

Characterisation of High Volume Fraction MMCs

Part I: Mechanical Properties

Summary

Many of the target applications for high volume fraction MMCs are aimed at utilising their excellent thermal performances in electronics packaging applications where the combination of low density, low CTE and high thermal conductivity are attractive properties. The mechanical properties are also important and cannot be ignored, because many of the components also have a structural function.

In this study conventional room temperature tensile, dynamic modulus and fracture toughness measurements have been made on representative materials, including two candidate substrate and packaging MMCs.

Results are presented together with comments and advice regarding the suitability of the above testing methods and recommendations for improving the quality of the data and measurements.

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1 INTRODUCTION

Metallic composites have been in development for a number of years and have now reached a reasonably mature stage. They remain of interest to a range of industrial sectors as they offer real performance benefits over traditional engineering materials, by providing lightweight design solutions to a wide range of components. To date most of the applications have been structural, such as brake, engine and transmission parts, lightweight panels, shrouds, struts, tubes and frames and general engineering components, utilising materials with relatively low (typically 15-25%) volume fractions of reinforcement. The latest generation of materials, which include novel multicomponent metallic laminates and high volume fraction metal composites (HVMC), containing up to 70% reinforcement are being developed more for their functional performance. An area of interest with considerable market value is in electronics and instrumentation where thermal performance and related properties are key factors. A recent review of the range of applications and measurement issues is presented in Ref 1.

The emphasis within this Measurement Note is on the mechanical properties and mechanical test methods, but the thermal and functional properties are crucial to many of the applications, particularly in the electronics industry. Some of the issues associated with measuring the thermal properties, such as the thermal diffusivity, thermal expansion, specific heat and thermal conductivity, of these candidate materials are covered in an accompanying Measurement Note [2].

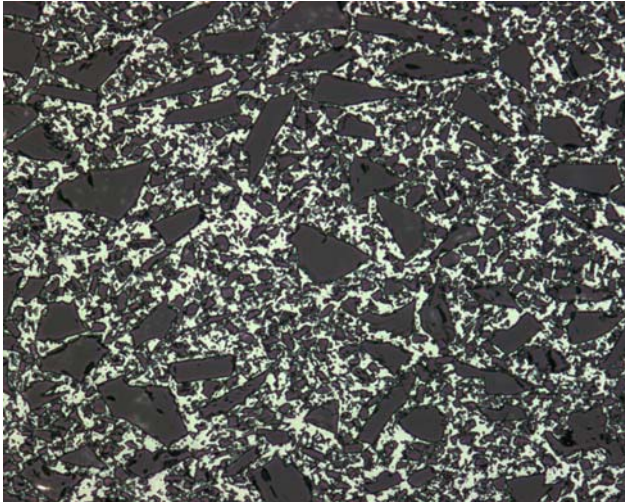
2 MATERIALS EXAMINED

Three commercially available materials have been examined in this study, with the volume fraction of reinforcement ranging from 40-70%. The materials are detailed in Table 1 below, and micrographs presented in Fig 1.

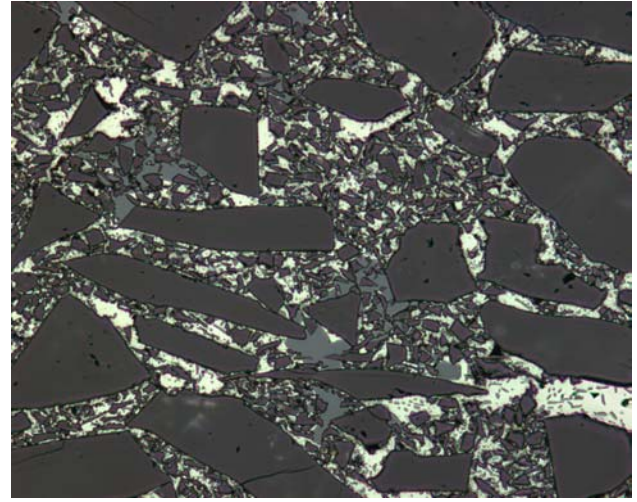
Material	Details
CPS AlSiC-9	63% SiCp / A356
Denka	70% SiCp / Al
AMC640xa	40% SiCp/Al 6061 T1

