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COORDINATOR: Prof. M.H. Lewis, University of Warwick, U.K.

Partners:

1. Department of Materials and Manufacturing Engineering
   University of Plymouth, U.K.

2. Centre for Advanced Materials
   Department of Physics, University of Warwick, U.K.

3. Department of Metallurgy and Materials Engineering
   Katholicke Universiteit, Leuven, Belgium.

4. IFI Joint Ceramic Research Centre
   FORBAIRT, Dublin Ireland.

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'Engineered Fibre Strengthened Ceramic Composites; Structural Integrity and Performance for Energy Conversion and Processing Systems'

M.H. Lewis and S. Sutherland, University of Warwick, Coventry, UK.

O. van der Biest, E. Vanswijgenhoven and M. Wevers, University of Leuven, Haverlee, Belgium, B3001.

G. West and K. Plucknett, University of Plymouth, U.K.

D.M.R. Taplin, University of North London, London N7 8DB, UK.

T. Kelly and M. Murphy, Forbairt, Dublin 9, Ireland.

Abstract. Glass and glass-ceramic matrix composites containing SiC fibres have been studied with a view to their application in energy-conversion systems of high efficiency. The microstructure and their thermal and oxidative/corrosive stability have been investigated, principally for Pyrex glass/SiC and Barium Magnesium Aluminium Silicate (BMAS)/SiC composites. Mechanical properties of these composites have been evaluated over a range of *temperature* under conditions of monotonic loading, creep and fatigue. The data has been interpreted and subsequently simulated using analytical models together with micromechanical measurements of fibre/matrix interface parameters.

1. Introduction

The development and use of monolithic engineering ceramics had been only partially successful and limited to either low-stress applications or components in which fracture did not precipitate failure of the engineering system. The requirement for enhanced performance and efficiency in energy-conversion systems, such as the gas-turbine, has motivated the development of ceramic matrix composites (CMCs) for high-risk components.

Up to 1990 CMC development was essentially at the research laboratory level; early work in the UK on carbon fibre/glass matrix composites was followed by development of polymer-precursor SiC fibres (in Japan) which stimulated the fabrication of glass-ceramic matrix composites (at UTR and Corning, USA) and SiC/SiC (CVI-matrix) in France. R & D at that stage was focused on modelling of the basic stress-strain response in relation to fibre and interface properties. Very limited studies had been conducted on high temperature microstructural stability and timedependent or cyclic deformation. These were the major themes within the research described here in which two types of silicate-matrix/SiC fibre composite were studied;

SiC (Nicalon)/Pyrex (borosilicate) glass matrix
SiC (Nicalon)/Glass-Ceramic (aluminosilicate) matrices with differing composition

The main components of the research program me may be identified with the following objectives: -

(i) To define microstructure (fibre, matrix and interface) within glass and glass-ceramic matrix composites in relation to process route/matrix chemistry and microstructural changes due to high-temperature exposure.

(ii) To define basic macro-mechanical and micromechanical (interface) properties using time-independent tests (constant imposed strain rate) and micro-indentation ('push-down' and 'push-through') tests on fibres.

(iii) To measure time-dependent deformation (creep) over a range of temperature or stress, to identify microscopic mechanisms for creep with reference to individual CMC component properties and to model the creep response under constant stress and cyclic loading.

(iv) To measure the response to rapid cyclic stress (fatigue), to assess fatigue lifetimes and to understand and model mechanisms for damage accumulation and failure.

(v) To assess the combined influence of high temperature oxidising and corrosive atmospheres on microstructural and mechanical degradation.

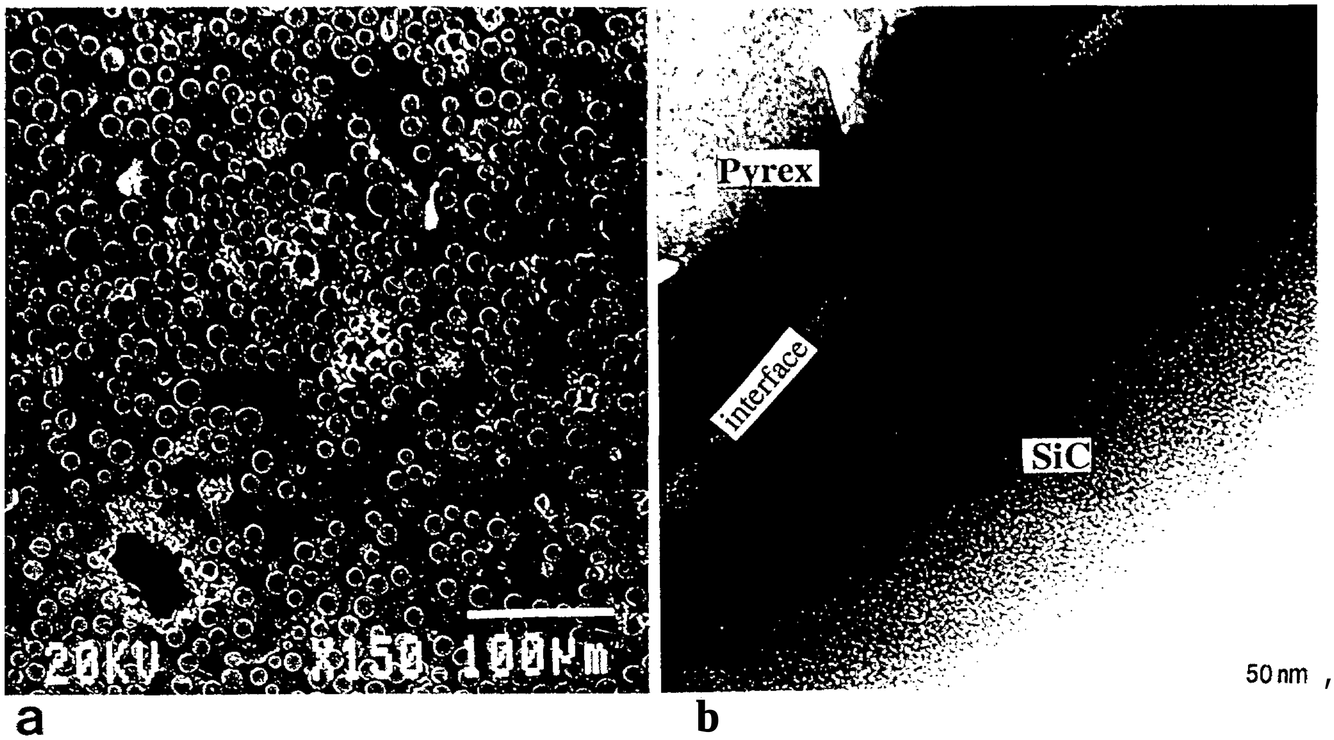
2. Technical Description

2.1. Materials and microstructural characterisation. CMCs used in this project were obtained from industry sponsors and other commercial sources in the form of hot-pressed tiles - 3mm thick. Most of the research was conducted on unidirectional and cross-plied (0/90°) fibre architectures. The various sources of supply, with brief explanation of constitution and microstructure (analysed via XRD, SEM and TEM), were:-

