

**CPD3 T3: Thermal
Cycling and Mechanical
Properties of a
Unidirectional Titanium
Matrix Composite and a
Cross-Ply Ceramic Matrix
Composite**

A J Gant and R Morrell

July 1999

© Crown copyright 1999
Reproduced by permission of the Controller of HMSO

ISSN 1361-4061

National Physical Laboratory
Teddington, Middlesex, UK, TW11 0LW

Extracts from this report may be reproduced provided the source is
acknowledged and the extract is not taken out of context.

Approved on behalf of Managing Director, NPL, by Dr. C. Lea,
Head, Centre for Materials Measurement and Technology.

July 1999

CPD3 T3: Thermal Cycling and Mechanical Properties of a Unidirectional Titanium Matrix Composite and a Cross-Ply Ceramic Matrix Composite

A J Gant & R Morrell

Centre for Materials Measurement & Technology
National Physical Laboratory
Teddington, Middlesex, UK, TW11 0LW

ABSTRACT

Recently there has been some interest in the use of ceramic matrix composites and titanium matrix composites in high temperature aerospace applications; specifically jet engine components such as bladed rings in engine compressors. Such materials have been characterised extensively as far as interfaces and a range of mechanical properties are concerned either at ambient or elevated temperatures, but relatively little work has been done on evaluating structure/property relationships in such materials subjected to thermal cycling to elevated temperatures, representative of in-service conditions. The materials used in the present study and subjected to thermal cycling were a unidirectional titanium/silicon carbide MMC and a cross ply barium osumilite glass ceramic CMC. The effects of thermal cycling on the interfaces in these materials were studied at room temperature, with and without subsequent mechanical testing. Scanning electron microscopy was primarily used to characterise the interfaces, together with monotonic and fatigue data in uniaxial tension; the latter being used, where possible, to relate interface response to bulk mechanical properties. In both the MMC and the CMC, property changes were observed after cycling to the lowest temperatures in the test regime; 450°C and 500°C respectively. Degradation of the CMC was progressive across the temperature regime studied, whereas in the TMC no consistent trends in bulk mechanical properties were seen due to the effect of microscopic interfacial changes being effectively masked by changes in matrix condition, caused by thermal cycling.

CONTENTS

1	INTRODUCTION	1
2	EXPERIMENTAL	1
	2.1 MATERIALS	1
	2.2 TEST PROCEDURES	2
3	RESULTS	6
	3.1 METALLOGRAPHIC AND CERAMOGRAPHIC EXAMINATION OF INTERFACES	6
	3.2 UNIAXIAL TENSILE TESTING	11
	3.2.1 90° MMCs	11
	3.2.2 0° MMCs	14
	3.2.3 CMCs ((0,90) Laminates)	16
	3.3 FRACTOGRAPHY OF POST-TENSILE SPECIMENS	18
	3.3.1 90° MMCs	18
	3.3.2 0° MMCs	19
	3.3.3 CMCs ((0,90) Laminates)	21
	3.4 UNIAXIAL (TENSILE) FATIGUE TESTING	23
	3.4.1 90° MMCs	24
	3.4.2 0° MMCs	26
	3.4.3 CMCs ((0,90) Laminates)	27
4	DISCUSSION	29
	4.1 MMCs	29
	4.2 CMCs ((0,90) LAMINATES)	30
5	SUMMARY	31
6	SUGGESTIONS FOR FURTHER WORK	32
7	REFERENCES	32
8	ACKNOWLEDGMENTS	34

1 INTRODUCTION

This paper discusses the mechanical properties and interfacial characteristics of a titanium matrix composite, namely Ti-6%Al-4%V with unidirectional SiC fibres, and a ceramic matrix composite, namely barium osumilite glass ceramic with cross ply SiC fibres, in the as-received condition, and after repeated thermal cycling. Both ambient temperature monotonic and mechanical fatigue behaviour in the as-received condition and in the post heat treated condition are reported for each type of composite, with the behaviour of the interfaces during thermal fatigue investigated.

The primary driver for the development of titanium matrix composites (TMCs) has been the gas turbine industry. As designers look for ways to increase the performance of aircraft jet engines, ever more stringent requirements are placed on the materials to be used. These include high operating temperatures, long expected lifetimes, extreme consequences of structural failures and the need to reduce weight. The principal advantage of using TMCs is the weight saving in elevated temperature applications [1]. Perhaps the most significant high temperature application of the type of composite reported herein is the TMC bladed ring (bling) [2]; this will eliminate the conventional dovetail blade arrangement by allowing radical design changes in aeroengine compressors [3]. Bling designs exploit the longitudinal strength of the composite to carry high hoop stresses as well as maintaining sufficient transverse strength to carry the radial load. Demonstrator bling components have been manufactured and component weight reductions of up to 70% are envisaged [2]. Components for other applications such as links and actuators for moving exhaust flaps (giving a 40% weight saving over IN 718 parts) and engine ducts and cases (giving a 40% increase in stiffness and 50% weight reduction compared with conventional monolithic titanium components) have also been manufactured from TMCs [2,4]. The CMC material reported on is also a candidate material for such high temperature aeroengine applications [5]. Given the nature of the application, the effect of thermal cycling in these materials is considered to be an aspect of their behaviour worth investigating. To date little systematic work has been carried out to investigate the effect of thermal fatigue on candidate TMCs in atmospheric air. The work reported herein has been conducted under environmental conditions which were considered to be relevant (in atmospheric air and to the limits of the expected operating temperature envelope) to the intended applications.

2 EXPERIMENTAL

2.1 MATERIALS

The starting materials used were a unidirectional titanium alloy/SiC composite and a barium osumilite glass/SiC ceramic composite. The titanium matrix composite was based on a matrix of Ti-6%Al-4%V, an alpha-beta titanium alloy, with coated (Sigma) SiC fibres manufactured by DERA (Advanced Metal Composites Ltd.), Farnborough. The nominal fibre diameter was 100 μm , and the mean interfibre spacing was 45 μm . The Sigma 1240 fibres consist of a tungsten core (12 μm diameter), a 100 μm SiC fibre, a 1.3 μm diameter carbon coating (put down in two sub-layers, and a 1.3 μm TiB_x layer [6]. This outer layer consists of a mixture of TiB₂, TiB and free boron. The composite was received in the form of sheet, 1.4 mm thick. Specimens were cut, using a wire erosion technique, from the sheet to give specimen test

pieces with fibres orientated in the 0° and in the 90° directions; giving $(0)_8$ and $(90)_8$ lay-ups respectively. Accurate positioning of the specimens was maintained throughout the cutting operation to ensure maintenance of the correct fibre orientation. After the specimens were cut to shape, the orientation of the fibres was checked before any mechanical testing was commenced. A diagram showing the dimensions of the specimens is shown in Figure 1.

The barium osumilite glass ceramic composite was a cross ply $(0,90)_{12s}$ composite received in the form of sheet, 3 mm thick, and was supplied by AEA Technology plc, Harwell. The material was cut into the form of “dog-bone” shape specimens, whose dimensions are shown in Figure 2.

2.2 TEST PROCEDURES

In the first instance MMC (gauge length 20 mm) and CMC samples (gauge length 25 mm) of the dimensions shown in Figures 1 and 2 respectively were subjected to uniaxial tensile testing to rupture at room temperature. In order to minimise the risk of cracking of the sample at the grips, aluminium tabs were bonded onto the ends of the samples. The tabs and the corresponding specimen ends were coated with epoxy adhesive and a layer of silicon carbide grit introduced between the tab and the specimen end; this arrangement is illustrated in Figure 3. The tab-epoxy-abrasive-epoxy-composite sandwich was then bonded and allowed to harden. A thin layer of epoxy on both the surface of the tab and that of the specimen end ensured that the silicon carbide grit was bonded in contact with both the ductile tab (into which the grit makes an impression) and the specimen. Samples were then tested at room temperature in an Instron 8561 servo-electric machine under uniaxial tension until rupture occurred. Extension was measured by means of a side entry extensometer. Whilst it was realised a strain gauge could have been used, it was felt that due to the oft occurring random, localised nature of failure in these types of material, an extensometer would be more appropriate for the measurement of strain over the entire gauge length, provided that accurate specimen alignment could be ensured. Firstly the specimen was loaded into the machine and aligned. The double extensometer was positively located on the side of the specimen, to avoid slipping, by means of freshly mixed high temperature cement. Wet cement was carefully positioned on the side face of the specimen, then the extensometer (zeroed) driven into the wet cement to make an impression, withdrawn to allow the cement to dry, and then reintroduced to the specimen so that the extensometer conformed to the now dry indentation left in the cement.

Samples were optically aligned prior to closure of the grips and before testing in order to avoid the generation of bending stresses. The loading rate used in the uniaxial tests was 100 N sec^{-1} , with the test rig running under load control. Sample position, extension and applied load were recorded by datalogging software at a rate of 5 Hz. After testing the specimen fragments were retained for fractographic examination and for sectioning to examine the specimen microstructure.

Further batches of samples were subjected to a thermal cycling regime prior to tensile testing to failure at room temperature. These were 0° and 90° MMC samples and the $(0/90)$ CMC. Samples were tested in a Severn Furnaces R1071 thermal cycling rig. The MMCs were cycled 100 times from room temperature to 450°C , 550°C , 650°C and 700°C respectively. The heating and cooling rates used were both 4°C.sec^{-1} ; the total duration of cycling was

approximately five hours. The CMCs were cycled 100 times from room temperature to 500°C, 600°C and 700°C respectively at the same heating and cooling rates as the MMCs. The maximum temperatures used covered the range in which interfacial changes might be anticipated.

Of the thermally cycled specimens (both CMC and MMC), half were subjected to uniaxial tensile testing to rupture at room temperature in the same manner as the samples tested without any prior heat treatment, while the rest were reserved for mechanical fatigue testing; also at room temperature.

Mechanical fatigue testing was undertaken on both the 0° and 90° MMCs and the CMC, in both the as-received and in the heat treated conditions, in uniaxial tension. Samples were aligned and gripped in the same manner as in the monotonic test regime. For all mechanical fatigue testing, a stress ratio of $R=0$ was used. Samples were cycled from zero load to 2 kN in the case of the CMC (nominal stress of 0.10 GPa) and the 90° MMC (nominal stress of 1.18 GPa). For the 0° MMC, samples were cycled from zero load to 7 kN (nominal stress of 4.13 GPa). These loads were based on the perceived commencement of inelastic behaviour in the monotonic loading regimes; this will be discussed in greater detail in the results section. As with the monotonic testing, constant load amplitude fatigue testing was performed under load control, using a uniform linear loading and unloading rate and a cycling frequency of 0.1Hz; thus giving a “saw tooth” profile. Load controlled fatigue is the most common control mode for these materials, primarily because of the limitation of thin TMC specimens in supporting compressive loads [7]. All samples were subjected to 20,001 cycles, unless premature catastrophic failure occurred. Due to the nature of the software operating the datalogger, it was not possible to start a fatigue test from the zero quadrant of a fatigue cycle; i.e. under this test regime, zero load. Samples were therefore loaded in this case to the mean load used in the fatigue regime (i.e. 1 kN or 3.5 kN as applicable) using a simple ramp control program and held at the mean load whilst the control file for the fatigue test was loaded. Data from the ramp loading stage was logged at a rate of 10 Hz. The fatigue regime was then commenced in the first quadrant, again at 1 kN or 3.5 kN as applicable. Data were logged during the fatigue regimes in a periodic manner, with every thousandth cycle being recorded, commencing with cycle 1 (i.e. cycles 1, 1001, 2001, etc. logged), at a rate of 10 Hz.

Metallographic and ceramographic examination of the composites (matrices and interfaces) were undertaken in the as-received state, after the various thermal treatments, and after post-thermal cycling tensile testing and mechanical fatigue testing. Samples selected for metallographic examination were sectioned at their mid points to give an in-plane cross section or a cross section perpendicular to the tensile axis and then cold embedded in a hardenable resin. Mounted specimens were ground and then diamond polished, with a finishing stage performed using a colloidal silica suspension.