Table 1: Details of materials examined

Both the Denka and CPS materials were supplied as flat plates with a slight domed profile for improved thermal contact with cold components and heat sinks; the AMC material was in the form of plate approximately 12mm thick. It is clear from the micrographs that the three materials are very different. The Denka and CPS are cast materials that have been developed specifically for the electronics market and for their thermal performance; the AMC 640xa is produced by the powder route and is aimed at structural, mechanical and thermal applications, so the mechanical properties are probably of greatest importance in this case. The volume fraction of SiC reinforcement in the CPS and Denka MMCs are ~63 and 70% respectively, compared with 40% for the AMC640xa. The size of the distribution is also an important factor and varies considerably in the three composites – the AMC material contains a very fine (3µm) and uniform distribution of SiC particulate, whereas the CPS and Denka materials both contain bimodal particle size distributions, with the largest SiC particles approximately 50-100µm in size. (Note that the AMC micrograph is at x500 magnification, compared to the CPS and Denka materials, which are at x200).



CPS AlSiC-9 (x200)



Denka MMC (x 200)



AMC 640xa (x500)

Fig 1: Micrographs of the 3 materials examined in this study

3 TENSILE TEST RESULTS

Room temperature tensile tests were carried out on flat rectangular dog-bone specimens, 100mm long with a gauge length cross-section of 6mm x 3mm. Specimens were spark machined from plate, with the top and bottom faces ground flat and parallel. Tests were carried out in crosshead control in an Instron test machine, fitted with a universal joint and wedge grips to aid alignment. Testing was carried out in accordance with EN 10002-1 [3], the current standard tensile test method for metals. During the test the strain was measured using strain gauges bonded to both sides of the specimen to take into account any misalignment and bending. Specimens were initially only loaded to ~0.1% strain to measure the modulus over the early part of the curve. After unloading, the specimens were then tested to failure. Representative tensile stress-strain curves for the 3 materials are given in Fig 2 and 3 below.

EN10002-1 does not include specific information on modulus measurement (although ASTM E111 [4] does). To address this issue, through the work on MMCs and the recent EU TENSTAND project, NPL has pioneered and promoted improved strain measurement and analysis procedures to give accurate modulus data. Relevant information is given in Refs 5-8.

