack growth, the force it was carrying is shared by the surviving fibers. For the same microstructural features as above, random load sharing prevails. So, the resulting force increments Δf on surviving fibers can be assumed to follow a statistical distribution, with $\Sigma \Delta f = f$, f is the force which was carried by the fiber which broke.
- When a group of fibers fails $\Sigma \Delta f = \Sigma f$, so that a larger force increment operates on the surviving fibers. It depends on the size of fiber cluster. The sizes of fiber clusters exhibit a statistical distribution.

In a multiscale approach to failure, modeling of matrix fragmentation and associated stress-state on fibers is the first important step. Then, modeling of stress state changes and fiber strength degradation as a function of time is the second one. The ultimate failure occurs when the stresses reach the resistances of critical fibres.

Attempts to predict lifetime in the frame of a multiscale approach have been made on 1D C/C composites [45] and 2D woven SiC/SiC [47]. The approach to calculating the lifetime of 2D woven SiC/SiC composites at high temperature ($< 1000^{\circ}\text{C}$) under a constant load was based on the rupture time of tows subjected to a uniform stress-state and it integrated several statistical features. At these temperatures, SiC filaments experience slow crack growth (Fig. 22). The model reproduces the above mentioned failure modes.

7. Conclusions

Damage modes in woven composites are hierarchically-organised, sequential and stochastic as a result of microstructure and the respective properties of constituents. Multiple cracking initiates in the matrix from macroscopic and then microscopic flaws. As it proceeds, the load is progressively transferred to the tows.

The woven structure involves various length scales. Associated scale effects inherent to brittle ceramics delineate steps in the load transfer. Damage in the matrix and then in the tows is responsible

for truncation of flaw strength distributions leading to a reduced distribution of critical filaments that governs ultimate failure. As a consequence, the 2D woven SiC/SiC composites exhibit a limited scatter in strength, and a limited sensitivity to scale effects. These remarkable features can be affected by processing defects, such as local strong bonds at interfaces.

The damage modes under tensile load govern the resistance to fatigue at high temperature in aggressive environment like air, when the reinforcing fibers become exposed to overloading and weakening by oxidation or slow crack growth.

Probabilistic models allow the determination of damage kinetics taking into account flaw strength distribution characteristics and constituent properties. They allow also predictions of the stress-strain behavior, and of the influence of elastic and fracture characteristics of fibers and matrix, and resistance of interfaces.

Composites are more complex than most homogeneous materials. However, they offer a remarkable versatility in microstructure. Microstructure-behavior relations allow one to capitalize on this outstanding advantage.

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