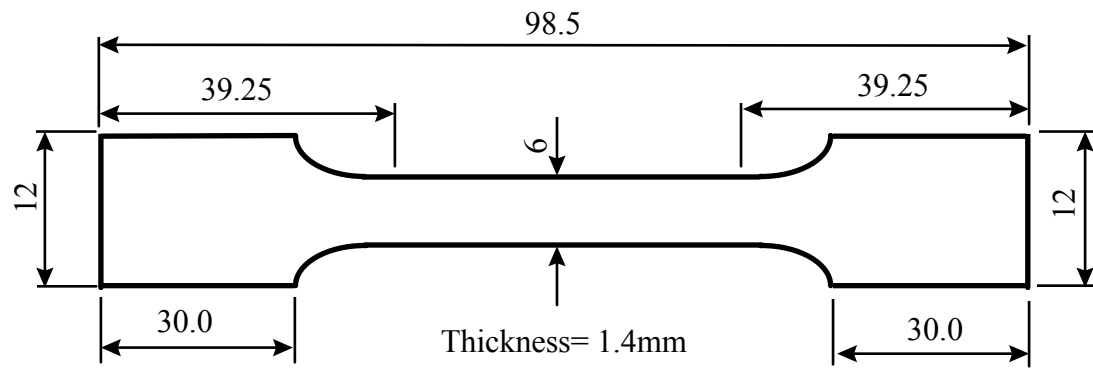


Figure 1: MMC Tensile/Fatigue Specimen Dimensions (mm)

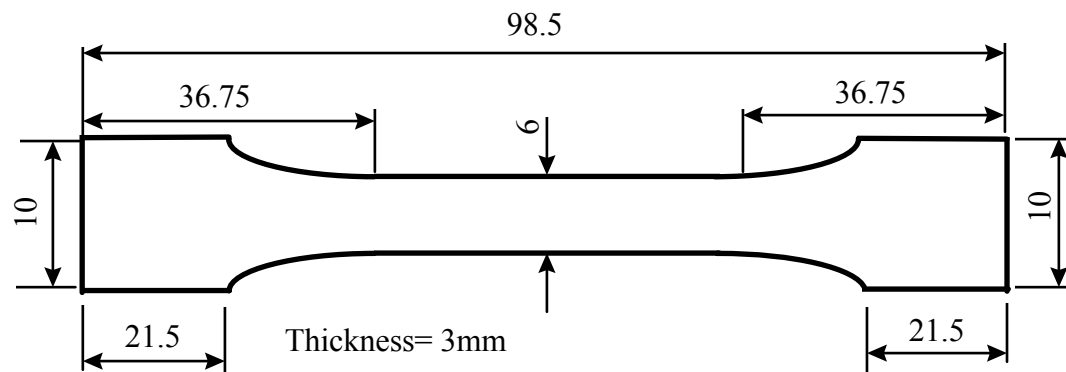


Figure 2: CMC Tensile/Fatigue Specimen Dimensions (mm)

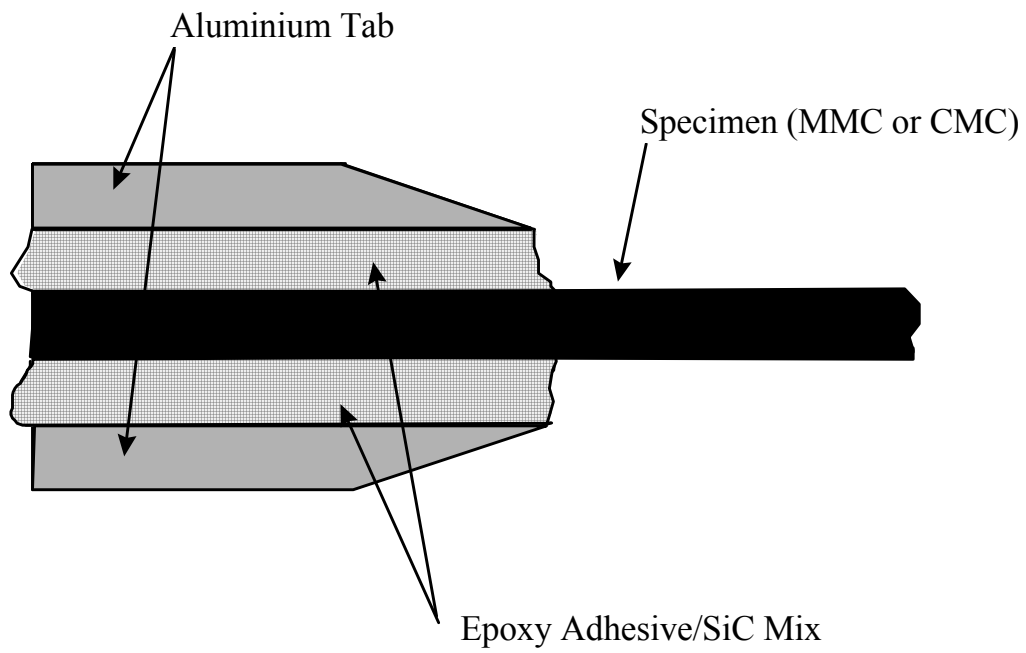


Figure 3: Specimen End-Tab Bonding Arrangement

3 RESULTS

3.1 Metallographic and Ceramographic Examination of Interfaces

A schematic diagram of a cross section through a fibre and the interfacial regions in the MMC is shown in Figure 4.

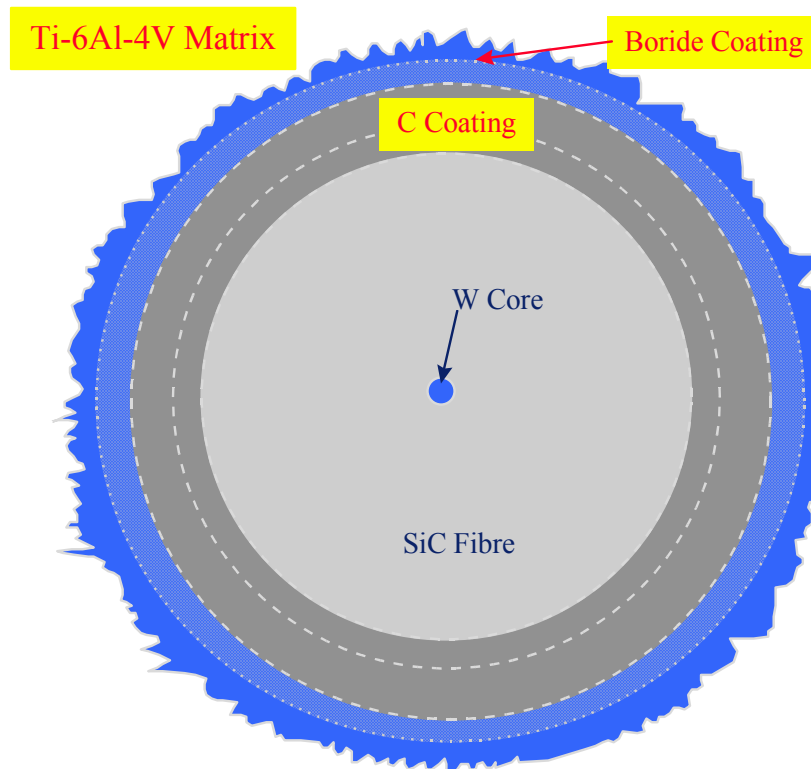


Figure 4: Schematic of Fibre and Interfacial Layers in MMC (Ti-6Al-4V + Sigma 1240 SiC Fibre)

0° and 90° MMC: As-Received and After Thermal Cycling (Without Subsequent Tensile Testing)

There was no detectable degradation in terms of changes in the appearance of the interfaces after thermal cycling across the temperature range under investigation (relative to the as-received samples so examined); both in terms of cracking and debonding. Despite careful ceramographic preparation, localised spalling of the boride layer had occurred both in the as-received and the thermally cycled specimens.

90° MMC: Post-Tensile in The As-Received Condition and After Thermal Cycling

The samples were sectioned at the mid-point after testing and then surfaces parallel to the tensile axis were polished for examination (see Figure 5), enabling fibre and interface cross-sections to be studied.

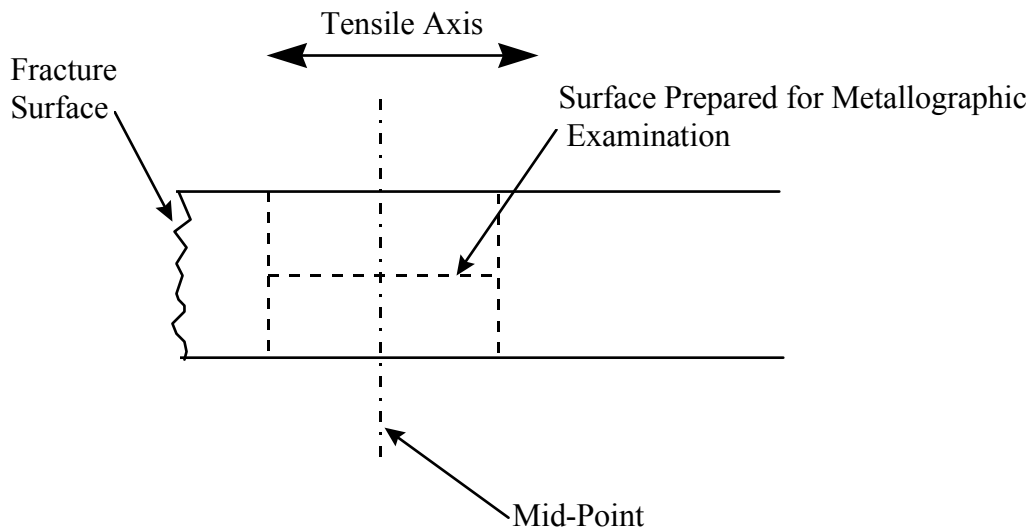


Figure 5: Schematic Diagram of Sample Sectioning for Metallographic Preparation and Examination

After cycling to 450°C and then tensile testing, there appeared to be tangential detachment occurring between the carbon sub-layers, most noticeably in the interfacial regions perpendicular to the tensile axis; see Figure 6. There were also fine radial cracks in the outer (boride) layer perpendicular to the tensile axis.

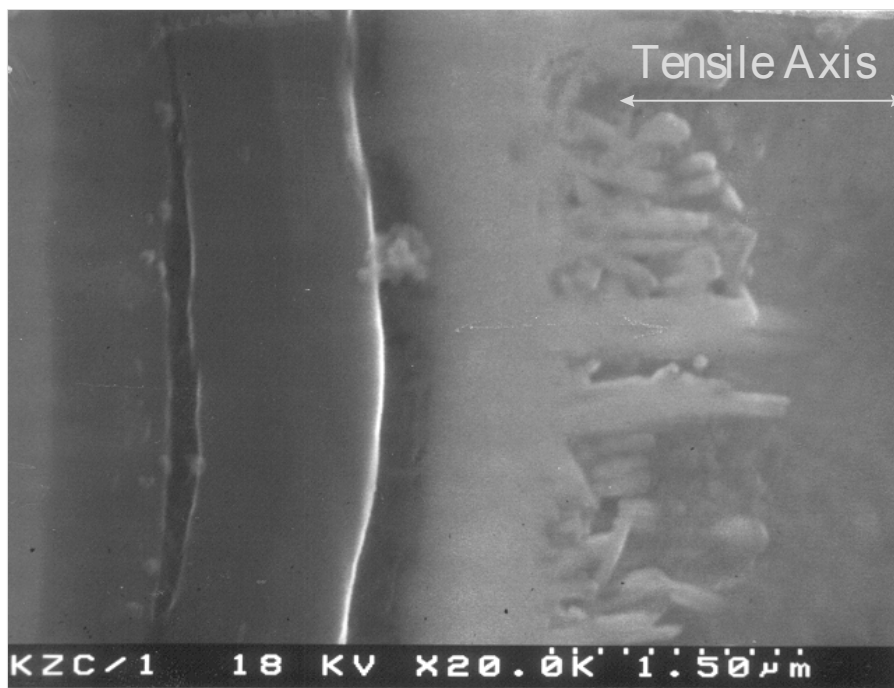


Figure 6: 90° MMC After Cycling to 450°C and Tensile Test

After cycling to 550°C and then tensile testing, tangential detachment of what appeared to be sub-layers within the carbon layer, and also tangential detachment between the outer carbon sub-layer and the boride layer was observed to have occurred, again in the regions perpendicular to the tensile axis (Figure 6). Discrete, radial cracks were also observed in the

boride layer (deviating only approximately $\pm 5^\circ$ from the perpendicular to the tensile axis). Also within the carbon layer, radial cracks were present that showed a similar range of orientations to their counterparts in the boride layer. Whilst cracks were frequently found in both layers within quite close proximity ($\sim 5 \mu\text{m}$), rarely were single tangential cracks found which had traversed both layers; the more common occurrence was seen to be discrete tangential cracks in each layer, with substantial interlayer (carbon-boride) separation between the cracks.

After cycling to 650°C and then tensile testing, short range radial cracks were seen in sectioned samples extending approximately $3 \mu\text{m}$ into the matrix from the outer boride layer (see Figure 7). These radial cracks were orientated approximately $\pm 20^\circ$ from the perpendicular to the tensile axis and appeared to deviate little within the matrix from their radial orientation. There was also no detectable evidence of blunting.

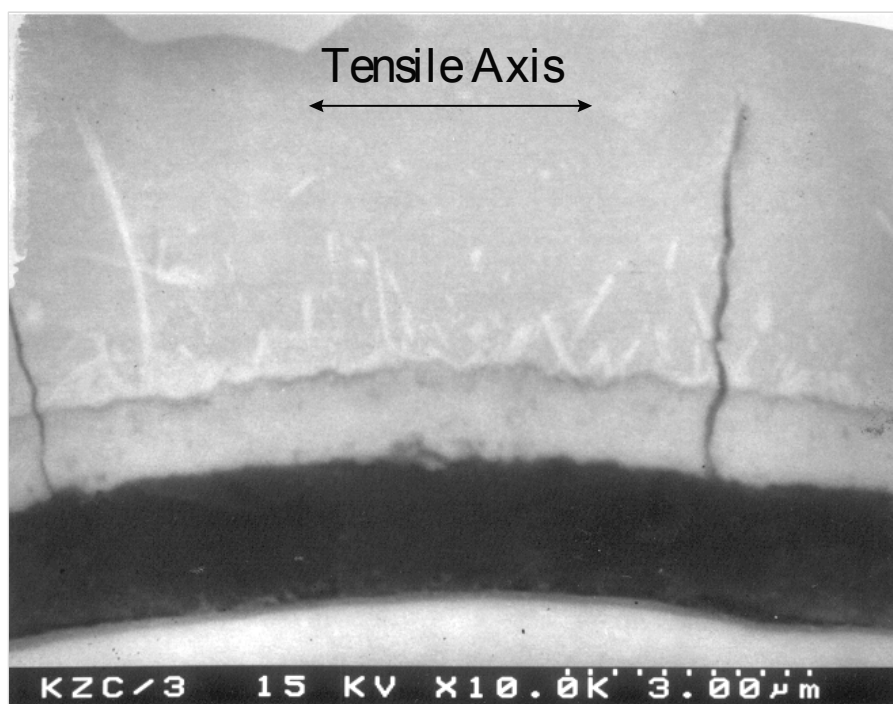


Figure 7: 90° MMC After Cycling to 650°C and Tensile Test

After cycling to 700°C and then tensile testing, short range tangential cracks were seen in sectioned samples extending approximately $3 \mu\text{m}$ into the matrix from the outer boride layer. (see Figure 8). However, in this case there appeared to have been substantial crack tip blunting, both in the boride layer and in the adjoining matrix. The condition of the matrix after cycling to 700°C was further investigated by performing microhardness tests; which indicated an appreciable softening of the matrix had occurred; discussed in section 4.1.