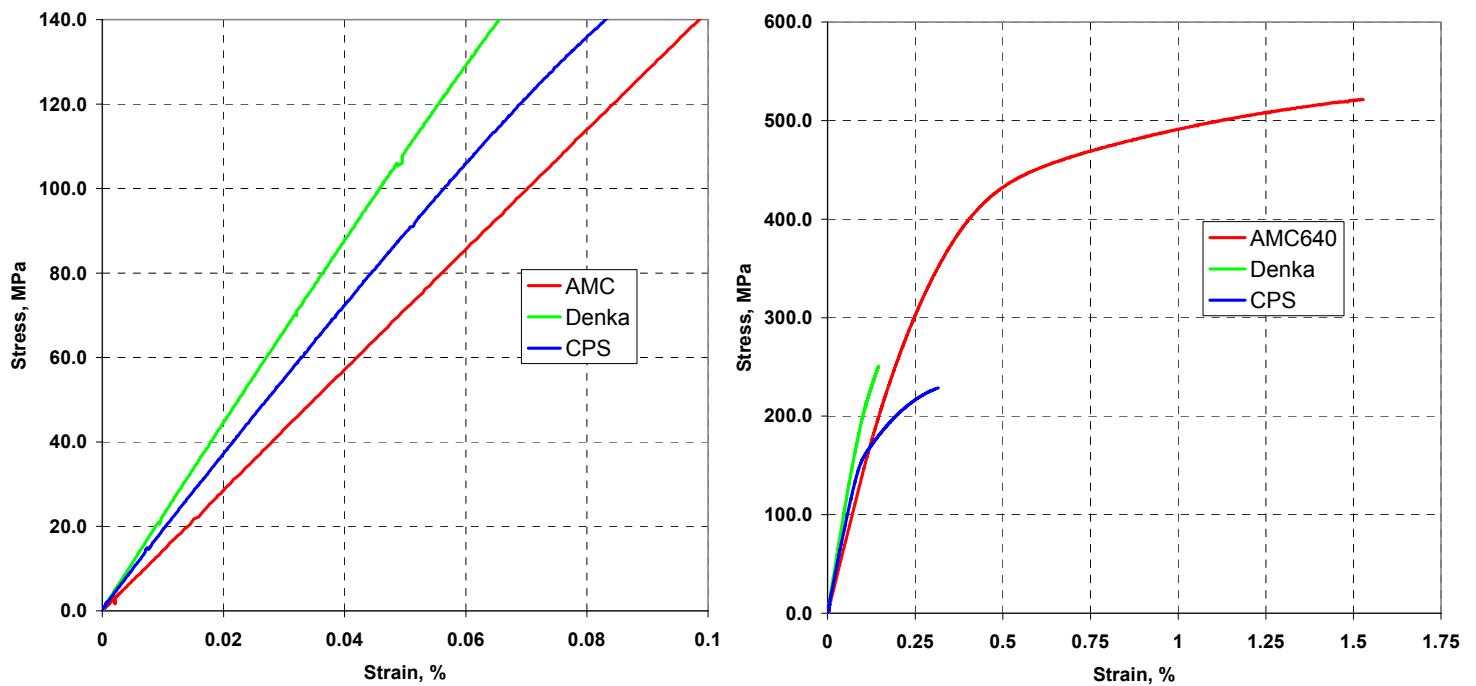


Fig 2: Typical stress-strain curves for the 3 materials, including the expanded early part showing the linear modulus region

The tensile results are summarised in Table 2 along with the fracture toughness test data. Mean room temperature modulus values measured from the tensile tests were 140.7 GPa, 169.9 GPa and 213.9 GPa for the AMC, CPS and Denka materials respectively. Due to the brittle tensile behaviour, it was not possible to measure a sensible proof stress value for any material other than the AMC, which had values for $R_{p0.2} = 440$ MPa, although two tests on the CPS material did give proof stress values.

A number of repeat measurements were carried out for each material and generally the Denka and CPS materials showed greater variability in the modulus, tensile strength and strain to failure. The highest volume fraction materials did not realise their theoretical strength and premature failure appeared to be initiated by the presence and failure of the large SiC particulates. As might be expected, the materials containing the highest levels of reinforcement had the highest modulus values, but failed at very low tensile strains (typically less than 0.3%). The AMC material showed greater ductility due to the reduced level of reinforcement and the smaller size of particulate used, and indeed the target application for the AMC material is more likely to be for high-value structural rather than functional applications.

Greatest difficulties were encountered with the tensile testing of the CPS and Denka materials due to the very limited plasticity. Results were in reasonable agreement with the manufacturer's data, but in 6 out of 10 tests on the higher volume fraction CPS and Denka materials, specimens failed in or close to the serrated jaw faces of the grips. This is an important practical issue with such brittle materials and consideration could be given to using other methods of gripping such as flat-faced hydraulic grips, pin loaded clevis-type arrangements, or special alignment fixtures as are commonly used in ceramics testing. An

alternative approach using bend testing could be carried out to measure the rupture strength but this is not directly comparable to the tensile strength for design purposes. Users should consider their applications and requirements carefully in planning the type of tests and data generated.

4 VARIATION OF MODULUS WITH TEMPERATURE

Due to the difficulties in obtaining reliable modulus data from the tensile tests carried out in accordance with EN 10002-1, many users turn to dynamic methods. Although in this study, the modulus values measured from the tensile tests were in good agreement with both the manufacturer's datasheets and the theoretical predictions from the Eshelby model [9], additional dynamic modulus measurements were carried out to confirm the static measurements and examine the variation of modulus with temperature, over a range representative of (but probably higher than) the temperatures encountered in target applications.

Measurements were made using the Impulse Excitation Technique (IET), on flat bar specimens approximately 100 mm x 10 mm x 2mm, machined from the plates. The IET method involves the excitation of a testpiece by a small mechanical impulse and subsequent analysis of the fundamental resonant frequency of flexural vibration via a microphone or piezo-electric transducer. In this study the testpieces were tested in flexural mode, supported at the nodes of resonance in a furnace and struck by small ruby balls dropped down a guide tube to hit the centre of the testpiece.

The equation used to calculate modulus [10] is:

$$E = 0.9465 \frac{m}{w} f^2 \left(\frac{L}{t} \right)^3 T_1$$

where m is the mass of the testpiece, w , L and t are the dimensions, f is the fundamental resonant frequency for flexural vibration and T_1 is a shape factor, which for a rectangular bar is:

$$T_1 = 1 + 6.585 \left(\frac{t}{L} \right)^2$$

Further details and background of the IET method are given in Refs 10-13. The mass and dimensions of each testpiece were measured prior to testing, and measurements of the resonant frequency were taken at intervals of 25°C during the heating cycle, with the specimen held at temperature for 10 minutes prior to taking the readings to ensure temperature uniformity. Five measurements were made at each temperature interval, and the mean frequency values used as to calculate the value for elastic modulus. The theoretical errors in measurement of modulus by dynamic methods are small, of the order of $\pm 1\%$. Results are presented in Figure 4, plotted against the test temperature. Data generated previously on a range of aluminium alloys (green) and other MMC systems (red) [14] are also included for comparison.