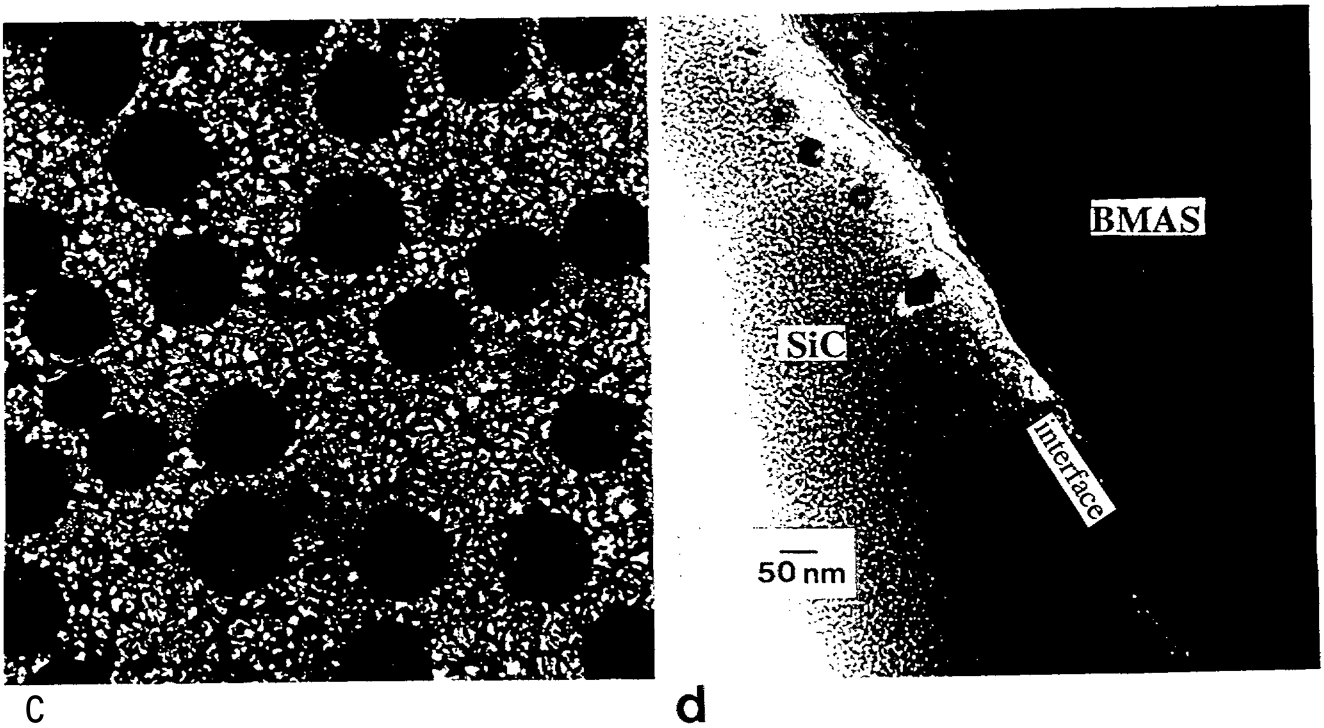
(a) Pyrex glass matrix/Nicalon fibre - mainly from Pilkingtons (good matrix density but some unwanted crystallisation of SiO_2 polymorphs), used especially for medium temperature deformation in modelling the regime of elastic fibre/creeping matrix. The microstructure provides an isotropic single phase matrix of similar thermal expansion to Nicalon fibres, with limited residual thermal stress. Film-matrix interfaces contain carbon enrichment due to limited 'in-situ' reaction of type $SiC + O_2 \rightarrow SiO_2 + C$ at the relatively low hot-pressing temperature (Fig. 1 a,b).

(b) BMAS (barium magnesium aluminosilicate) matrix/Tyranno fibre - from AE Technology, (Harwell) was the major source of GCMC. The BMAS matrix contains a crystalline phase mixture of Ba osumilite ($BaO.2MgO.3Al_2O_3.9SiO_2$), cordierite ($2MgO.2Al_2O_3.5SiO_2$), celsian ($BaO.Al_2O_3.2SiO_2$) and small quantities of mullite ($3Al_2O_3.2SiO_2$). A substantial ($\sim$50nm) carbon rich interface layer, formed by in-situ oxidation, also contains TiC particles due to reaction with Ti which is a minor component of the Tyranno fibres (Fig. 1 c,d).

(c) CAS (calcium aluminosilicate) matrix/Nicalon fibre - from Corning Glass, via Rolls Royce (high quality, negligible matrix porosity - used as a reference material, with



SiC/Pyrex



SiC/BMAS

Fig. 1.

limited commercial availability). The matrix is mainly crystalline anorthite ($CaO.Al_2O_3.2SiO_2$) with -50 interface zone of carbon enrichment, similar to BMAS composites.

(d) MAS (magnesium aluminosilicate) matrix/Nicalon fibre - from Pilkingtons. Unforseen problems with matrix crystallisation treatments required the substitution of MAS tiles hot pressed at Rolls Royce to a Warwick prescription used in a small part of the project.

2.2. **Mechanical Testing Methodology.** Bend-testing of rectangular bars, with axes parallel to the fibres or to the 0° ply direction, was adopted as a survey technique, using low specimen volumes, to determine property-trends with varying thermal or corrosive treatment and to sample the quality of hot-pressed tiles with meaningful statistics. -However, tensile tensile was a priority in studies of creep, fatigue and creep/fatigue over a range of temperature which required the development of special specimen shapes and the use of short-furnaces on commercial test machines.

A novel high-speed CNC diamond machining facility has been developed to enable high-precision rapid prototyping of various test specimen geometries, with minimal material waste. Typically, for the creep programme, tensile specimens with 40mm x 4mm x 3mm gauge section were machined with wedge-shaped ends of matching profile to the superalloy wedge-shaped grips.

Short furnaces (Pyrotherm-UK or **MIS-USA)** with > 1300°C constant-temperature capability over the gauge length were interfaced with an Instron 1185 load-frame (for creep) or MTS 810 servo-hydraulic machine (for fatigue). Strain measurement was achieved with MTS low-contact force extensometers and the provision of low electrical 'noise' in the environment together with thermal stability (using thermostatic water-cooling) was critical for long term creep and fatigue testing.

The measurement of interracial (fibre/matrix) properties was accomplished on 'as-received' and thermally-treated specimens using indentation methods. Fibre 'pushdown' tests were performed on a mum-indentation system or in a specially-developed SEM based microindentor. Interface debond energies (Γ) and shear stresses τ, derived from these tests, were used to assess interface modification due to oxidative heat. treatments and as input to the modelling of stress-strain response. Typical data is exemplified in Table 1.

Table 1
Interracial Properties

Material	$2\Gamma(Jm^{-2})$	τ(MPa)
BMAS - as received Tyranno - 1200 °C/100hr	1.2 ± 1.6 1.8 ± 0.8	25 ± 7 28 ± 5
Pyrex - as received Nicalon - 500 °C/100hr	8 ± 10 15 ± 6	40 ± 6 **90 ± 50**
MAS - as received Nicalon - 1200 °C/100hr	1.4 ± 1.6 **0**	29 ± 9 **5.6 ± 3.5**
CAS - as received Nicalon - 1200 °C/100hr	9.6 ± 1.2 11.8 ± 1.8	25 ± 3 25 ± 3

3. Results and Discussion

3.1. Monotonic stress-strain behaviour. The principal mechanical design parameters (matrix cracking stresses σ_m^1 and σ_m^2 (0° and 90° plies, respectively) ultimate failure stress σ_u elastic moduli (E and Poisson coefficient) have been measured for the Pyrex and BMAS CMCS and the development of microstructural 'damage' has been assessed via microscopy, acoustic emission and composite stiffness.