Figure 8: 90° MMC After Cycling to 700°C and Tensile Test

CMCs ((0,90) Laminates)

The samples were sectioned in the as-received condition and after thermal cycling respectively. Samples tensile tested in the as-received condition and in the post thermally cycled condition were sectioned at the mid-point after testing in the same manner as the MMCs and then surfaces parallel to the tensile axis were polished for examination, enabling fibre and interface cross-sections to be studied in a similar manner to the 90° MMCs. In the unstressed samples, whether as-received, or after thermal cycling, no interface damage could be detected at all. However, of the post-tensile specimens, cross sections polished parallel to the tensile axis did show (partial) debonding of fibre-matrix interfaces in the 90° fibres in those previously cycled to 600°C and 700°C respectively (see Figures 8, 9, 10). The debonding observed was also linked to inter-fibre cracking through the matrix, and could encompass several fibres, giving a crack-debond-crack system, whose net direction was approximately parallel to the tensile axis. Locally, such cracks were orientated between approximately 15° and 60° to the tensile axis, and were noticeably more numerous in the sample cycled to 700°C and then tensile tested than that cycled to 600°C and tensile tested.

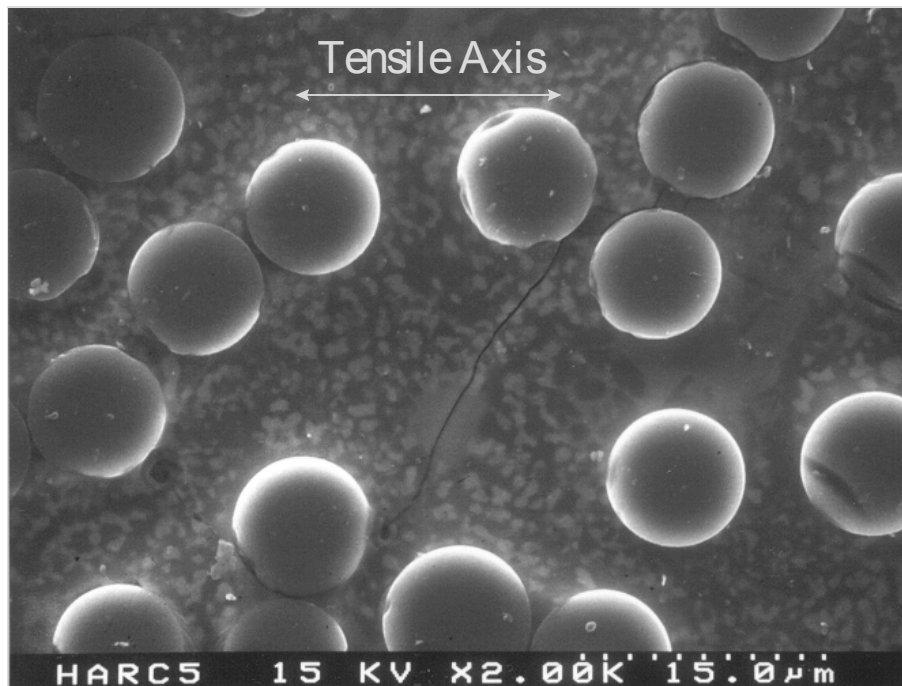


Figure 9: CMC After Cycling to 700°C and Tensile Test

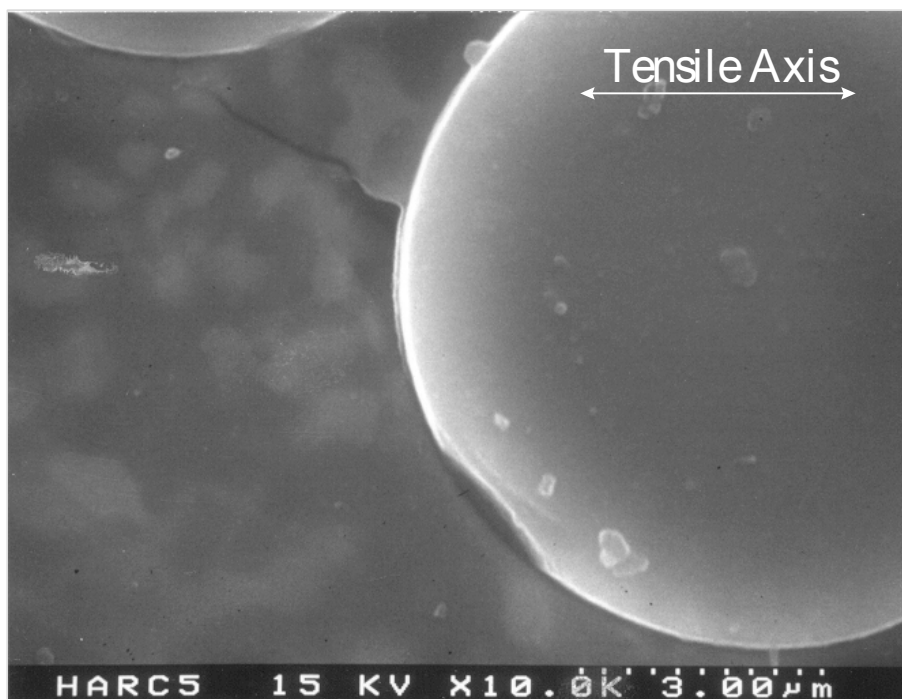


Figure 10: CMC After Cycling to 700°C and Tensile Test (Remote from Fracture Surface)

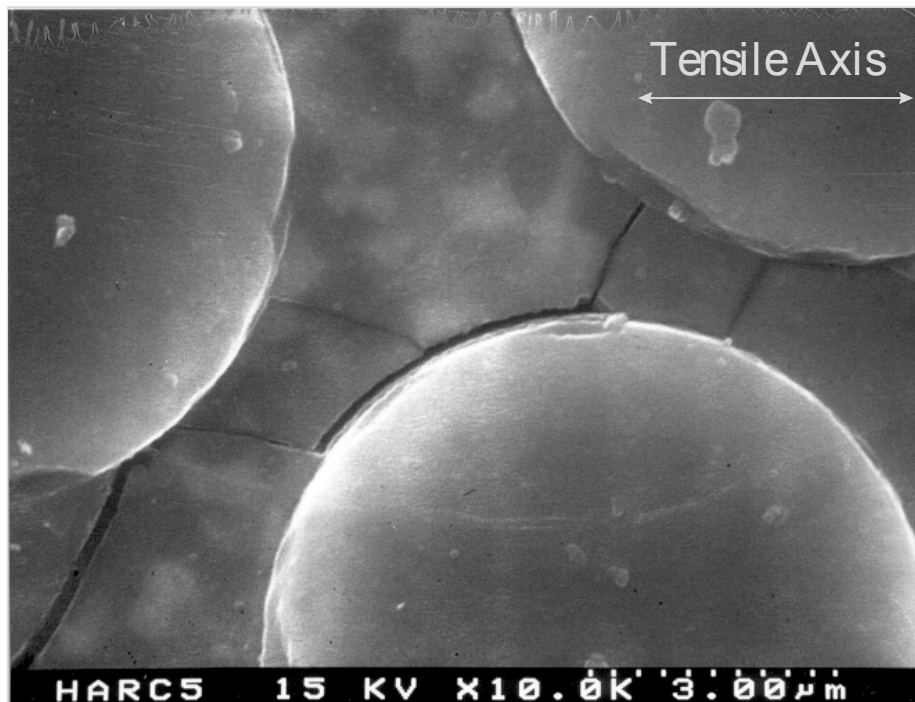


Figure 11: CMC After Cycling to 700°C and Tensile Test (Remote from Fracture Surface)

3.2 Uniaxial Tensile Testing

Data from the uniaxial tensile tests were plotted in the form of load-extension responses. From these initial loading modulus, the strain at which inelasticity first occurred and the strain to failure were extracted and summarised.

3.2.1 90° MMCs

From the summary tensile plot shown in Figure 12 and the bar chart of ultimate tensile strength values shown in Figure 13, it can be seen that all of the thermal treatments degraded the ultimate tensile strength (UTS) of the 90° MMCs. However, this drop is small (around 5% maximum), and could even be accounted for by slight differences in specimen alignment. The important difference is in the strain to failure; see Figure 14. In the sample cycled to 450°C there is almost no change in this parameter, whilst after treatments at 550°C and 700°C, ultimate strain increased, but a dramatic fall off in strain to failure was seen in the sample previously having completed 100 thermal cycles to 650°C. It can be seen that most of the plots are bilinear; an initial linear extension was recorded, followed by an inelastic region, thereafter an almost perfectly plastic zone was seen. These responses are, by convention, denoted as stage I, stage II and stage III respectively. Stage III can be seen to be virtually absent from the sample cycled at 650°C, implying a drastically curtailed plastic response. During stage I the modulus of the as-received specimen is almost twice that of any of the thermally treated specimens; 89 GPa (see Figure 12). The modulus during stage I appears to be relatively insensitive to thermal cycling temperature (ranging from 47 GPa to 57 GPa), save for the slight stiffening (15% increase relative to the other thermally treated specimens) after the 650°C treatment. Departure from linearity (i.e. the end of stage I) was not

significantly affected by thermal treatment, apart from at 650°C, where the proportional limit increased (in applied stress) by approximately 10%.

Summary Tensile Plot for 90° MMCs

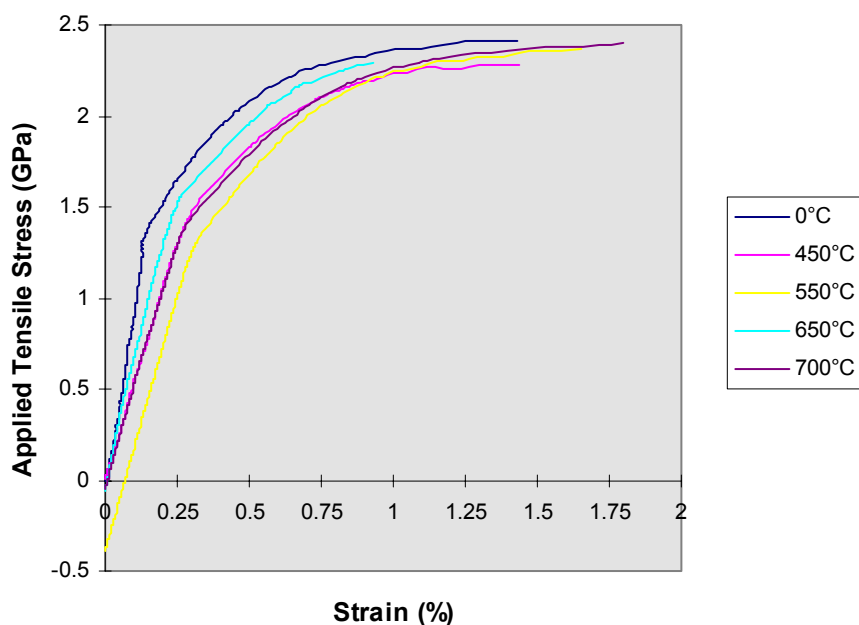


Figure 12: Summary tensile plot for 90° MMCs in the as-received condition (0°C) and after thermal cycling (to 450, 550, 650 and 700°C).

Ultimate Tensile Strength vs. Thermal Cycling Temperature for 90° MMCs

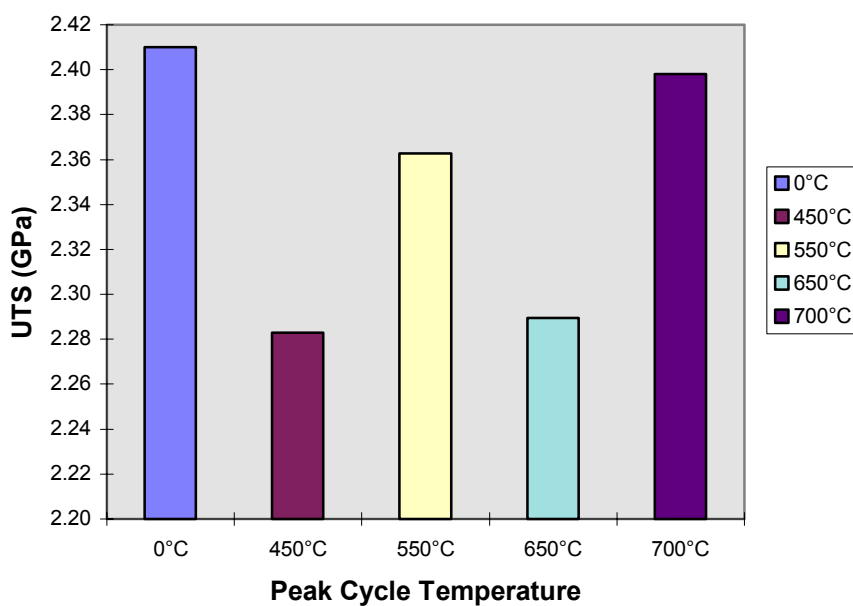


Figure 13: Bar chart of ultimate tensile strength of 90° MMCs in the as-received condition (0°C) and after thermal cycling (to 450, 550, 650 and 700°C).