The room temperature modulus values measured from the dynamic method were 141.5 GPa, 170.1 GPa and 217.3 GPa for the AMC, CPS and Denka materials respectively and these compared very favourably with the corresponding mean values measured from the NPL tensile tests detailed previously.

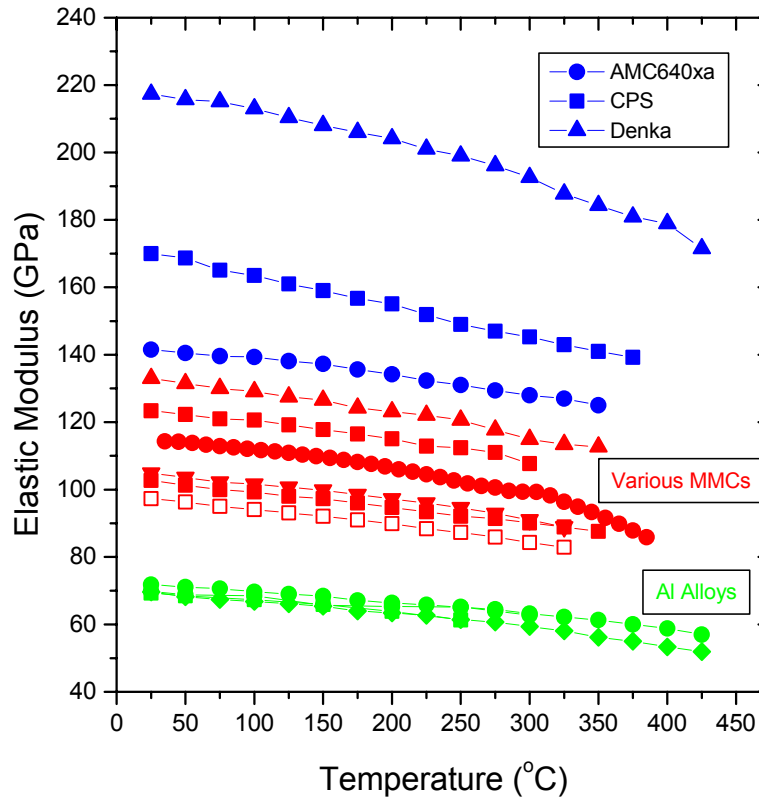


Fig 4: Variation of modulus with temperature

Results show that the variation of modulus with temperature is reasonably linear, but the slope begins to fall more rapidly at higher temperatures. This drop seems more pronounced in the Denka material and could be a function of the large particle size, matrix chemistry, heat treatment and composite stability. Tests carried out in the NPL ETMT miniature thermo-mechanical test system to examine the dimensional stability of the materials [14] indicated that the Denka and CPS materials were more susceptible than the AMC 640xa to creep and changes caused by thermal cycling.

A limitation of the IET and other dynamic methods is internal damping, which causes a loss of signal as the temperature increases. Internal damping occurs in all materials including metals and ceramics, but it is difficult to predict exactly when this will occur. There are several mechanisms that can contribute to the damping mechanism, including the movement of dislocations, viscous sliding between grains, degradation of interfaces, point defects and thermo-elastic effects. In the present study for all the materials examined, no measurements could be made above about 425°C. During the tests the approach of the temperature limit could be observed on the frequency analyser display as a broadening in the peak of the fundamental frequency accompanied by a sharp reduction in amplitude.

5 FRACTURE TOUGHNESS RESULTS

The plane strain fracture toughness, (K_{IC}) is an important parameter in designing against material failure. It characterises the resistance of a material to fracture in the presence of a sharp crack under tensile loading, where the state of stress near the crack front is triaxial plane strain, and the crack-tip plastic region is small compared with the crack size and specimen dimensions. A valid K_{IC} value is believed to represent a lower limiting value of fracture toughness, and is a key parameter in design for estimating the relationship between failure stress and defect size for a material in service under similar stress state conditions.