An example of tensile stress-strain response (Fig.2) for the cross-plied (0°- 90°) BMAS composite shows a typical CMC behaviour with σ_m^1 and σ_m^2 characterised by a successive modulus reduction followed by progressive load-transfer to 00 fibres with their subsequent failure and 'pull-out'. The marked changes at higher temperature are due to the onset of matrix plasticity (lZOO°C) and the premature failures (at 700°C and 1200°C) due to the ingress of oxidising atmospheres to the fibre/matrix interface via matrix microcracks. These trends, shown in more detail from the bend data covering the complete test-temperature range (Fig.3), emphasise the importance of this carbon-rich interface oxidation above ~500°C. This is also apparent in (non-microcracked) preannealed specimens, in which oxidation occurs via 'channeled' reaction down fibre/matrix interfaces from exposed fibre ends arid becomes a specimen size/time dependent phenomenon. An inhibition of the channeled oxidation occurs at high temperatures (1 OOO-12OO"C) due to passive oxidation of fibre ends by formation of silica 'plugs'. This phenomenon has been extensively studied as a means of optimizing a 'pretreatment' for high temperature testing at stresses below the matrix cracking thresholds. An example of this data for as-received (AR) and preannealed or unquenched (UQ) cross-plied BMAS materials is shown in Fig.4. As a *consequence all* high temperature testing was preceded by a 1-2 hour oxidising pretreatment at 1000-11OO"C.

3.2. Stress-strain modelling. A comprehensive micromechanics-based model has been developed to simulate stress-strain response during monotonic, cyclic and fatigue tension testing. The model uses matrix properties and damage development in matrix, fibre and interface as input parameters and simulates the relationship between longitudinal stress, longitudinal strain and transverse strain. It uses different 'unit cells' of CMC, combined in series, parallel or in serial combination of parallel cells. The cells consist of a single fibre within a matrix in different damage states (multiple matrix cracking, interface debonding, fibre fracture and pull-out). Bearing in mind the uncertainties and residual stresses in fibre and matrix, due to thermal mismatch, the correspondence between experiment and model simulation is remarkably good. An example for a U.D. BMAS/SiC composite is shown in Fig.5.

3.3 Notch-sensitivity. An insensitivity to stress/strain - concentrating sites is a key factor in application of shaped CMC components, mechanical interconnects (bolt or rivet holes) and surfaces subjected to accidental mechanical damage. A prerequisite for notch insensitivity is fibre matrix debond in the matrix-cracking 'process-zone' and a sufficiently high ratio of fibre strength to interface shear stress (τ), which for silicate matrix CMCs is sensitive to processing temperature and matrix chemistry. For BMAS/Tyranno (0/90°) CMCS notch-sensitivity has been assessed by measuring the mean failure stress for tensile specimens containing plane (diamond sawn) notches of differing depth (Fig. 6). The insensitivity of failure stress (as opposed to load) for different notch depths (expressed as a ratio to initial width of gauge section) is clearly demonstrated and in dramatic contrast to the behaviour of 'brittle' monolithic ceramics in which fracture stress varies as $1/\sqrt{a}$ according to a Griffith-

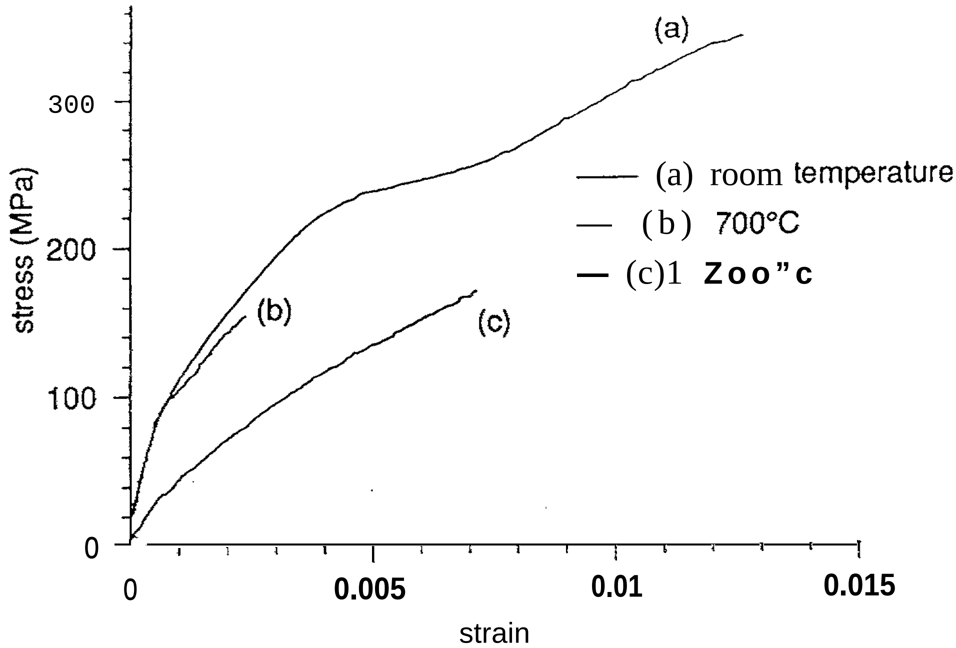


Fig.2. Stress vs strain plotd for BMAS/Tyranno at a) room temperature, b) 700°C and c) 1200°C