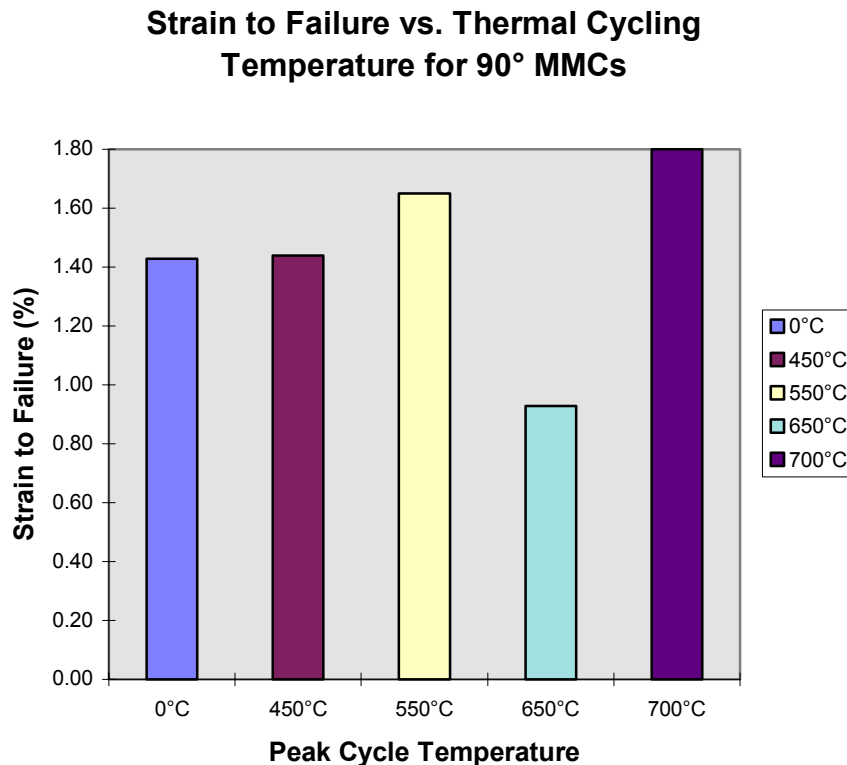


Figure 14: Bar chart of strain to failure of 90° MMCs in as-received condition (0°C) and after thermal cycling (to 450, 550, 650 and 700°C).

3.2.2 0° MMCs

In terms of ultimate tensile strength (see Figure 15 and Figure 16), there was a progressive drop with cycling temperature, save for the sample which had undergone 100 thermal cycles to 650°C; in this case UTS recovered to 99.5% of the strength achieved by the as-received specimen. Prior thermal cycling was seen to have reduced strain to failure in all cases, although amongst those samples thermally cycled, no general trend could be observed as far as this parameter was concerned; some recovery in strain to failure was seen in the sample cycled to 650°C prior to uniaxial tensile testing. In the initial stages (i.e. low load) of uniaxial tensile loading, all samples, with the exception of those previously cycled to 550°C and 650°C showed a linear response. It can however be seen that the mean modulus taken across the zero load to 7kN range (just below the proportional limit where applicable), thermal cycling has stiffened the response, apart from that performed up to the maximum temperature of 700°C. In the latter case the modulus closely matches that of the as-received specimen. The strain to failure of the 0° MMCs, relative to the as-received specimen, can be seen to drop for all thermal treatments by between 20% and 40% (see Figure 17), but no clear trends emerge in terms of relative changes in ultimate strain with the different elevated temperatures. In terms of strain to failure, the 650°C sample is least affected.

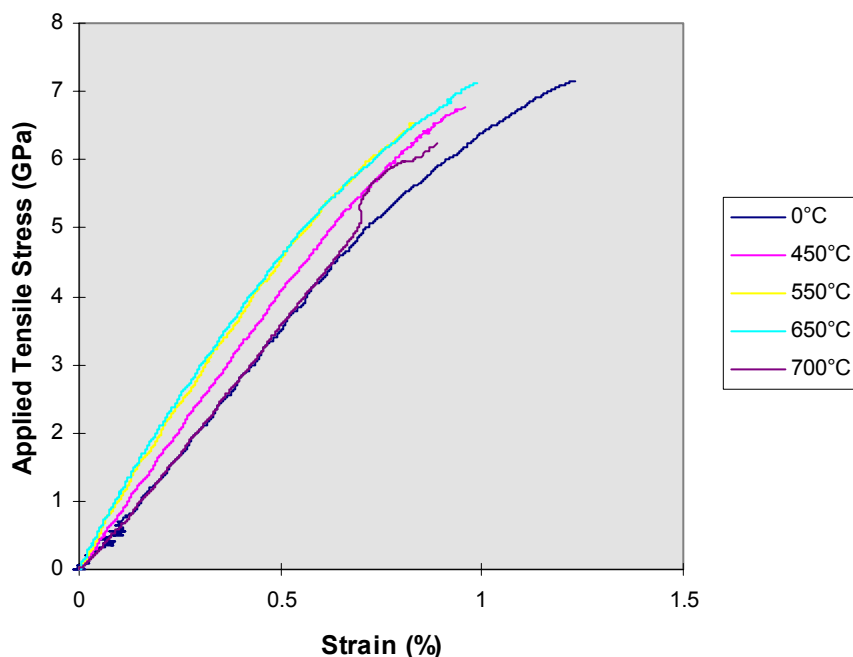
Summary Tensile Plot for 0° MMCs

Figure 15: Summary tensile plot for 0° MMCs in as-received condition (0°C) and after thermal cycling (to 450, 550, 650 and 700°C). Tests performed at room temperature.

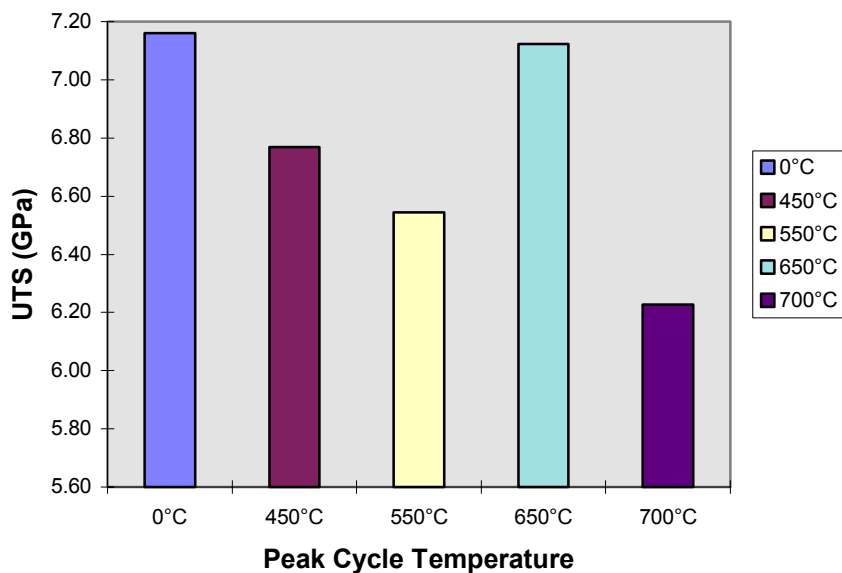
Ultimate Tensile Strength vs. Thermal Cycling Temperature for 0° MMCs

Figure 16: Bar chart of ultimate tensile strength of 0° MMCs in as-received condition (0°C) and after thermal cycling (to 450, 550, 650 and 700°C). Tests performed at room temperature.

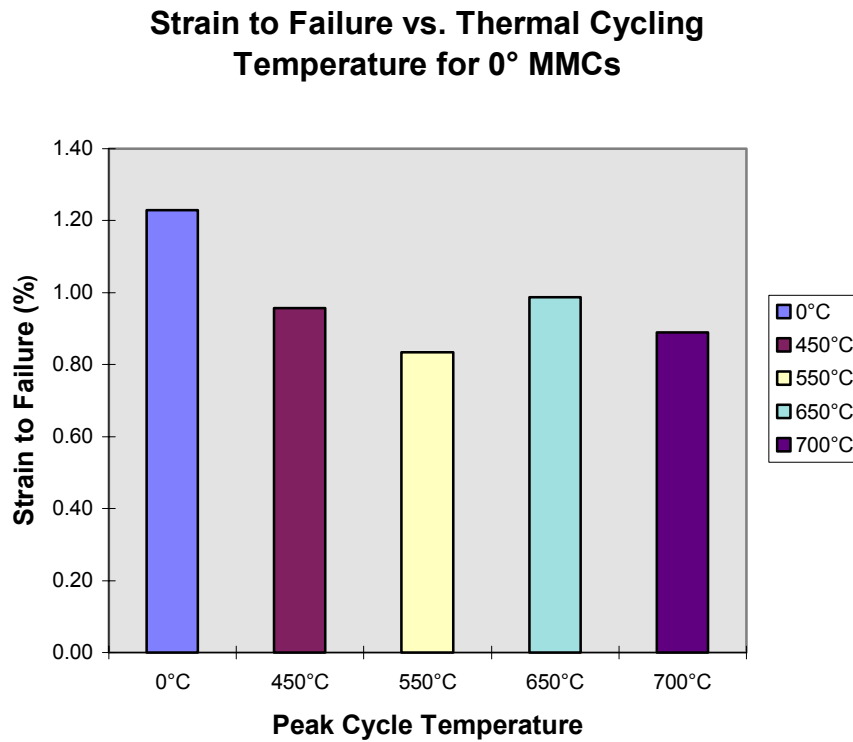


Figure 17: Bar chart of strain to failure of 0° MMCs in as-received condition (0°C) and after thermal cycling (to 450, 550, 650 and 700°C). Tests performed at room temperature.

3.2.3 CMCs ((0,90) Laminates)

It can be seen that the effect of thermal cycling on the uniaxial tensile behaviour of the ceramic matrix composite is to produce a pronounced bilinear response (see Figure 18 for a summary of results), rather than a near-linear response with a gradually decreasing tangent modulus. The bilinear response was seen in all samples subjected to peak thermal cycling temperatures of 500°C, 600°C and 700°C respectively. The response can be characterised by three stages. Stage I is an initial linear stress/strain response, stage II a non-linear “knee”, and stage III a final, approximately linear response. It will be observed that the initial tangent modulus in the sample tensile tested in the as-received condition is comparable with the moduli of the thermally fatigued specimens across most of their stage I response. There also appears to be little effect of cycling temperature (across the temperature range investigated) on the modulus value during stage I of the stress-strain response; it is consistently higher than the tangent modulus of the uncycled specimen across the same strain range. Also the onset of non-linearity in the stress-strain response of the thermally cycled specimens does not seem to vary with peak cycling temperature. The most notable changes to the uniaxial tensile response of the CMCs to rupture were in the ultimate tensile strength (see bar chart shown in Figure 19). There is a progressive reduction in ultimate tensile strength across the range of peak thermal cycling temperatures investigated, until after cycling to 700°C, the value has dropped to approximately 65% of the strength attained by the sample in the as-received condition. In terms of strain to failure (shown in bar chart form in Figure 20), the effect of cycling to 500°C is seen to be minimal, but thereafter there is a sharp and continued reduction in strain to failure with increased peak thermal cycling temperature, until after cycling to 700°C, the strain to failure is approximately 55% of the as-received value.

Summary Tensile Plot for CMCs

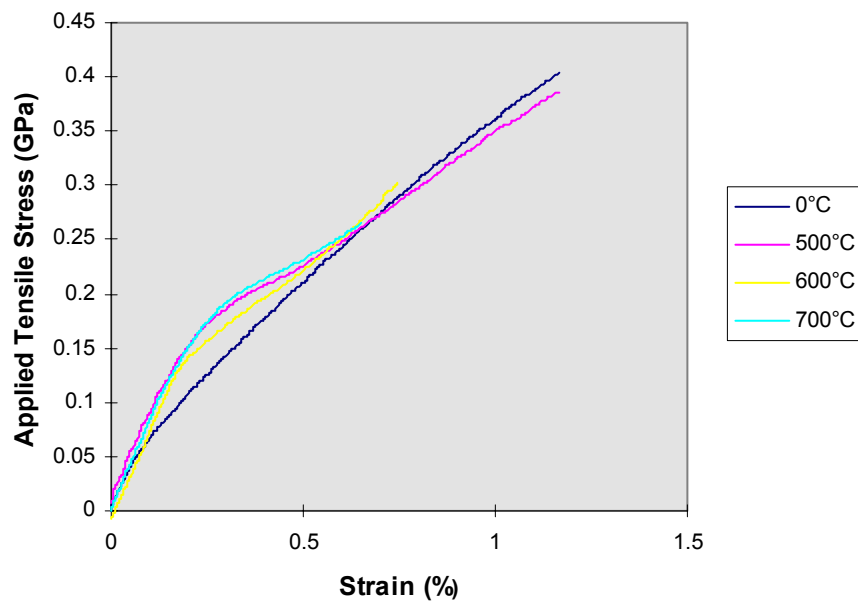


Figure 18: Summary tensile plot at ambient temperature for CMCs in the as-received condition and after thermal cycling (to 500, 600 and 700°C).

Ultimate Tensile Strength vs. Thermal Cycling Temperature for CMCs

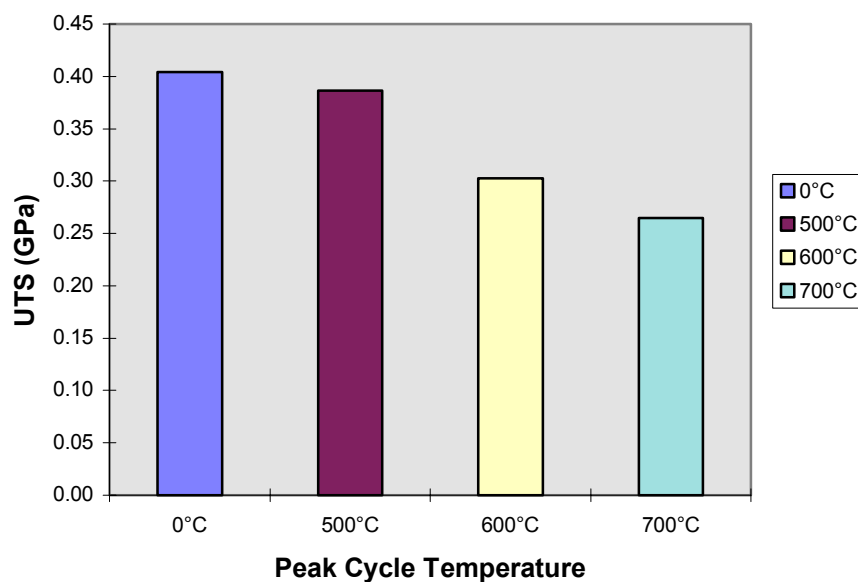


Figure 19: Bar chart of ultimate tensile strength of CMCs at ambient temperature in as-received condition and after thermal cycling (to 500, 600 and 700°C).