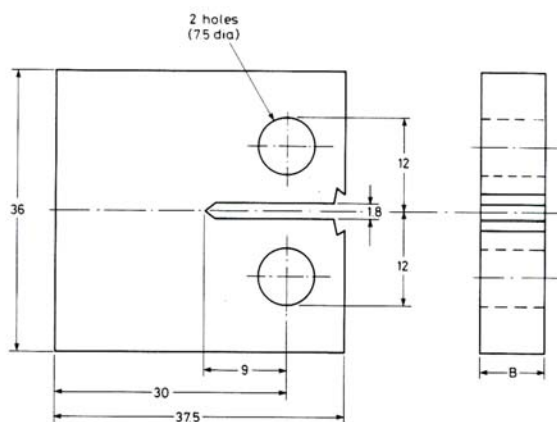
At present there are no standard fracture toughness test procedures specifically for MMCs and the conventional standards for metals ASTM E399 [15] and BS 7448 [16] are normally used. Previous studies have highlighted the difficulties of obtaining valid K_{IC} data for these type of materials, largely because of excessive crack curvature, non-linearity of the load-displacement trace, or out of plane crack propagation [17].

The plane strain K_{IC} fracture toughness test involves the loading to failure of pre-cracked notched specimens in tension or three-point bending. The test is unusual in that the calculation of a valid toughness value can only be determined after the test has been completed, via examination of the load-displacement plot and measurement of the fatigue pre-crack crack length. The provisional fracture toughness K_Q is first calculated from the following equation:

$$K_Q = (P_Q/BW^{1/2}).f(a/W)$$

.... where P_Q is the load corresponding to a defined increment of crack length, B is the specimen thickness, W the width, and the function $f(a/W)$ is a geometry dependent factor that relates the compliance of the specimen to the ratio of crack length and width. Only when specific validity criteria are satisfied, can the provisional fracture toughness K_Q be quoted as a valid plane strain fracture toughness K_{IC} .

The fracture toughness results made in this study were generated from tests on CT specimens, with width $W = 30$ mm and height $N = 36$ mm, and the nominal thickness of the plate material $B = 5$ mm (see Fig 5).



For direct comparison, the AMC specimens were machined to the same dimensions despite larger thicknesses of material being available. Integral knife-edges were machined into each specimen, 7.5 mm from the loading line, to locate the arms of the COD gauge. Results are summarised in Table 2, along with the tensile data and plotted in Fig 6.

Fig 5: Details of CT fracture toughness specimen

Material	Tensile Results					Fracture Toughness Results			
	Test No.	E (GPa)	R _{p0.2} (MPa)	R _m (MPa)	A (%)	Test No.	K _Q (MPa√m)	K _{IC} (MPa√m)	Reason *
AMC	1	140.1	444	512	1.52	1		17.4	
	2	140.6	452	525	1.33	2	16.0		2
	3	141.2	442	514	1.46	3		15.7	
	4	140.9	445	514	1.41	4		16.2	
	5	140.6	448	518	1.70	5	17.6		2, 3
	Mean	140.7	446.2	516.6	1.5	Mean	16.8	16.4	-
CPS	1	171.0	235	240	0.38	1	12.3		2
	2	170.5	-	220	0.22	2	10.8		3
	3	168.2	-	180	0.18	3		10.2	
	4	170.3	-	215	0.22	4	11.1		2
	5	169.5	218	225	0.32	5	9.4		2
	Mean	169.9	226.5	216	0.26	Mean	10.9	-	-
Denka	1	217.6	-	261	0.15	1	11.6		1, 2
	2	210.2	-	252	0.18	2	10.3		1
	3	209.6	-	276	0.20	3	10.1		1, 3
	4	215.4	-	221	0.17	4	8.8		1, 2
	5	216.7	-	258	0.22	5	9.2		1
	Mean	213.9	-	253.6	0.18	Mean	10.0	-	-

*Key: 1 – No Rp0.2, so thickness criteria not satisfied
2 – Asymmetric crack front
3 – a/W out of range