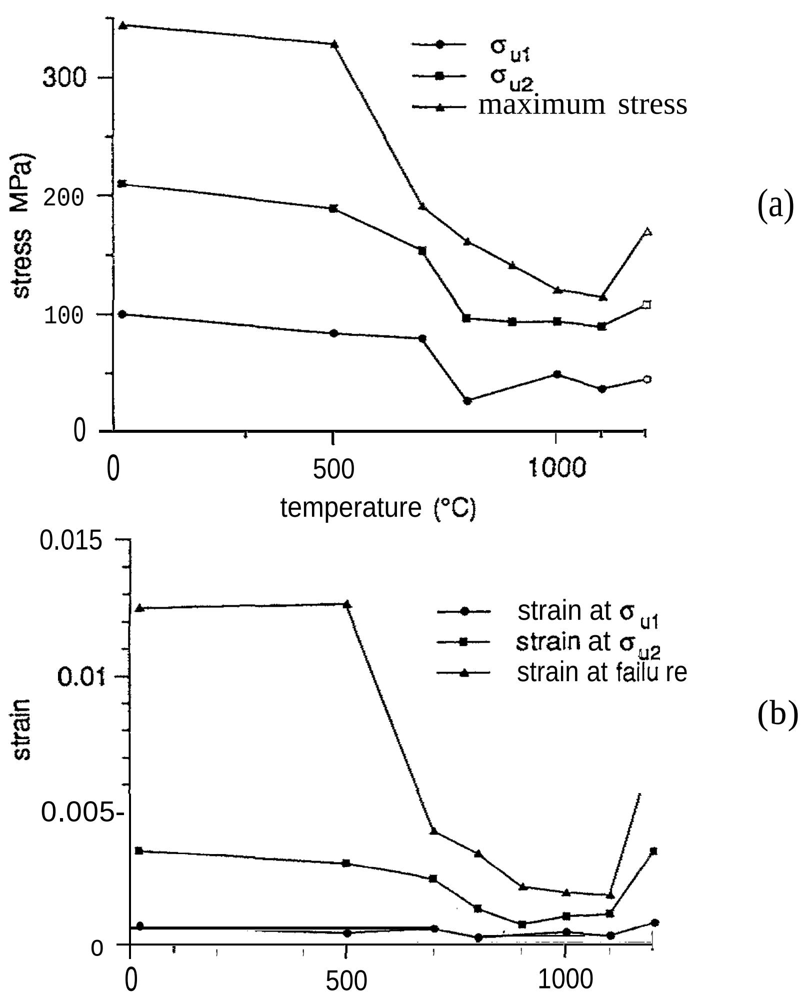
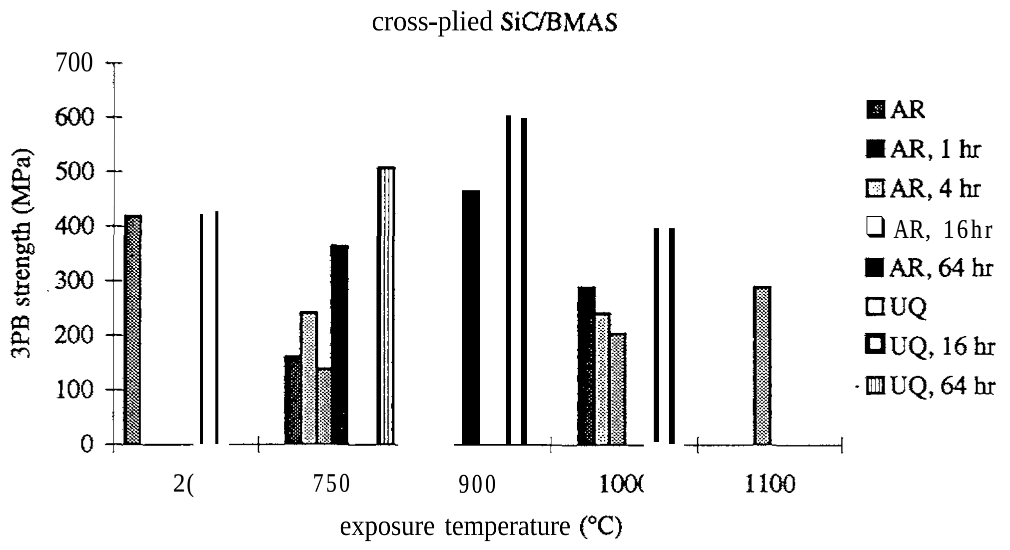


Fig.3. Variation of a) microcracking stress, MOR b) strain with temperature.



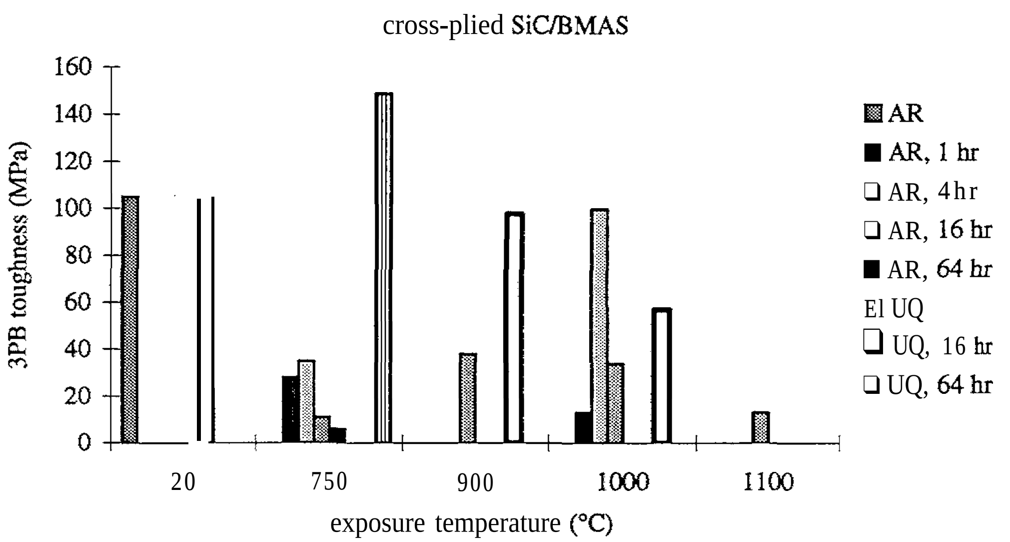


Fig.4. The three point bending strength and toughness of as received and pre-treated cross-plied SiC/BMAS after exposure to hot air (at a given temperature, the value bars for the as received material are given on the left, for the pre-treated material at the right)

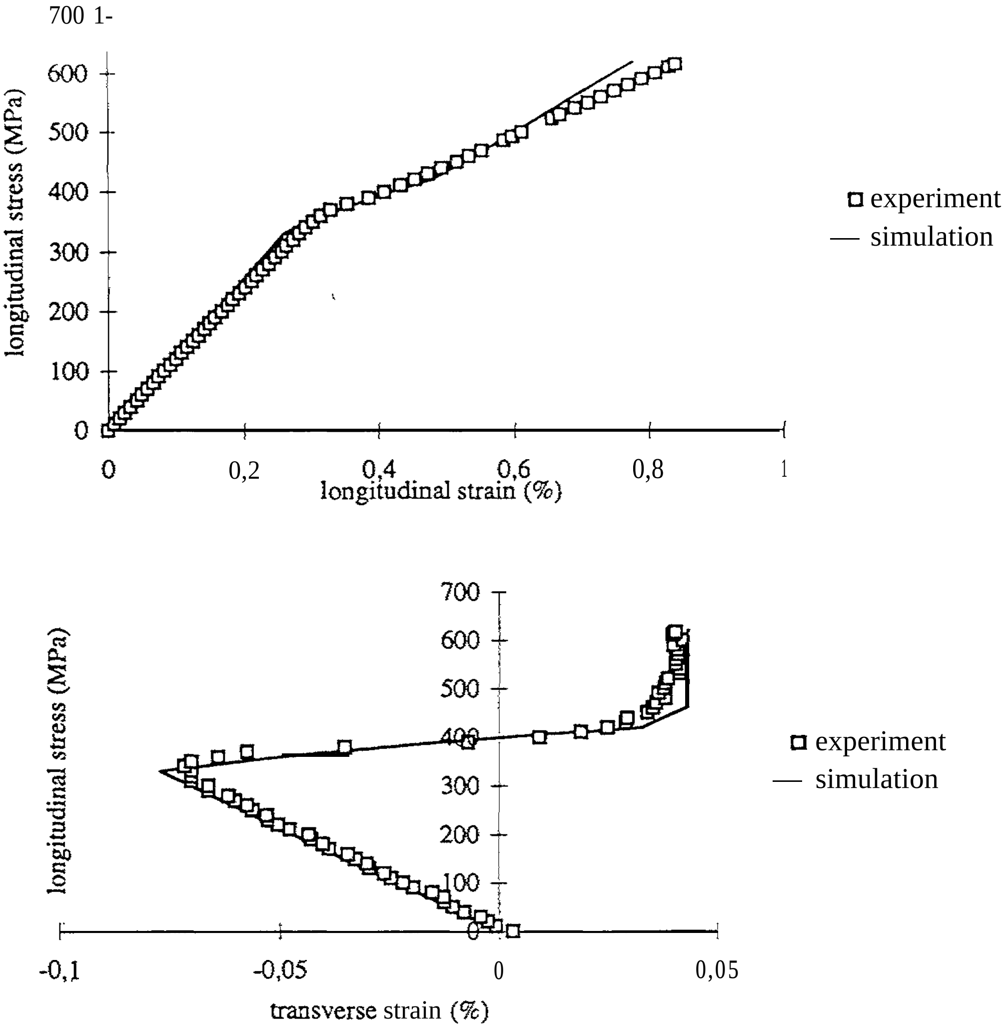


Fig.5. Comparison between the experimentally observed and theoretically predicted tensile response of uni-directional SiC/BMAS (all input parameters except the in situ fibre strength and the residual stress state in fibre and matrix were determined experimentally)