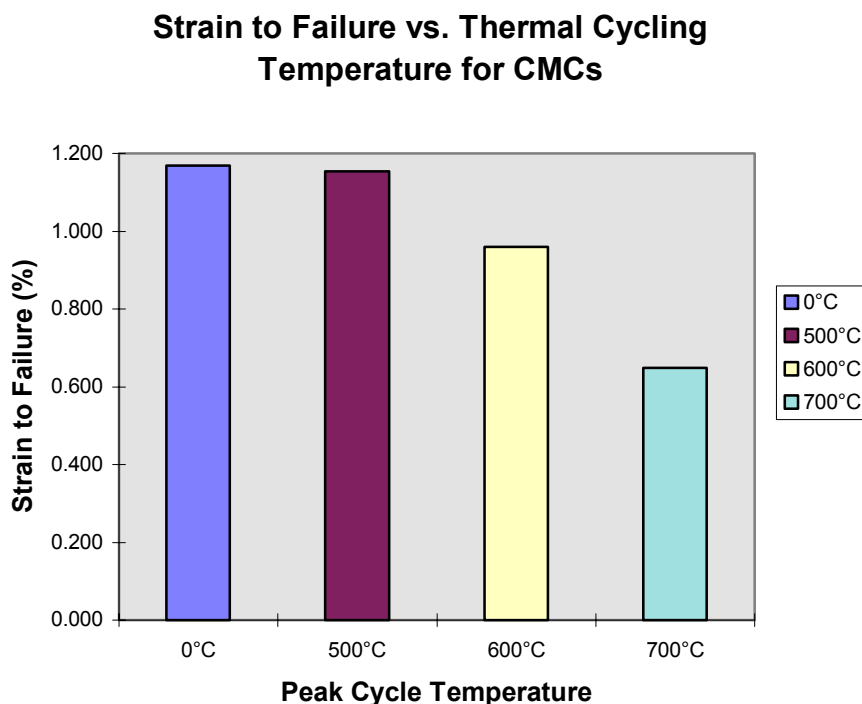


Figure 20: Bar chart of strain to failure of CMCs at ambient temperature in as-received condition and after thermal cycling (to 500, 600 and 700°C).

3.3 Fractography on Post-Tensile Specimens

Fracture surfaces of samples subjected to uniaxial tensile testing were examined after rupture to determine the bulk fracture modes in the matrices and to examine the fibre-matrix interface after rupture.

3.3.1 90° MMCs

Fractographs of the MMC tensile tested in this orientation can be seen in Figure 21 and Figure 22. In the main body of the matrix, the fracture pattern was one of dimpled rupture which had occurred by tensile overload. In the matrix regions just beyond the outer fibre coating, failure appeared to be by mode I, deduced on the basis of the apparent directionality of the dimples, in. This was confirmed by examining fracture surfaces on both fragments of the ruptured test pieces. There were isolated regions of debonding between the SiC fibre and the matrix, but this occurred in all samples, irrespective of cycling temperature, or whether tested in the as-received condition. The bulk matrix fracture mode and that in the matrix did not change perceptibly with cycling temperature.

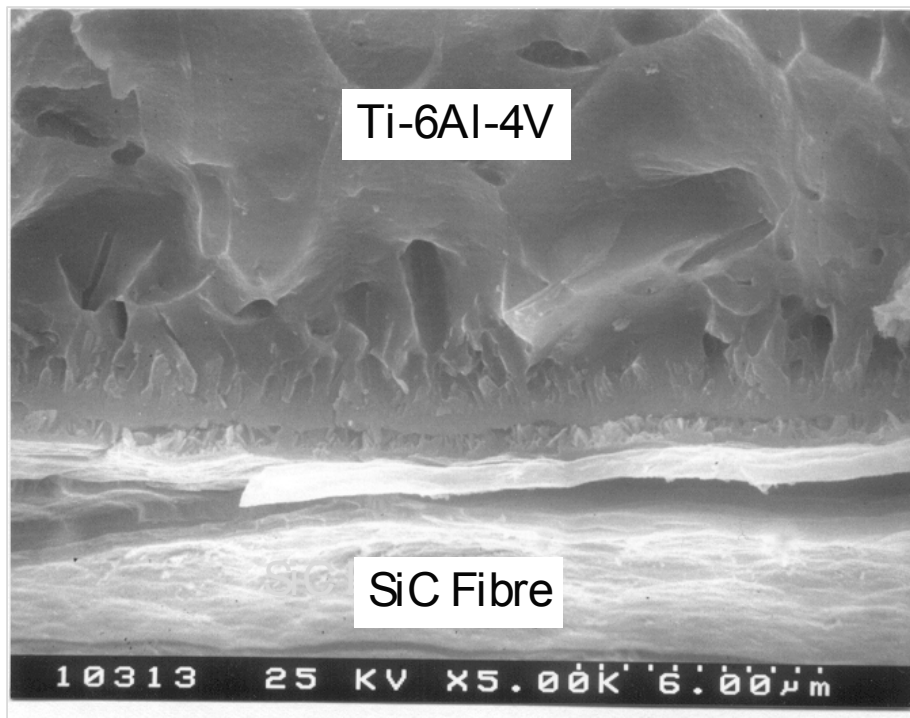


Figure 21: Interfacial fracture region in 90° MMC Cycled to 650°C and ruptured under uniaxial tensile load

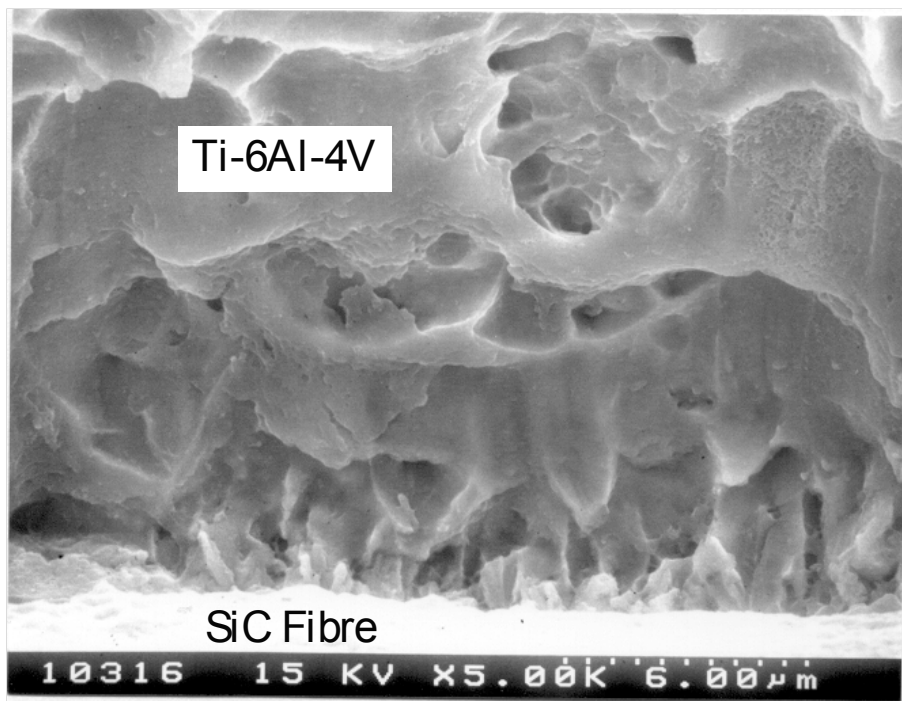


Figure 22: Interfacial Fracture Region in 90° MMC Cycled to 700°C and Ruptured under Uniaxial Tensile Load

3.3.2 0° MMCs

It was observed that in the main body of the matrix the most common fracture mode was ductile rupture by tensile overload. From observations of sectioned post-tensile samples and

the actual fracture surfaces, it appears that ultimate failure was by tensile overload (ductile rupture) in the matrix. However, there also appeared to be isolated areas of quasi cleavage. Pronounced steps in the matrix between fibres and orientated parallel to the tensile axis were often seen, primarily in the as-received sample; perhaps indicating multiple origins of catastrophic failure. In the vicinity of the matrix-fibre interface in the as-received sample and that cycled to 450C, both mode I fracture and shear deformation appeared to have occurred, whereas in the samples cycled to higher temperatures, shear deformation was the far more predominant mode at the interface. It also seemed to be the case at the higher temperatures that the extent of shear deformation in the matrix surrounding the fibres was far greater (see Figure 24 and Figure 25), and the contribution of dimpled rupture due to tensile overload conversely smaller, prior to final (catastrophic) failure occurring in the matrix. A certain amount of fibre pullout occurred in all specimens, with no discernible trends seen due to thermal cycling. However, radial fibre-matrix separation was far more marked in the as-received sample than those which had undergone thermal cycling (see Figure 23), though this may be due to more pronounced Poisson effects in the matrix at the higher strains to failure seen in the as-received sample. After cycling to the higher temperatures there appeared to be much more widespread cracking within the outer fibre layers, with both radial and tangential cracks seen (see Figure 24 and Figure 25).

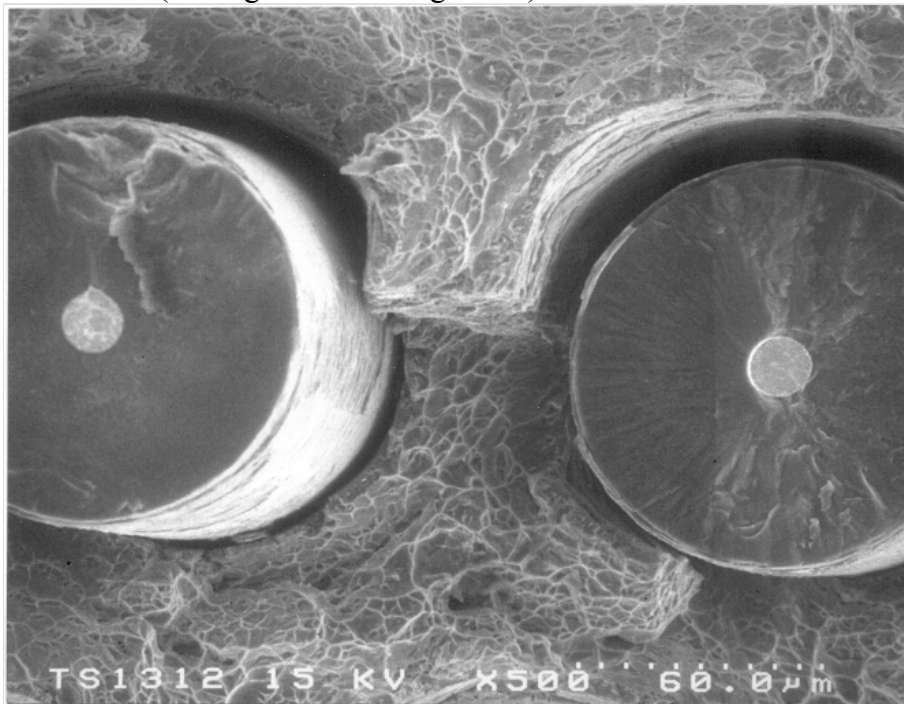


Figure 23: Fracture Surface in 0° MMC As-Received (not thermally cycled) and Ruptured under Uniaxial Tensile Load

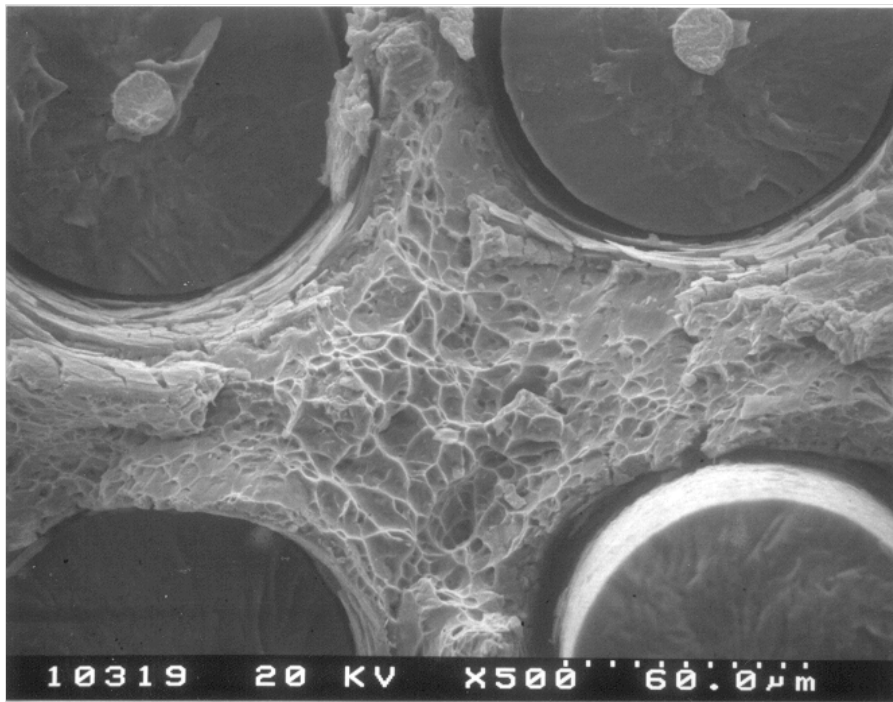


Figure 24: Fracture Surface in 0° MMC Cycled to 650°C and Ruptured under Uniaxial Tensile Load.