Table 2: Summary of tensile and fracture toughness data

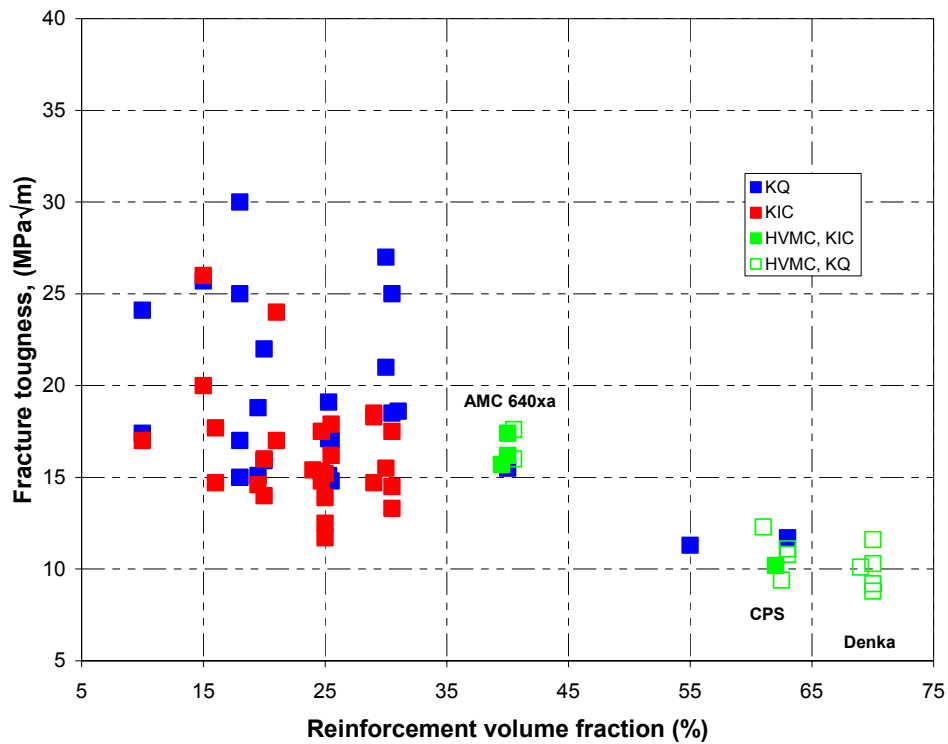


Fig 6: MMC fracture toughness data plotted against volume fraction of reinforcement. Literature data [21] and measurements made on materials in this study.

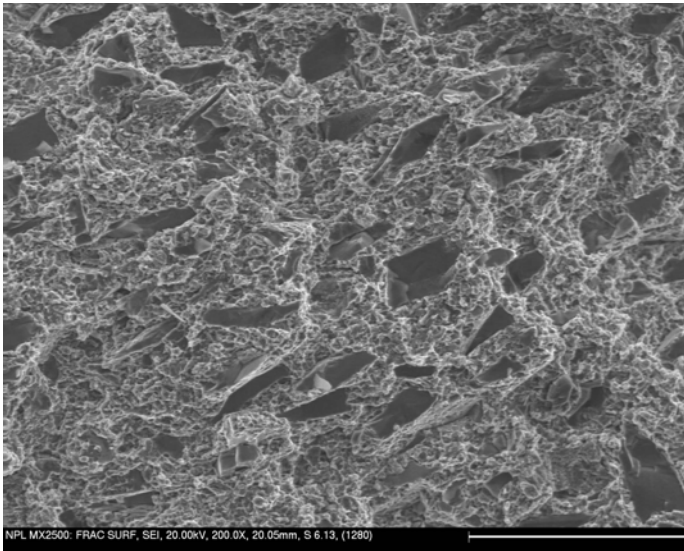
The figure also shows the data for a variety of metal composites, plotted against volume fraction of reinforcement [18]. For the most commonly used structural materials with 15-30 volume percent reinforcement there is a large degree of overlap because different combinations of matrix material, reinforcement and heat treatment conditions are included - all of which have an important effect on the fracture behaviour.

Five tests were carried out for each material. Of these none of the tests on the Denka material were valid, and only one and three tests gave valid K_{IC} results for the CPS and AMC material respectively. Typically repeat measurements varied by as much as $3 \text{ MPa}\sqrt{\text{m}}$, which represents a significant scatter ($\sim 30\%$) for the high volume fraction materials. K_{IC} values for the AMC 640xa ranged from $16.0 - 17.6 \text{ MPa}\sqrt{\text{m}}$.