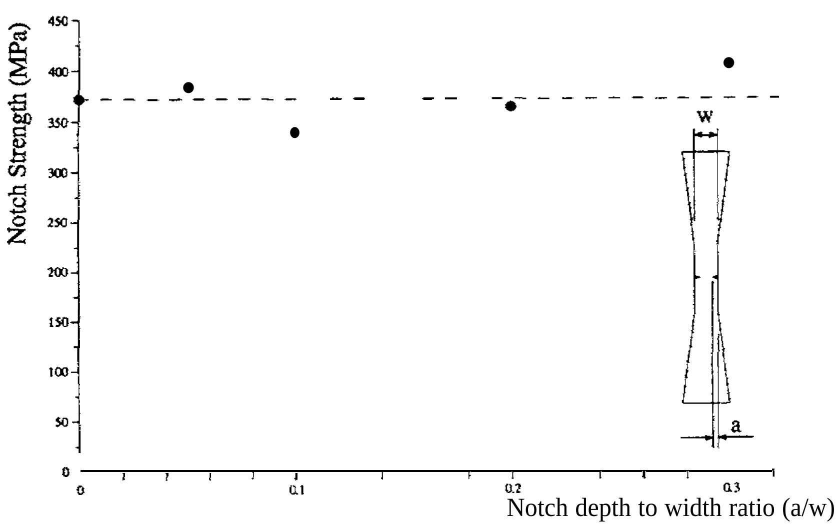


Fig.6. Effects of notch depth to width ratio on tensile strength of 0,90 BMAS/ Tyranno in air at room temperature.

type relation.

3 . 4 High-temperature creep. Creep has been measured both in bend and tension, at stress levels below σ_m^2 to inhibit early oxidation-induced fibre fracture'. Below - 800°C fibres exhibit negligible creep but extend elastically during the progressive load-transfer from a creeping matrix exemplified by Pyrex (borosilicate) glass. Hence a Pyrex/Nicalon CMC is an ideal experimental system for comparison with a theoretical model consisting of a viscous element (matrix) in parallel with an elastic element (fibres). The correlation

between tensile creep curves, for 3 temperatures, with a model strain (ϵ) - time (t) relation

$$\left[\epsilon_c = \frac{\sigma_f}{E_f} = \frac{\sigma_c - \sigma_{mo} V_m e^{-t/\theta}}{E_f V_f}\right]$$

is shown in Fig.7a. In this relation σ_f, σ_c, and σ_{mo} are stresses

in fibre (variable), constant applied stress σ_c and initial matrix stress σ_{mo}, θ is the 'time-constant' for load-transfer, which is a function of matrix viscosity (and hence temperature). After a time -56 the fibres carry over 99% of the applied load and the transient creep is complete.

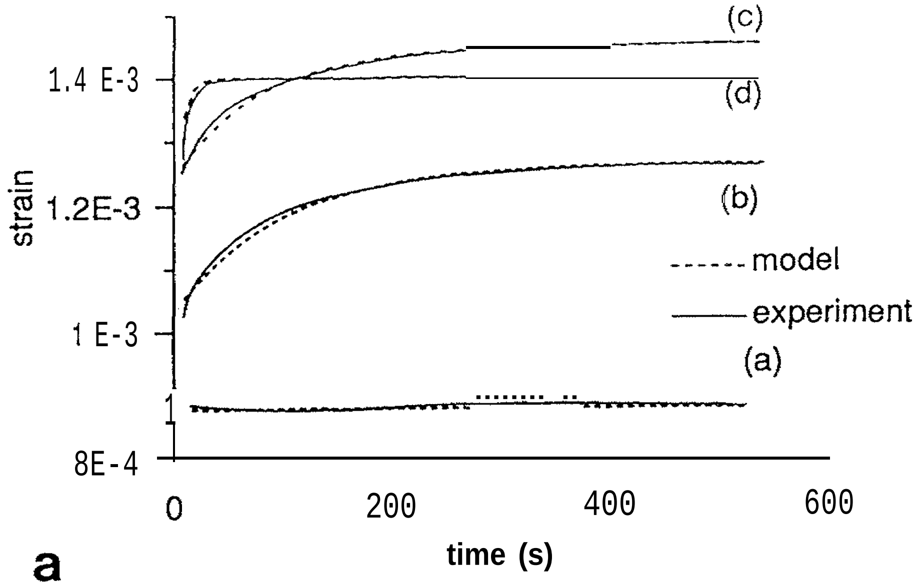
Above ~1000°C both silicate matrix and Nicalon (or Tyranno) fibres undergo measurable creep with a load-transfer transient followed by a pseudo-steady-state strain rate controlled by 0^0 fibres. This has been demonstrated for BMAS, CAS and MAS matrix CMCs which may be approximately modelled by a modified creep

relation; $\epsilon_c = \frac{\sigma_f}{E_f} + K\sigma_f^n t^p$, where n and p are stress and time exponents for fibre creep

(-1 for Nicalon and Tyranno, representing Newtonian creep of a pseudo-amorphous solid). Fig.7b (for BMAS) is an example, which also shows that steady state creep is not achieved at 1200°C (i.e. $p \neq 1$) due to time dependent changes in fibre structure. The final creep rates are matrix-insensitive, demonstrated by the range of glass-ceramic composition used in this programme and compared with isolated fibre creep rates in Fig.8. The relatively good CMC creep rates and low stress sensitivity compared to a typical superalloy (NASAIR 100) are also demonstrated.

Cyclic creep (or 'low cycle fatigue') experiments on BMAS/Tyranno CMCS substantiate the creep model, with considerable creep recovery on the unloading cycle due to matrix compression by elastically strained fibres. A simple model for load-transfer has been used to explain the change in creep recovery ratio with number of cycles. This phenomenon is exemplified for a flexural test on U.D. specimens (Fig.9a) together with a model plot of stress redistribution between the CMC components (Fig.9b).