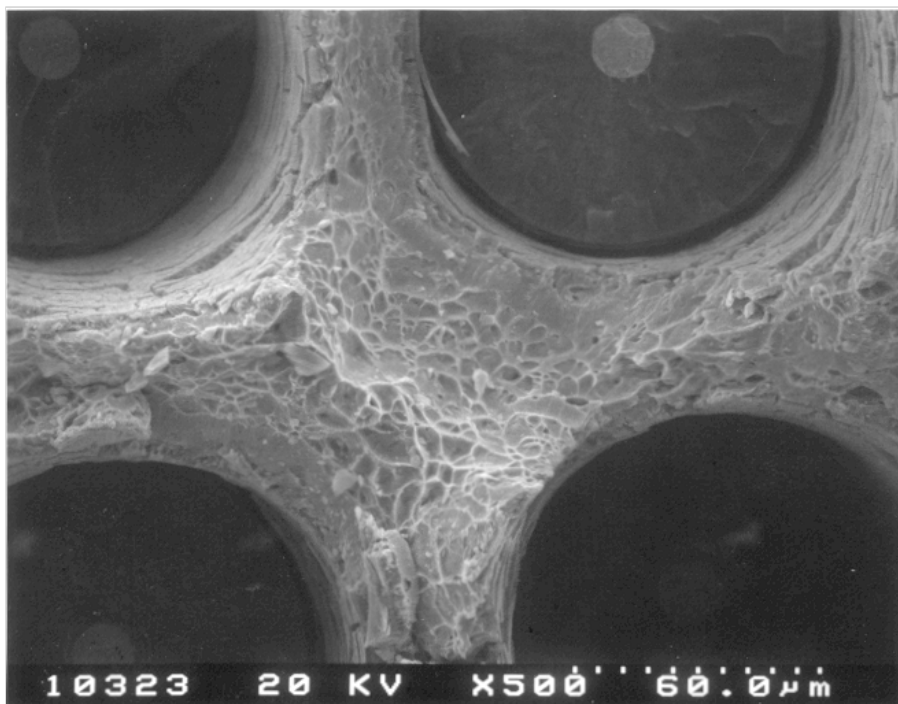


Figure 25: Fracture Surface in 0° MMC Cycled to 700°C and Ruptured under Uniaxial Tensile Load

3.3.3 CMCs ((0,90) Laminates)

Fibre fracture was seen to occur in a brittle manner, with failure origins identifiable in most cases; these were seen to be both internal and surface origins. There appeared to be no

discernible trends in location of failure origins with thermal cycling temperature. Significant pullout had occurred in the 0° orientated fibres prior to final failure of all specimens examined, though extensive delamination in surface 0° plies was observed only in the specimen tested in the as-received state. Samples sectioned and polished after tensile testing to give ceramographically prepared surfaces parallel to the tensile axis (including a section of the fracture surface) showed that large bundles of 0° fibres had detached themselves by matrix cracking; where the matrix cracks encountered fibres, debonding of the fibre-matrix interface occurred, rather than fibre fracture. Frequently the whole fibre circumference was seen to have undergone debonding. Cracks extending up to $\sim 400\ \mu\text{m}$ were seen to extend back from the fracture surface, approximately parallel to the tensile axis, and often along the interface between the 0° and the 90° plies, though they often also deviated to run diagonally through the 90° ply (by matrix cracking and 90° fibre interface debonding) to eventually encounter the next ply interface. Such crack systems were seen to commonly alternate several times between two adjacent ply interfaces (i.e. two $0^\circ/90^\circ$ and $90^\circ/0^\circ$ interfaces) by means of off-axis propagation through the 90° ply by matrix cracking and matrix-fibre debonding); see Figure 28.

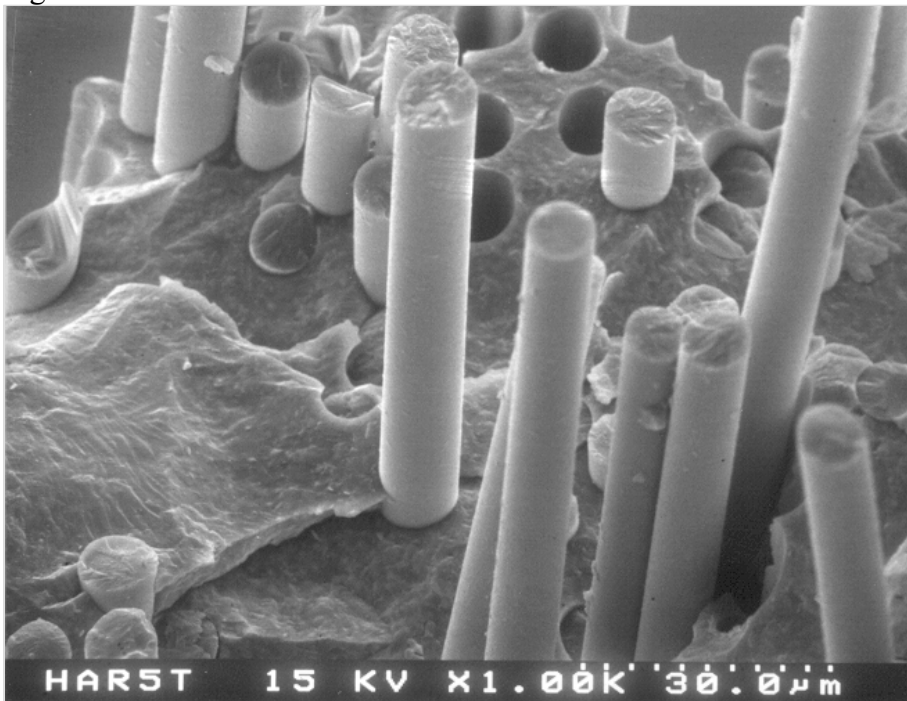


Figure 26: Fracture Surface of CMC Cycled to 700°C (90° Fibres)

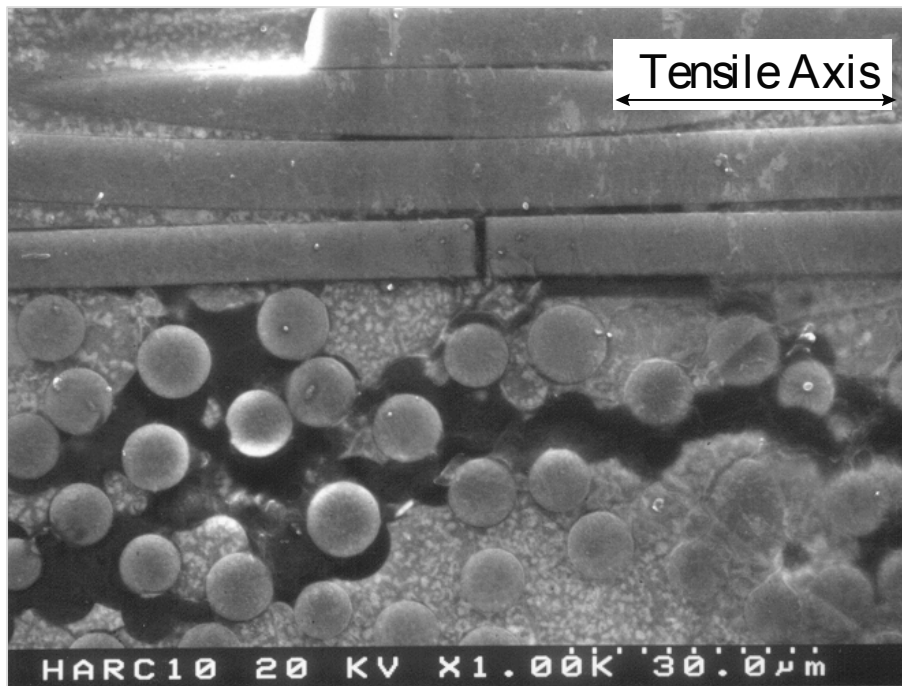


Figure 27: Cross Section of Region Near Fracture Surface in 600°C Thermally Cycled CMC

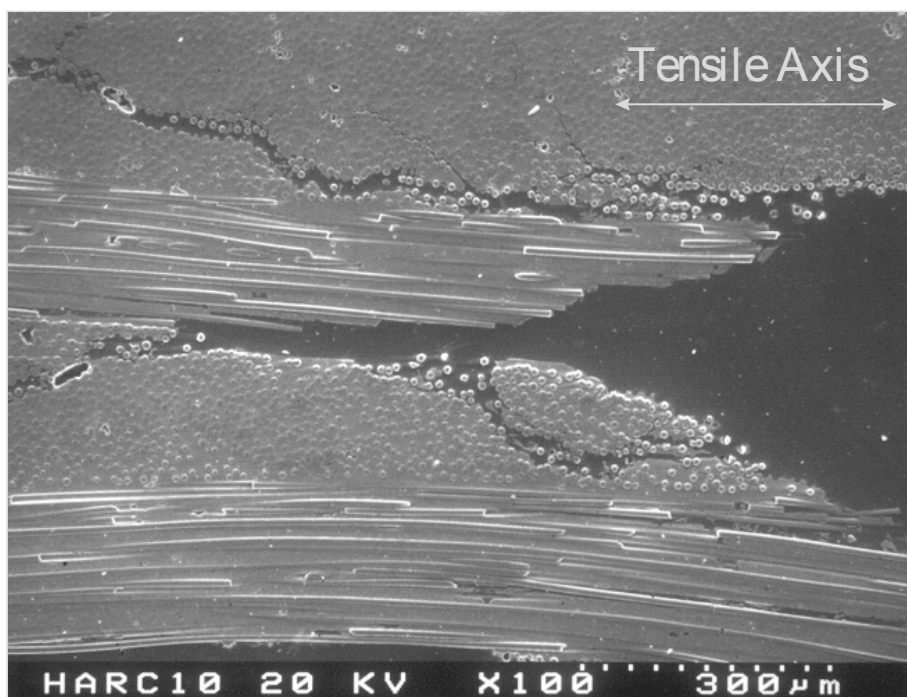


Figure 28: Cross Section Including Region of Fracture Surface in 600°C Thermally Cycled CMC

3.4 Uniaxial (Tensile) Fatigue Testing

Due to the number of data points logged, summary details were extracted from the raw data, where necessary manipulated, and plotted in summary graphs. The load-extension responses for the first and last recorded cycles were plotted; these were usually the first and 20,001st cycles, but where premature rupture had occurred, the last recorded cycle was plotted. The

unloading (secant) modulus was plotted for the cycles recorded, as well as the net cumulative inelastic strain (i.e. strain offset on unloading, at zero load).

3.4.1 90° MMCs

The maximum load used in the fatigue regime was based on the onset of stage II of the stress-strain response under the uniaxial tensile test regime; the reasoning being that inelastic behaviour might be expected if the onset of stage II was entered, but that premature catastrophic failure could be avoided by not using peak loads towards the upper limit of stage II. Because only every 1000th cycle was recorded, plots of individual cycles in themselves were not considered to be of great value. However, parameters which could be extracted for the whole of the fatigue regime and plotted in summary form can be useful in trying to establish trends in behaviour. To this end, secant (unloading) modulus and inelastic strain (relative change in strain offset at zero load after unloading) have been plotted and are shown in Figures 29 and 30 respectively. In terms of modulus, the most striking feature to observe is that all the thermal treatments used degrade secant modulus, implying that damage is experienced by the 90° MMCs after each of the thermal cycling regimes used. However, no clear trends emerge as far as the relative effects between the elevated cycling temperatures are concerned. Modulus values obtained in the as-received composite are reasonably stable across the mechanical fatigue regime, as are those produced by the specimens previously cycled to 550°C and to 700°C respectively. The sample previously cycled to 450°C prior to mechanical fatigue can be seen to have undergone a slight drop in modulus across the test regime. The behaviour of the sample previously cycled to 650°C is markedly different in that an initial increase in modulus is seen (from the first to the 1001st cycle), followed by a perceptible and continual drop across the whole of the mechanical fatigue regime. This would imply progressive damage occurring at a steady rate.

Inelastic strain at zero load for the unloading part of each cycle has been plotted and shows changes in strain relative to that at zero load on the loading portion of the first cycle (see Figure 30). Since the inelastic strain is plotted for zero load, it should correspond to purely plastic strain. It can be seen from Figure 30 that for the specimen in the as-received condition that there is a slight increase in inelastic strain across the mechanical fatigue test regime; this is also the case for those samples previously thermally cycled to 450°C and 550°C and 700°C. The behaviour of the sample cycled to 650°C prior to mechanical fatigue was very different; a large initial increase in inelastic strain in between the first and the 1001st cycles, followed by a progressive drop across the remainder of the mechanical fatigue test regime. It will be observed that the large initial change in modulus of this sample occurred across the same initial part of the fatigue regime.

Modulus Behaviour of Thermally Treated 90° MMCs During Mechanical Fatigue Tests

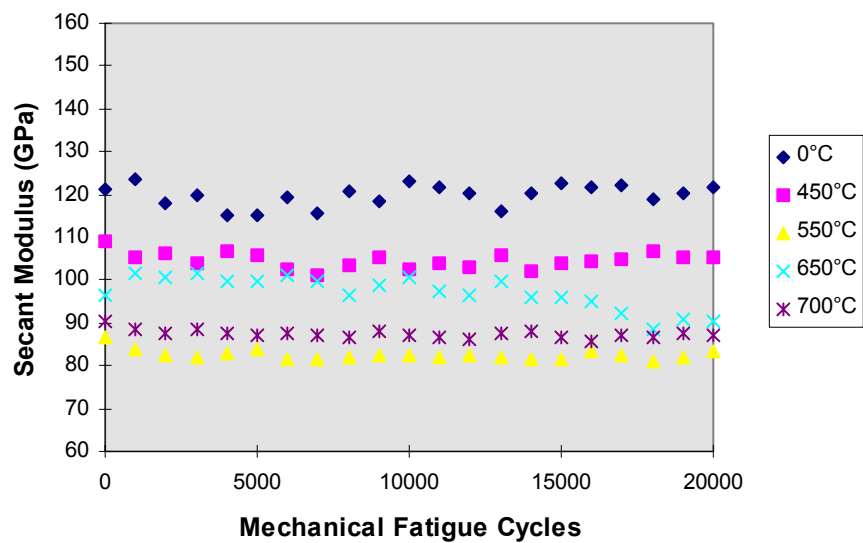


Figure 29: Secant (unloading) modulus in mechanical fatigue regime for 90° MMC.