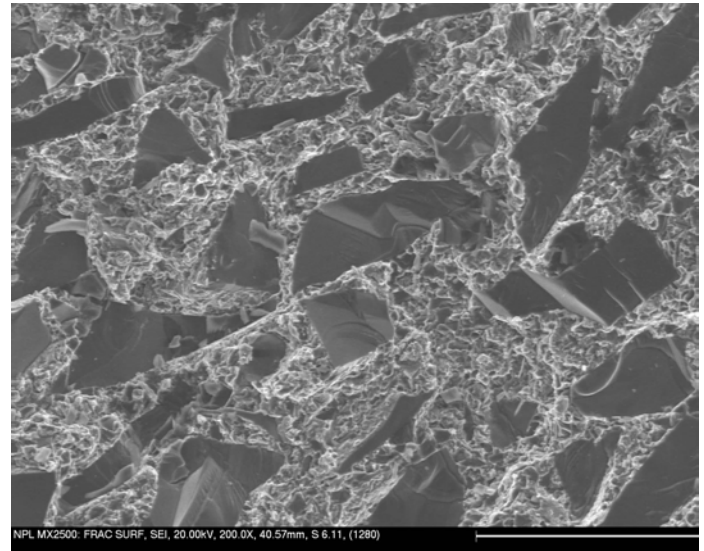
For the Denka material, it was not possible to measure a value for $R_{p0.2}$ from the tensile tests so the thickness validity criterion could not be applied and all the results are quoted as provisional K_Q values. There were also problems with controlling precracking, and despite using decreasing ΔK conditions, several tests failed during the precracking stage. The excessive crack curvature previously seen in tests on thick MMC specimens containing 25% volume fraction of reinforcement was not a problem, but there was evidence of non-linearity of the load-displacement trace, which might be an indication of crack closure during precracking. This was more noticeable in the tests on the CPS and Denka materials, and examination of the path of the surface cracks on a polished specimen indicated that the fatigue precrack generally travelled in the matrix, around the particulate and this probably supports the increased levels of crack closure in the materials with largest particles and rougher fatigue surface.

Due to the thin specimen geometry no test suffered from excessive crack curvature, but some were invalid due to the asymmetry of the pre-crack. Better success with the SENB geometry or consideration should be given to using other fracture toughness test methods such as those developed for monolithic ceramics [19,20] or the Palmquist test procedure, which is used for hard metals [21]

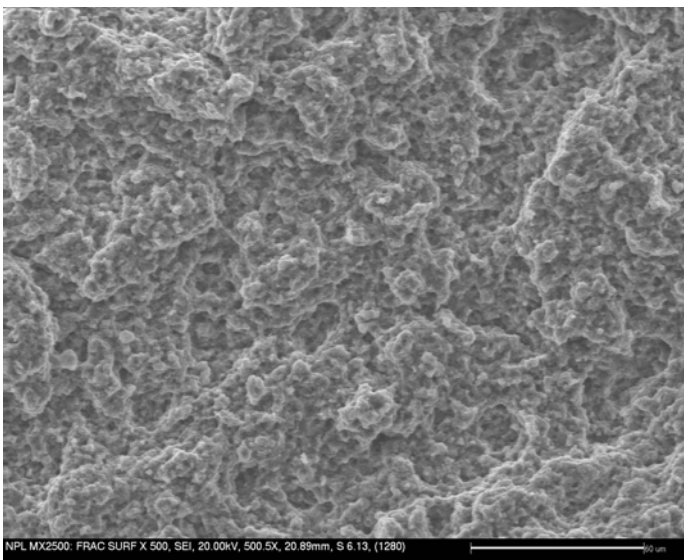
Fig 7 shows the typical features of the fracture surfaces from the fracture toughness tests. For comparison, examples are presented at the same magnification as the optical micrographs in Fig 1. On a macroscopic scale the fracture surfaces of were very flat, and had a similar appearance to the tensile test specimens. For the AMC material the fracture appears to have occurred predominantly through the matrix and very few broken particles can be seen on the fracture surface. For the CPS and Denka materials, there is clear evidence of particle fracture and cleavage. In some cases the reinforcement has evidence of matrix material adhering to the surface, indicating that failure has occurred at the interface, but the predominant failure mechanism is by particle fracture, with limited matrix ductility. These observations are in agreement with studies carried out previously [17,18].