Creep lifetimes (stress-rupture) have been assessed for relatively few tests in bend and tension, with a conclusion that oxidation-induced fibre degradation is the major limit. In fully dense CMCS this only occurs above the matrix microcracking threshold. Below the microcracking stress, oxidative degradation is limited to oxygen transport through the silicate



Experimental and model curves for Pyrex/Nicalon at
(a) 400°C, (b) 460°C, (c) 500°C and (d) 550°C

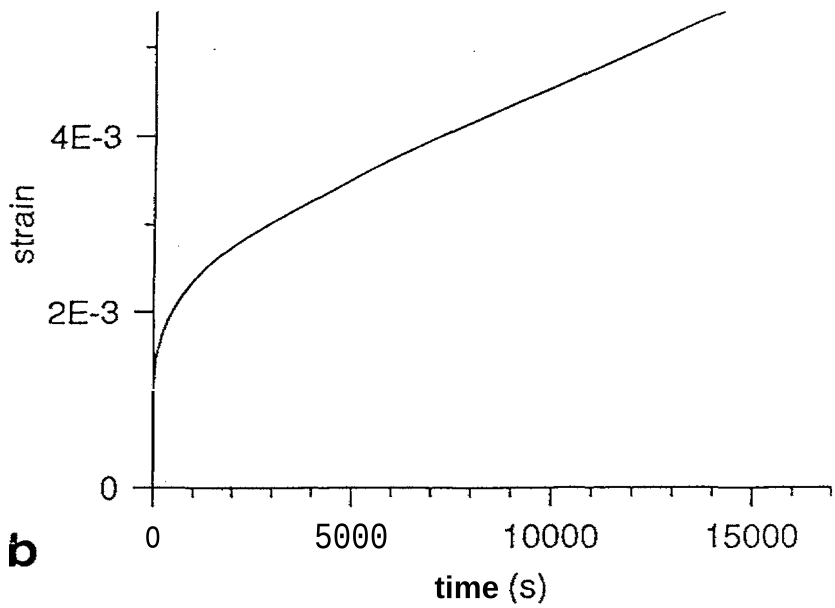


Fig.7. Typical strain vs time plot for BMAS/Tyranno at 1200°C and 60MPa

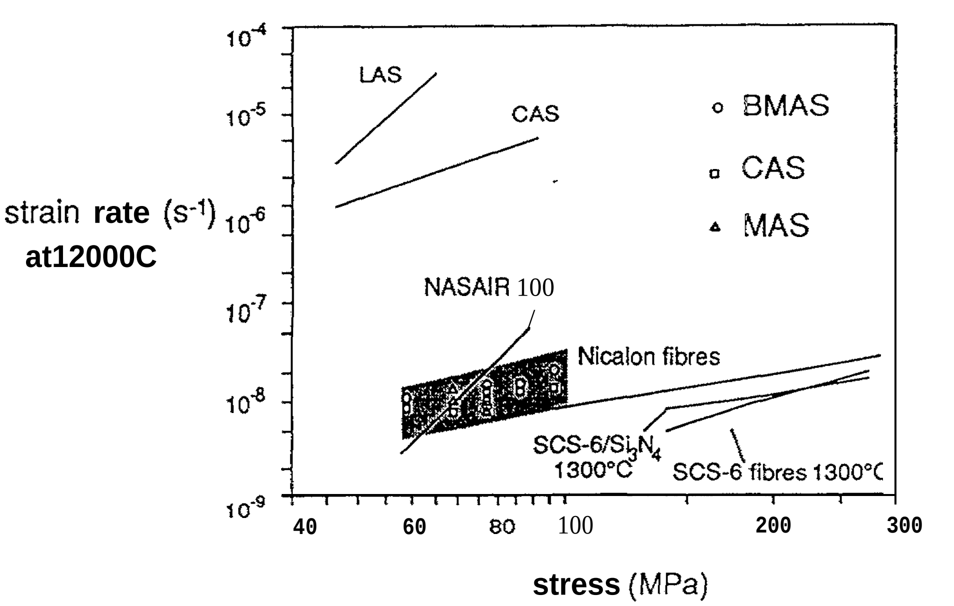
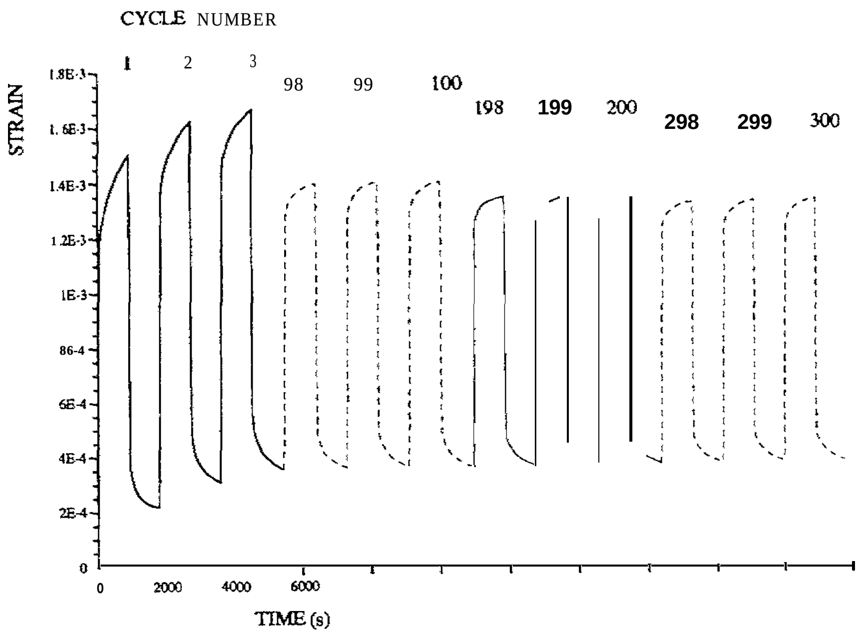


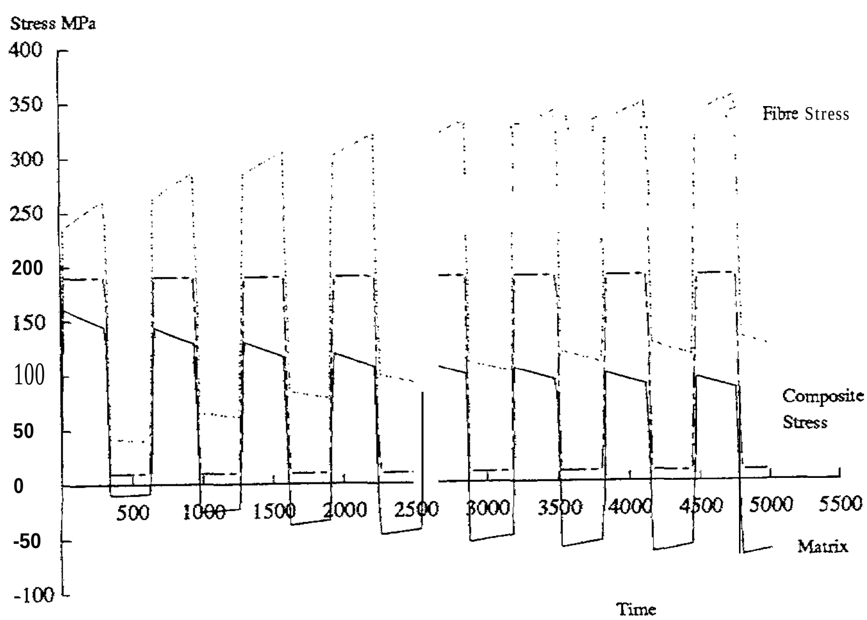
Fig.8. Comparison of creep strain rates for various CMCs and values of selected matrices and fibres in isolation.

a



Schematic of selected cycles showing strain during cycling at 1100°C

b



Distribution of stress during cyclic creep between composite components. Primary creep disregarded.