Inelastic Strain Produced in 90° MMCs Previously Subjected to Thermal Cycling

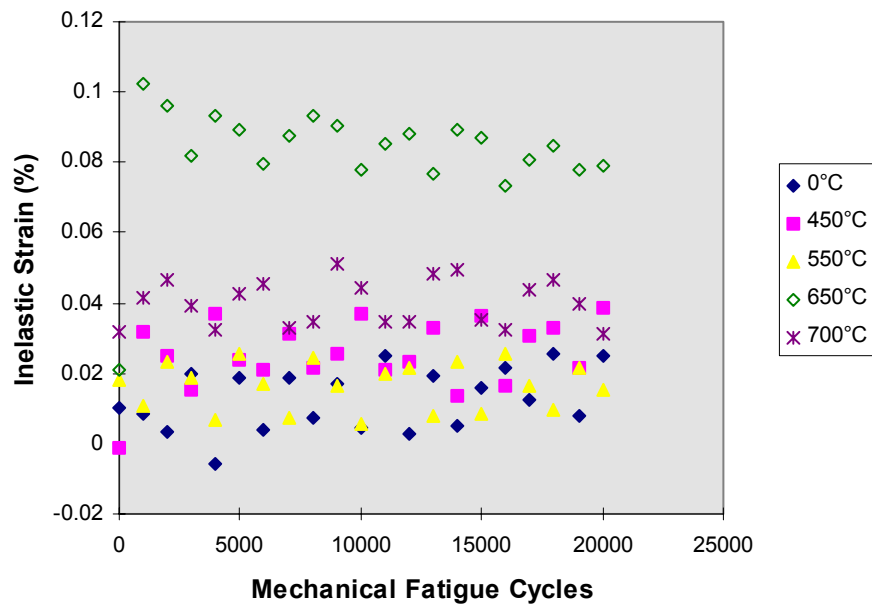


Figure 30: Inelastic strain (at zero load on unloading cycle) in mechanical fatigue regime for 90° MMC.

3.4.2 0° MMCs

The maximum load used in the fatigue regime was based on the proportional limit (where applicable) seen in the monotonic uniaxial tensile responses of these samples. Secant (unloading) modulus and inelastic strain data have been extracted from individual fatigue plots in a similar manner to the 90° MMCs; see Figure 31 and Figure 32. In general terms, the changes in modulus were not as great as those seen in the 90° MMCs; this was probably due to the in-plane stiffness contributions from the SiC fibres. As can be seen, the effect of cycling to 450°C is minimal, but though modulus was degraded by cycling to higher temperatures, thereafter it remained reasonably constant; there being little variation between values obtained after cycling to 550°C, 650°C or 700°C. Also, in terms of modulus, there was little prior warning of premature failure; however since cycle logging was only performed for every 1000th cycle, any modulus degradation occurring before failure is unlikely to have been captured [7]. Equally, fatigue cracks in these materials usually initiate early in the regime and then propagate extremely slowly (relative to the unreinforced equivalent titanium) [8].

The only sample of this type to actually complete the test (20 001 cycles) was that which had been thermally cycled to 700°C. Fatigue crack growth through the matrix at least in this material is likely to have been impeded by its far greater ductility than all the other samples. The fatigue life of titanium- 0° SCS6 silicon carbide composites (SiC with carbon coating only) has been found to be strongly dependent on the maximum strain produced in the fatigue regime [9], this in turn has a profound effect on the cumulative damage mechanisms which can operate. In the case of the fatigue regime reported here, the net strains produced at the peak load during each cycle (within the bounds of 0.30% and 0.65% strain) should affect fatigue life by inducing both fibre, interface and matrix damage, with premature failure occurring in at least some of the samples; the latter has been confirmed in the present study. Fatigue lifetimes in Ti-SiC composites are usually classified according to maximum strain at failure and the failure mechanism. Briefly, region I corresponds to catastrophic failure (chiefly fibre failures), region II corresponds to fibre, matrix and interface cracking (progressive damage) and region III corresponds to runout [10-12]. Relatively few results have been reported on the isothermal fatigue of Ti-SiC composites in the stage II failure region; i.e. where fatigue life is governed by behaviour of matrix, fibre and interface. However, the lifetimes found for the 0° TMCs reported here do fall into the bounds of region II both in terms of cycles to failure and maximum strain at failure for 0° carbon coated SiC titanium composites (no published data exist for composites with SM1240 fibres under the same or similar conditions) [13]. Conversely, that runout occurred in all of the 90° composites agrees with other data (though again for composites with 6-SCS fibre, not SM1240); fatigue behaviour in these materials, like their 0° counterparts, has been found to fall into three regions on an S-n curve [14].

In terms of changes to inelastic strain across the mechanical fatigue regime (Figure 32), the sample in the as-received condition and also that cycled to 450°C both show a progressive increase across the regime. Note that the figures showing plots of inelastic strain give the apparent inelastic strain in each cycle, not the accumulated strain. The sample cycled to 550°C showed an initial increase in inelastic strain and thereafter a slight drop. That cycled to 650°C shows a large initial drop followed by a progressive, steady deterioration (i.e. apparent shrinkage); perhaps indicative of a progressive phase change [15] or work hardening in the matrix across the test regime.

Modulus Behaviour of Thermally Treated 0° MMCs During Mechanical Fatigue Tests

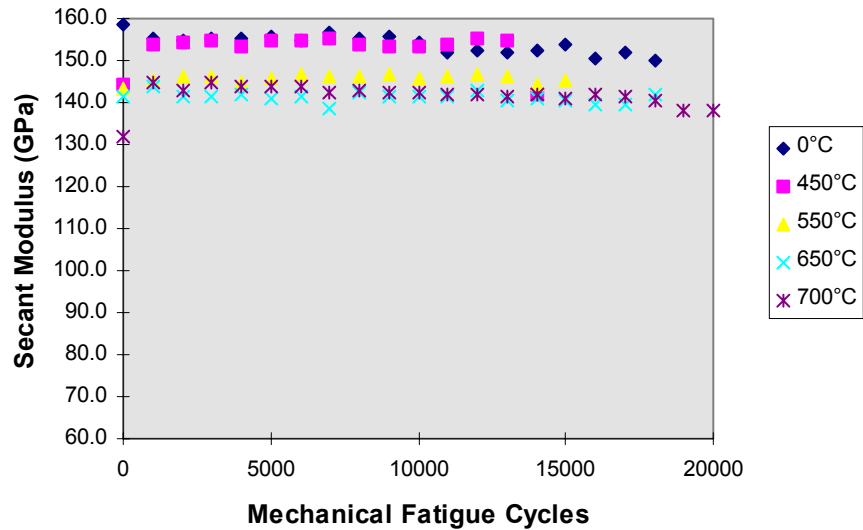


Figure 31: Secant (unloading) modulus in mechanical fatigue regime for 0° MMC.

Inelastic Strain Produced in 0° MMCs Previously Subjected to Thermal Fatigue

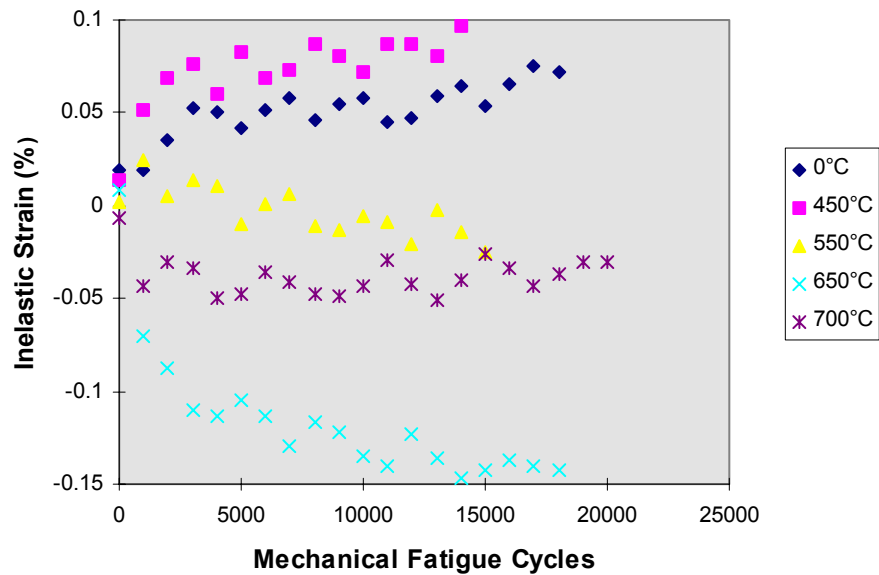


Figure 32: Inelastic strain (at zero load on unloading cycle) in mechanical fatigue regime for 0° MMC.

3.4.3 CMCs ((0,90) Laminates)

The maximum load used in the fatigue regime was based on the lowest value of the deviation of stage I behaviour from linearity, amongst the thermally fatigued composites. As in the case of the MMCs, because only every 1000th cycle was recorded, plots of individual cycles in themselves were not considered to be of great value. However, parameters which could be extracted for the whole of the fatigue regime and plotted in summary form can be useful in trying to establish trends in behaviour. To this end, secant (unloading) modulus and inelastic strain have been plotted and are shown in Figures 32 and 33. In terms of modulus, the most striking feature to observe is that all the thermal treatments used degrade secant modulus, implying that damage is experienced by the CMCs after each of the thermal cycling regimes used. However, no clear trends emerge as far as the relative effects of each thermal treatment are concerned. The effect of each of the cycling treatments is to degrade the secant modulus by approximately one third relative to the as-received condition. It will be observed that the largest increase in inelastic strain is in the samples cycled to 500°C and 700°C respectively, whereas the sample in the as-received condition and that cycled to 600°C showed no net change. One influence on the behaviour of the fatigue response in these materials is likely to be the proximity of the peak applied mechanical fatigue load with respect to the non-linear “knee” of the three stage uniaxial monotonic tensile response. It can be seen that the samples exhibiting the largest increases in inelastic strain are those whose non-linear “knees” of their three stage uniaxial monotonic tensile responses are closest to the peak load during the mechanical fatigue regime. The inelastic strain behaviour of the sample cycled to 500°C implies gradual damage accumulation across the mechanical fatigue regime, whereas the sample cycled to 600°C would appear to undergo considerable damage in the initial stages (within the first thousand mechanical cycles); thereafter little further damage accumulation occurred. These differing behaviours also appear to have a bearing on the secant modulus in these samples; that cycled to 500°C shows a steady, but nonetheless perceptible drop in modulus across the whole of the mechanical fatigue regime, whereas that cycled to 600°C undergoes a substantial drop in secant modulus within the first thousand cycles, thereafter remaining approximately constant.

Modulus Behaviour of Thermally Treated CMCs During Mechanical Fatigue Tests

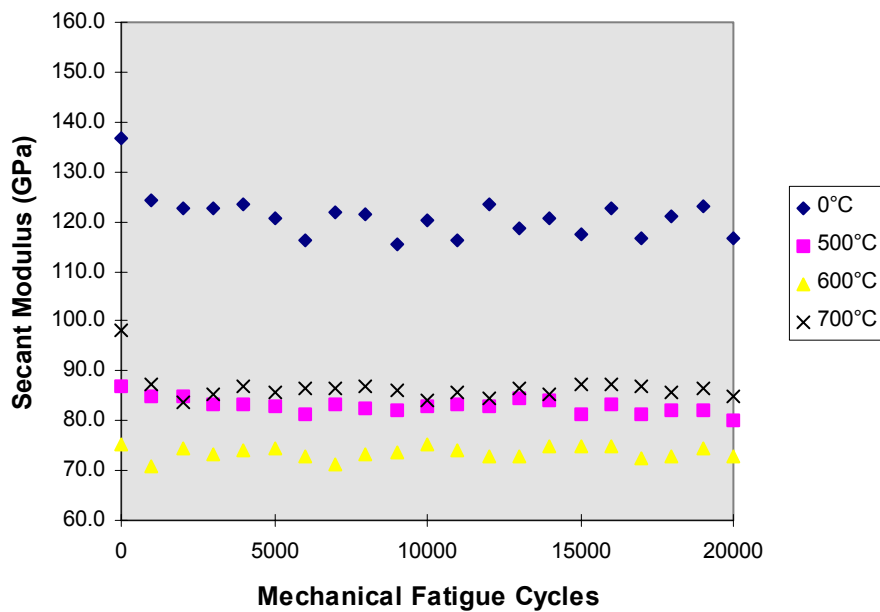


Figure 33 Secant (unloading) modulus in mechanical fatigue regime for CMC.

Inelastic Strain Produced in CMCs Previously Subjected to Thermal Fatigue

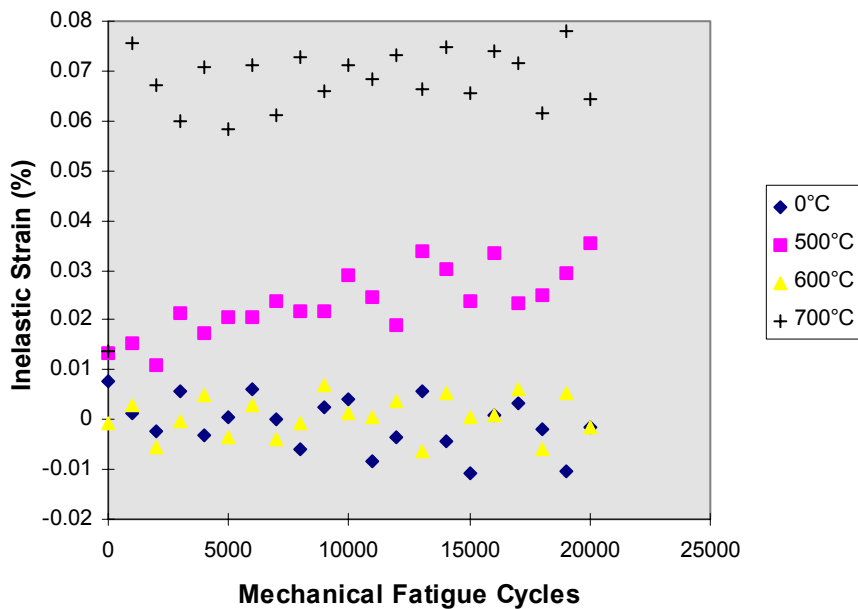


Figure 34: Inelastic strain (at zero load on unloading cycle) in mechanical fatigue regime for CMC.