CPS AlSiC-9 (x200)



Denka MMC (x200)



AMC 640xa (x 200)

Fig 7: Typical fracture surface appearance of materials examined

SUMMARY

For the AMC material with 40% SiC reinforcement the results generated by the conventional test methods for metals were generally satisfactory. The major difficulties were encountered with the higher volume fraction CPS and Denka materials, particularly with the fracture toughness testing and tensile testing due to difficulties with precracking and premature failures in the tensile test close to the grips. The highest volume fraction materials did not realise the theoretical strength, because of premature failure probably initiated by the failure of the large SiC particulates.

For the Denka and CPS materials with very high volume fraction of reinforcement more reliable mechanical property data is required and other test procedures – such as those developed for hard metals and ceramic materials could be considered.

ACKNOWLEDGEMENT

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REFERENCES

- [1] NPL Report MATC(A)128. *High Volume Fraction MMCs and Multicomponent Metallics - A Review of Applications and Measurement Issues*. JD Lord and P Rendle, May 2003.
- [2] NPL Measurement Note DEPC-MN 006. *Characterisation of High Volume Fraction MMCs, Part II – Thermal Properties*, J D Lord, Sept 2004
- [3] EN 10002-1: *Metallic Materials - Tensile Testing – Part 1: Method of Test at Ambient temperature*
- [4] ASTM E111: *Standard Test Method for Young's Modulus, Tangent Modulus and Chord Modulus*
- [5] NPL Measurement Note CMMT(MN)16. *Recommended Methods for Determining Young's Modulus from Tensile Tests*. J D Lord and B Roebuck. Aug 1997.
- [6] NPL Measurement Note MATC(MN)41. *Review of Methods and Analysis Software for the Determination of Modulus from Tensile Tests*. J D Lord. Dec 2002.
- [7] *Data Acquisition and Analysis of Tensile Properties for Metal Matrix Composites*, B. Roebuck, JD Lord, PM Cooper and LN McCartney. ASTM Workshop on Accuracy of Load and Strain Measurements, Miami, November 18, 1992. ASTM J. Testing and Evaluation, JTEVA, 22(1), 1994, 63-69.
- [8] TENSTAND WP3 Final Report - *Modulus Measurement Methods*. J Lord, M rides and M S Loveday, Sept 2004
- [9] *An Introduction to Metal Matrix Composites; Chapter 3: The Eshelby approach to modelling composites*. TW Clyne and PJ Withers, Cambridge University Press, 1993. ISBN 0 521 41808 9
- [10] ASTM E1876: *Standard Test Method for Dynamic Young's Modulus, Shear Modulus and Poisson's Ratio by Impulse Excitation of Vibration*
- [11] NPL Measurement Note MATC(MN)49. *Elevated Temperature Modulus Measurements using the Impulse Excitation Technique (IET)*. J D Lord and L P Orkney. July 2000.
- [12] *Dynamic Young's Modulus Measurements in Metallic Materials: Results of an Interlaboratory Testing Program*. A Wolfenden et al. Journal of Testing and Evaluation, Jan 1989, pp2-13

- [13] *Impulse Excitation Apparatus to Measure Resonant Frequencies, Elastic Moduli, and Internal Friction At Room and High Temperature.* G Roebben, B Bollen, A Brebels, J Van Humbeeck, O Van Biest. Rev. Sci. Instrum. 68 (12) Dec 1997, pp. 4511-15
- [14] NPL Measurement Note MATC(MN)55. *Rapid Prototype Testing of Multicomponent Metallics.* B Roebuck, April 2004.
- [15] ASTM E399: *Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials*
- [16] BS7448 Pt1:1991: *Fracture Mechanics Toughness Tests, Part 1. Method for determination of K_{IC} , critical CTOD and critical J values of metallic materials*
- [17] *Plane Strain Fracture Toughness Test Procedures for Particulate Metal Matrix Composites.* B Roebuck and J D Lord, Mat Sci & Tech, Dec 1990, Vol 6 pp1199-1209.
- [18] MMC ASSESS Technical report: *Fracture toughness test methods for PRM*, J D Lord, June 2004
- [19] NPL Measurement Note CMMT(MN)035. *Pre-cracking Testpieces of Brittle Materials for Fracture Toughness Measurement.* R Morrell, Jan 1999
- [20] Fracture Toughness Testing for Advanced Technical Ceramics – Internationally Agreed Good Practice, R Morrell, presented at the Materials Congress 2004, British Ceramic Transactions (in press)
- [21] NPL Good Practice Guide No. 9. *Measurement of Palmquist Toughness for Hard and Brittle Materials*, B Roebuck, E G Bennett, L A Lay, R Morrell, June 1998.

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