Fig.9.

matrix (which has been shown to be very slow) or 'channelled' oxidation of carbon-rich interfaces from exposed fibre ends. The latter mechanism may be inhibited by the passive oxidation pretreatment discussed in section 3.1. In laboratory tests it is clear that initial loading rate may influence the susceptibility to premature matrix microcracking. Low loading rates may allow matrix creep to occur with consequent load-transfer to the fibres before reaching the peak value. This is an additional factor to be considered in applications involving cyclic stressing, where frequency may be critical.

3.5 Fatigue and damage-development. Tension-tension fatigue tests were performed on flat 'dogbone' specimens or rectangular bars on SiC/BMAS and SiC/Pyrex CMCs using an MTS 810 servohydraulic machine at a frequency of 3Hz and stress ratio of 0.1, using a sinusoidal wave.

At ambient temperature the various CMC architectures (unidirectional-UD, cross-plied CP, angle-plied-AP and quasi-isotropic QI) could all sustain fatigue loading in a damaged condition but with reduced stiffness. The damage initiation stress and fatigue limit improve with the fraction of fibres aligned with the stress axis (Table 2)

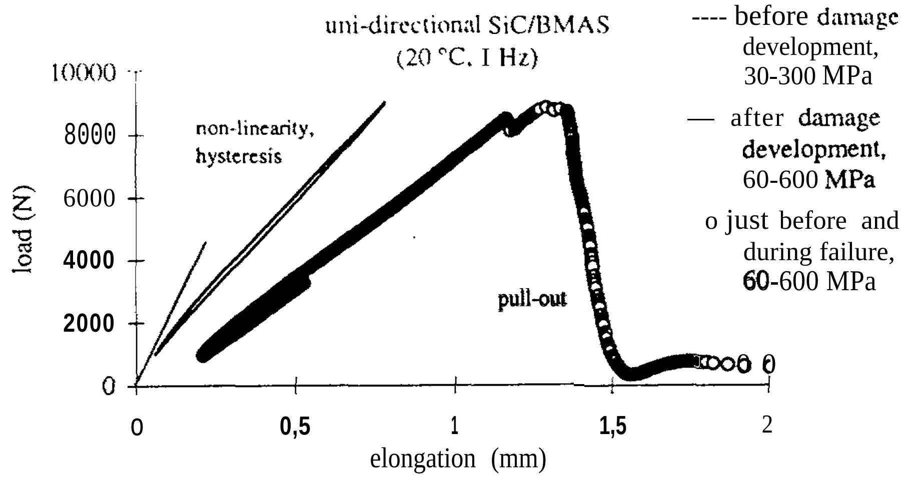
lay-up	UD	CP	AP	QI
damage initiation stress (MPa)	250	50	30	40
fatigue limit (MPa)	400	200	60	120

Table 2. Fatigue data for SiC/BMAS at 20°C

Fatigue 'damage' was normally in the same form as for non cyclic stressing, i.e. matrix cracking, fibre debond, fibre fracture and pull-out. Most of the damage occurred during the first few cycles, precipitating reduced stiffness and hysteresis in the stress-strain relation (Fig. 10a). The sensitivity of acoustic-emission in monitoring the different stages of microcracking is illustrated in Fig. 10b.

High temperature fatigue of SiC/BMAS laminates showed that oxidation of carbon-rich interfaces and fibre surfaces via microcracking exposure, above 500°C, resulted in a loss of fatigue damage tolerance. A 'brittle', essentially monolithic-like failure was due to silica 'bridging' of the oxidised interface and localised fibre strength loss in the microcrack contact zone, as observed in monotonic tensile tests. A more detailed study of interface oxidation mechanisms in relation to the onset of brittle failure has been conducted, which emphasises the critical role of microcrack oxygen transport during high-temperature testing and its general application to CMCS with carbon rich interface layers on Nicalon or Tyranno-type SiC fibres. Similar trends were observed for all fibre architectures; an example for UD specimens illustrating the fatigue failure probability with varying temperature and peak stress is shown in Fig. 11. At the highest temperatures (1000 - 1100°C for BMAS) matrix creep and stress relaxation was detectable even at these relatively high frequencies (3Hz).

3.6 The influence of environment on properties. Oxidative or corrosive environments are relevant to applications involving diesel or turbine combustion gases, heat exchangers and chemical plant. A survey has been conducted of the influence cm microstructure, strength and hardness, of exposure of the glass and glass-ceramic matrix CMCs to oxygen rich air, moisture-laden air, SO_2 and NO_2 - containing air up to 900°C.



Typical mechanical response during fatigue of tough SiC/BMAS
(no damage developing, damage developing but no failure occuring, damage developing and failure occuring)

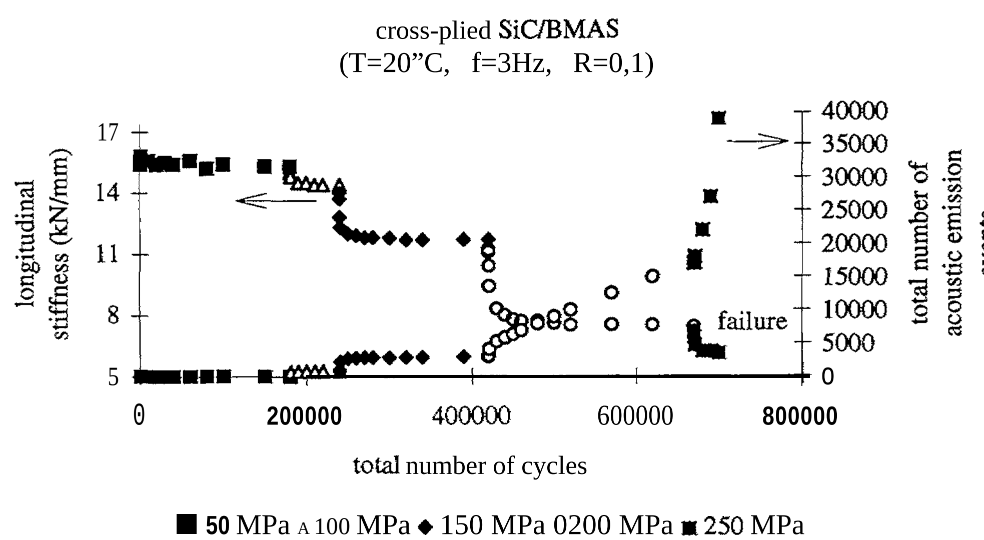


Fig. 10. Evolution of the acoustic emission activity around the peak stress and the Young's modulus during room temperature fatigue of cross-plied SiC/BMAS (damage in 90°-plies : saturation of AE, damage in 90°- and 0°-plies : continuous increase in AE, before failure : sharp increase in AE)