4 DISCUSSION

4.1 MMCs

It can be seen that the main effect of thermal cycling on interface behaviour is one of increasing the extent of damage in post-tensile specimens, initially by causing debonding in the carbon layer at low temperatures, and then inducing both debonding and cracking in the outer layer. After cycling at 650°C sharp, short range cracks were seen to penetrate into the matrix, but at 700°C extensive crack blunting occurred. In the light of this, an additional investigation was undertaken in order to ascertain the effect on matrix plasticity in the form of microhardness measurements with a Vickers indenter; the results are shown in Figure 35. Clearly some annealing of the matrix has taken place, with the exception of cycling to 650°C, where some recovery has taken place; perhaps indicative of a phase change. Strains in the 90° MMC are governed principally by the state of the matrix and the relative strains at least partly reflect the changes in microhardness, though apparent embrittlement of the sample cycled to 650°C is marked. A possible explanation is the promotion of beta phase formation in the matrix [16]. It is also evident that damage has been induced at all elevated temperatures; this can be seen from the modulus vs. time plots from the fatigue regime in the 90° orientation.

From inspection of sectioned samples and fracture surfaces it would appear that in the 90° orientation, reaction zone cracks are generated at subcritical loads, but that ultimate failure is by the growth of these cracks to a critical size, followed by tensile overload in the matrix; thus at least the condition of the matrix is likely to have a large influence on strain to failure.

In the 0° composites, ultimate failure was also caused by tensile overload in the matrix.

In terms of fatigue lives, the data generated in the present work agree with previous work on this type of composite with 6-SCS fibre [9]. It has been proposed that lifetimes can be estimated by strain response alone, and it would now appear that this might apply regardless of thermal history and fibre coating.

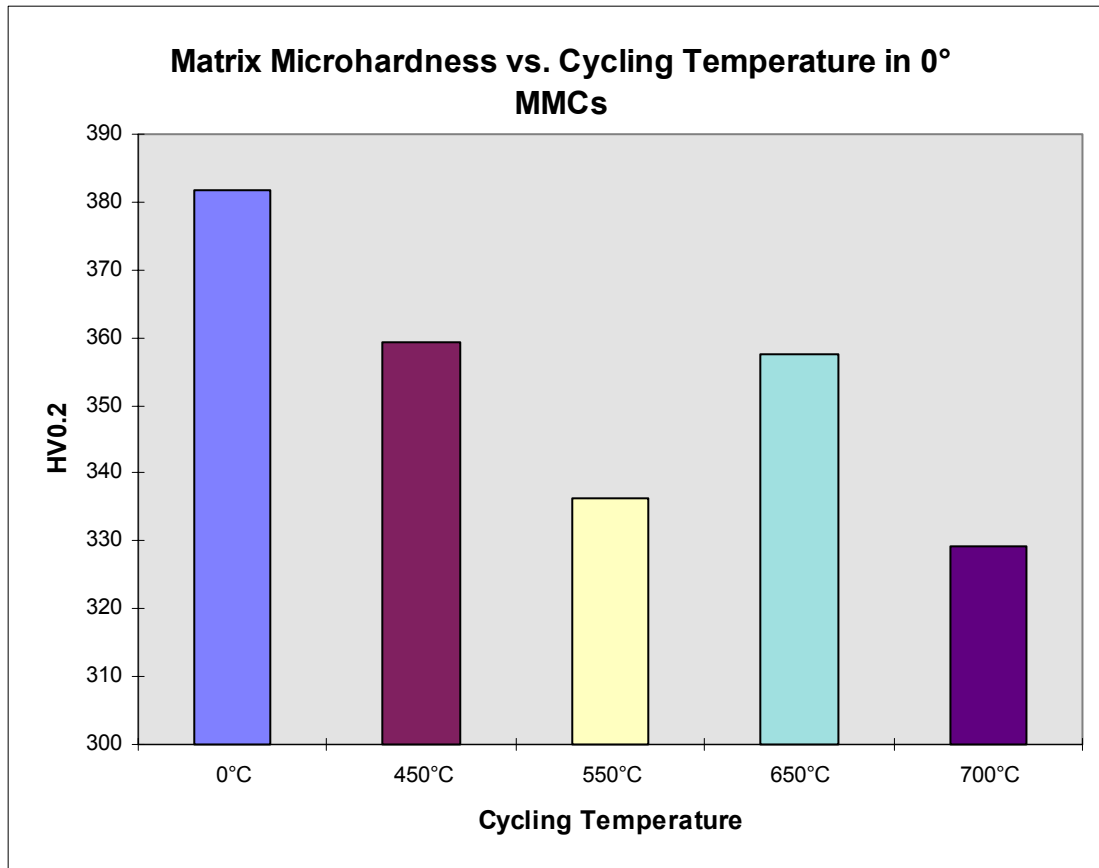


Figure 35: Matrix microhardness vs. cycling temperature for 0° MMCs.

4.2 CMCs ((0,90) Laminates)

In the un-stressed state it appeared that there was no perceptible effect on the interface in terms of debonding, etc., but after tensile testing, debonding coupled with matrix microcracking in the 90° plies became more abundant with progressively higher cycling temperatures. The initial tensile response of the CMCs subjected to thermal cycling was stiffer than those in the as-received condition; this maybe due to the interfaces being less predisposed to sliding, which would also create more favourable conditions for localised microcracking; this does seem to be borne out by the examination of post-tensile sectioned samples. Thermal cycling also produced a bilinear response on tensile testing; it would appear that at least one source of inelasticity was due to the microcracking/debonding regime. It seems probable that the drop in secant modulus after thermal cycling across the fatigue regime can be at least in part attributed to the greater propensity to undergo matrix microcracking and fibre-matrix debonding. The initial tensile response of the CMCs and examination of post tensile specimens after thermal cycling is consistent with increased fibre/matrix bond strength; resulting in interfacial shear stress, due to thermal cycling. Little has been reported on the effect of thermal cycling of CMCs on structure/property relationships, but the changes due to thermal cycling would appear to be consistent with the so-called silica “bridges” at interfaces [17-22]. This silica formation is preceded by oxidation of the carbon coating on the SiC fibre, which leaves the surface of the SiC fibre susceptible to the ingress of oxygen, and provided temperatures are high enough, oxidation reactions can occur [22]. However, the apparent increase in fibre/matrix bond strength in the present study occurred at lower temperatures than other studies. However, these references are to isothermal heat treatments in oxidising atmospheres; in the present work there

may be an additional effect due to thermal cycling. At the present time one can only speculate on reasons for the early onset of the apparent formation of silica bridges. Silica formation is dependent, amongst other factors, on the availability of oxygen at sufficiently high temperatures. It has been found that silica formation can form at fibre ends and then seal the end of the fibre by bridging the interface, preventing further so-called pipeline oxidation [23]. However, should the end seal be broken again, pipeline oxidation will occur again. Repeated heating and cooling of the specimens at relatively fast rates may cause the silica end plugs to break down (thermal shock) and hence enable further pipeline oxidation. It is clear though that thermal cycling does appear to promote interface changes after exposure to lower temperatures in an oxidising atmosphere than isothermal treatments.

5 SUMMARY

In the case of the 90° MMCs, there were no discernible changes to the physical nature of the interfaces after thermal cycling alone. However, subsequent mechanical testing at room temperature induced circumferential interlayer separation within the carbon sublayers after low temperature cycling, with radial crack growth into the matrix occurring after cycling to higher temperatures. However, the latter was also influenced to a large extent by the condition of the matrix. The initial (linear) tensile responses of this MMC would seem to point to changes in the interfaces after cycling to the lowest temperature studied. Ultimate tensile strains in this material it would appear are largely governed by the condition of the matrix after thermal cycling. Fractography indicates that failure initiates in the interface in both composite orientations (reaction zone cracks), but that in the 0° case, this leads initially to mode I failure in the matrix. In the 90° orientation, reaction zone cracks appear to precede shear deformation in the matrix (the latter after fibre fracture). Ultimate failure in both orientations was by tensile overload in the matrix (ductile rupture). However, in the 0° orientation, the relative contributions of shear failure and tensile overload to catastrophic failure appear to be related to matrix condition.

The CMC, like the MMCs, only showed signs of interface degradation (debonding, cracking) after tensile tests. The evidence points to increased matrix/fibre bond strength and hence increased interfacial shear stress, promoting interfacial debonding and inter-fibre microcracking. These changes appeared to have been initiated at the lowest temperature used in the thermal cycling regime, and their extent increased progressively over the regime studied. That changes occurred at the lower end of the thermal test regime and had a deleterious effect on mechanical properties is an important consideration when looking at new (or even existing) applications for this material. It seems likely that the interface changes are ones of carbon layer removal and the subsequent formation of silica by the oxidation of silicon carbide. However, the changes brought about by exposure of the CMC to elevated temperatures in the present study were initiated at lower temperatures than equivalent isothermal studies. In short, it would appear that a genuine thermal cycling effect occurred in the CMC, the nature of which requires further investigation for it to be resolved.

6 SUGGESTIONS FOR FURTHER WORK

Damage in the matrix and in the interfaces of the TMC has been detected after thermal cycling and rupture in uniaxial tension. Work, namely loading and unloading to a subcritical tensile stress, together with appropriate data collection should be able to discriminate between damage and plasticity, and by the use of strain gauges, produce Poisson ratio data. This, together with metallography of sections of the post-tensile specimens ought to give a broader picture of the state of the interfaces and their bearing on macroscopic mechanical properties. Further work could also be undertaken to resolve the causes of the apparently anomalous behaviour of the TMC after cycling to the upper range of temperatures investigated, though there are indications from previous studies that phase changes may be induced in this temperature range [15]; namely the promotion of beta phase formation in the matrix [16].

Further work is also needed to identify more closely the nature of interface changes in the CMC after thermal cycling, and to highlight any additional phenomena not usually associated with an isothermal heat treatment of this type of material in an oxidising atmosphere.

7 REFERENCES

- [1] - K. Upadhyaya: "Developments in Ceramic and Metal-Matrix Composites", TMS, Warrendale, Pennsylvania, 1992.
- [2] - "IHPTET: Technology Teams in Action", Turbine Engine Division, WL/POT, Wright Laboratory, Wright-Patterson Air Force Base, Ohio.
- [3] - P. Grant: "Fibre Reinforced Titanium Matrix Composites", Powder Metallurgy, 1997, 40, 2, 104.
- [4] - S.A. Singerman and J.J. Jackson: "Titanium Metal Matrix Composites for Aerospace Applications", Superalloys 1996 (Proc. 8th Int. Symp. on Superalloys; eds. R.D. Kissinger et al).
- [5] - R.W. Cain (ed.): "Encyclopaedia of Materials Science and Engineering", Supplementary Vol. 2, Pergamon, Oxford, 1990.
- [6] - M. Thomas, DERA: personal communication.
- [7] - W.S. Johnson: Journal of Composites, 1992, 24, 3, 187-196.
- [8] - P. Kantzos, J. Telesman and L. Ghosn: "Fatigue Crack Growth Behavior of SCS-6/Ti-24Al-11Nb Composite as observed Through an SEM Loading Stage", HITEMP Review 1989, NASA CP-10039, 66-1 - 66-10, NASA, Washington, D.C.
- [9] - T.P. Gabb and J. Gayda: Titanium Matrix Composites (eds. S. Mall and T. Nicholas), 169-207, Technomic, Lancaster, Pennsylvania, 1998.
- [10] - P.K. Brindley and P.A. Barlotta: "Isothermal Fatigue Behavior of SiC/Ti-24Al-11Nb", HITEMP Review 1991, NASA CP-10082, 46-1 - 46-13, NASA, Washington, D.C.

- [11] - R.T. Bhatt and H.H. Grimes: "Fatigue Behavior of Silicon Carbide Reinforced Titanium Composite", Fatigue of Fibrous Composite Materials, ASTM STP 723, American Society for Testing and Materials, Philadelphia, Pennsylvania, 1981.
- [12] - S.M. El-Soudani and M.L. Gambone: "Strain Controlled Fatigue Testing of SCS6/Ti-6Al-4V Metal Matrix Composite", Proc. On Fundamental Relationships Between Microstructure and Mechanical Properties of Metal matrix Composites" (eds. P.K. Liaw and M.N. Gungor), The Mining, Metallurgy and materials Society, Warrendale, Pennsylvania, 1990, 669-704.
- [13] - S.M. Jeng, P. Alassouer and J-M. Yang: Materials Science and Engineering A, 1991, 148, 67-77.
- [14]- B.P. Sanders and S. Mall: "Journal of Composites Technology and Research, JCTRER, 1996, 18, 15-21.
- [15] - L.N. McCartney, R. Morrell and J.D. Lord: NPL Report CMMT (A)56, 1996.
- [16] - Material Properties Handbook: Titanium Alloys (eds. R. Boyer, G. Welsch and E.W. Collings), ASM, Ohio, 1994.
- [17] - S.M. Bleay and V.D. Scott: Journal of Materials Science, 1992, 27, 825-838.
- [18] - E. Bischoff et al: Journal of The American Ceramic Society, 1989, 72, 5, 741-745.
- [19] - M.H. Lewis and V.S.R. Murthy: Composites Science and Technology, 1991, 42, 221-249.
- [20] - S.M. Bleay et al: Journal of Materials Science, 1992, 27, 2811-2822.
- [21] - M.W. Pharaoh, A.M. Daniel and M.H. Lewis: Journal of Materials Science Letters, 1993, 12, 998-1001.
- [22] - K.L. Luthra: Ceramic Engineering Science, 1987, 7-8, 649-653.
- [23] - K.P. Plucknett et al: Journal of Microscopy, 1995, 177, 251-263.

8 ACKNOWLEDGMENTS

The authors would like to acknowledge the assistance of AEA Technology plc, Harwell and DERA, Farnborough in supplying materials for this work, and also Dr. Martin Thomas of the latter organisation for his helpful comments.