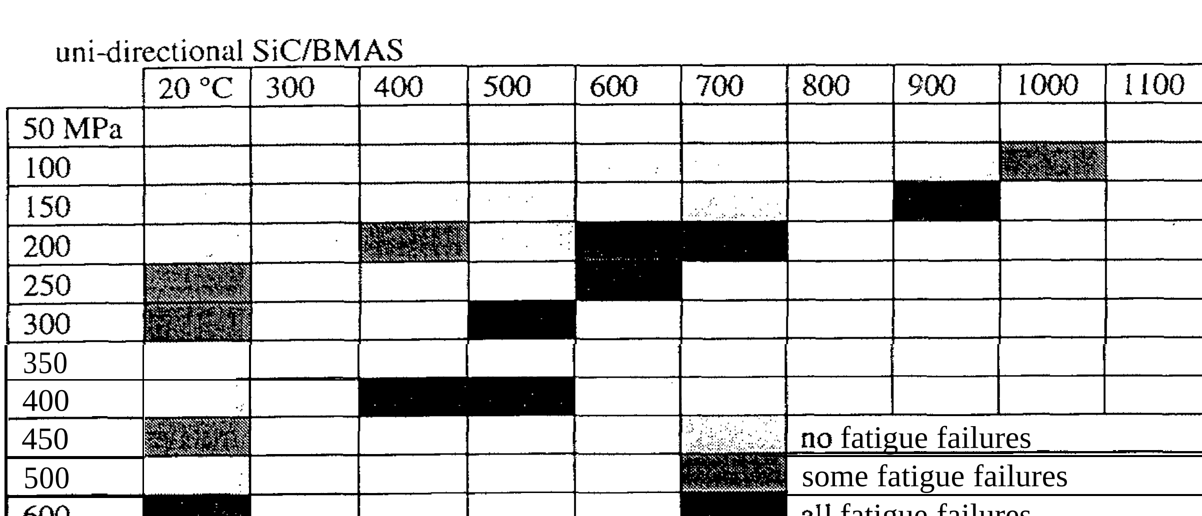


Fig. 11. Relation between fatigue survival and fatigue failure as a function of fatigue peak stress and fatigue temperature SiC/BMAS material,

For glass matrix composites (Pyrex/Nicalon) long exposure to all atmospheres (typically 21 days@ 500°C) resulted in strength-loss by at least a factor of 2 and a change from shear-failure to brittle, tensile fracture. Interface oxidation, independent of corrosive species, is the probable mechanism with crystallisation (of cristobalite, in the matrix) an additional factor causing microcracking due to internal stress.

For glass-ceramic matrix composites (CAS and BMAS) the large strength reductions and fracture mode changes do exhibit a dependence on corrosion chemistry and concentration but statistically valid differences are clouded by porosity levels within most hot-pressed tiles. The principal mechanism is again that of interface and fibre surface degradation, both pores and microcracks providing the transport paths for the oxidising atmosphere. An encouraging feature of the highest quality, low porosity, BMAS matrix composites is the relative insensitivity to different oxidising and corrosive heat treatments and a statistically - significant increase in bend fracture stress with the thermal treatment. An example is given in Table 3 which compares the 'as-hot-pressed' (unpreconditioned) strengths with those given an oxidising anneal (preconditioning) at 1200°C and subsequently exposed to SO_2-containing atmospheres at 900°C. The failure mode ('phased-delamination) is typified b y progressive shear fracture between plies accompanied by matrix cracking and fibre pull-out.

Table 3 Summary of Fracture Strengths and Modes of Failure for BMAS-SiC (Batch 3) GCCS

Unpreconditioned		Preconditioned		0.5% SO_2	
28-1	645 MPa	29-1	800 MPa	26-1	850 MPa
Phased Delamination		Phased Delamination		Phased Delamination	
28-2	436 MPa	29-1	736 MPa	26-2	892 MPa
Phased Delamination		Phased Delamination		Phased Delamination	
28-3	664 MPa	29-3	68 I MPa	26-3	782 MPa
Phased Delamination		Phased Delamination		Phased Delamination	
28-4	715 MPa	29-4	793 MPa	26-4	873 Mpa
Phased Delamination		Phased Delamination		Phased Delamination	
28-5	634 MPa	29-5	760 MPa	26-5	829 MPa
"Catastrophe" Delamination		Phased Delamination		Phased Delamination	
28-6	638 MPa	29-6	864 MPa	26-6	832 MPa
"Catastrophic" Delamination		Phased Delamination		Phased Delamination	
28-7	623 MPa	29-7	634 MPa	26-7	829 MPa
"Catastrophic" Delamination		Phased Delamination		Phased" Delamination	
28-8	623 MPa	29-8	752 MPa	26-8	848 MPa
Phased Delamination		Phased Delamination		Phased Delamination	
28-9	660 MPa	2 9 - 9	827 MPa	26-9	746 MPa
Phased Delamination		Phased Delamination		Phased Delamination	

4. **Conclusions**

During the initial period of this project few international laboratories had experience of processing and fabrication of long-fibre CMCs. Glass and glass-ceramic matrix composites were initially developed at AEA Harwell (later AEA Technology) and the process later transferred to Pilkington Research. Warwick University had initiated a parallel project, with Rolls-Royce collaboration, in developing novel borosilicate glass and MAS glass-ceramic matrix constitutions based on their expertise in the glass-ceramic field. Based on this combined experience, we now have a precise formulation for modified pyrex glass and non-stoichiometric MAS compositions together with optimised CMC processing schedule.

The major part of this project was subsequently conducted on commercially-available

BMAS glass-ceramic matrix tiles hot-pressed at AE Technology, Harwell. In the assessment of basic properties it became clear that the Harwell process had not been optimised and the data generated in the project enabled a marked improvement in process route.

The combined knowledge of constitution, processing and CMC fabrication, for glass and glass-ceramic systems, developed as part of this programme represents a European 'state of the art' which is now internationally competitive.

Within the difficult area of CMC tensile testing, involving high stresses, high anisotropy and high temperatures, with a need for sensitive strain monitoring, an additional feature has been the development of a high-speed CNC diamond machining facility to enable rapid, high-precision, prototyping of test specimen geometries.

The testing programme has also generated useful feedback to 'test-machine and component manufacturers, with instrumentation operating at the limits of mechanical and thermal stability, for example fluctuations in Instron load cell output due to thermal drift which is critical under 'constant load' (creep) control.

Simple models have been developed for monotonic and cyclic stress/strain response and for creep. The main purpose in developing analytical models for the stress/strain response of engineering solids is to predict this response in relation to the properties of microstructural components (e.g. moduli or creep rates of fibres and matrices together with interracial shear stresses and debond energies). Alternatively these individual phase or interface properties may be analysed from the observed composite deformation in combination with the models. In this project the simple modelling approach has been substantiated within a wide range of experimental test data.

At the initiation phase of the project glass and glass-ceramic matrix composites were considered to be ideal models for generic CMC behaviour but also to have potential as real high temperature materials. For example Nicalon/borosilicate composites were possible contenders for lightweight/stiff turbine compressor blades, operating to ~450°C, and refractory GCMCs as engine components and heat exchangers operating to at least 1200°C. Few of these applications are now relevant; in the lower temperature regime there is competition from Ti-based alloys and intermetallics (e.g. Ti_3Al or TiAl) and at higher temperatures the major problem is interface oxidation and, ultimately, fibre stability. Within this project a major contribution has been made to understanding these degradation mechanisms and hence redefining design limits within differing stress states, time and temperature dependencies. Hence a major motivation in subsequent project planning on CMC materials development has been that of oxidation-stable interfaces which retain the necessary debond/shear property.

The application of GMCS and GCMCs in their current microstructural states remains a possibility for low density rigid structures which demand corrosion resistance to moderate temperatures not accessible to PMCs. Economic issues relating to fibre and processing costs are likely to be critical.

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