Characterisation of High Volume Fraction MMCs

Part II: Thermal Performance

Summary

Accurate measurements of the thermal properties of high volume fraction MMCs are important as many of the target applications experience thermal loading during service and are seeking to exploit their unique combination of thermal properties.

Thermal diffusivity, specific heat, thermal expansion and thermal conductivity characteristics of candidate materials have been measured using a variety of techniques, including laser flash, DSC, conventional dilatometry, strain gauge methods and non-contact image correlation.

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 are of interest to a range of industrial sectors as they offer lightweight solutions and significant performance benefits over traditional engineering materials. 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, but the latest generation of materials, which include high volume fraction metal composites and novel multicomponent metallic laminates are being developed more for their thermal and functional performance.

One of the main driving forces in the electronics industry is the desire to downsize components and many of today's electronic products demand lightweight, low cost materials that provide increased functionality in a smaller volume. As the power handling requirements, operating temperatures and packing densities continue to increase the task of improving the thermal management capability of these devices poses a significant challenge for materials and electronic engineers.

Electronic packaging is a rapidly developing market for high volume fraction MMCs, based on the ability to develop materials with coefficients of thermal expansion (CTE) comparable to electronic materials and ceramic substrates. Most of the currently available materials are based on aluminium alloys that are lightweight and have good thermal conductivity. A range of reinforcement types is used but mostly it is either SiC or Al_2O_3, up to volume fractions of 70%. The materials are being used in place of Cu/W (copper tungsten) and Kovar in an increasing number of thermal management applications, and whilst the thermal conductivity is similar to that of Cu/W, the density is approximately one half, which makes Al-SiC a good choice for weight-sensitive applications. Target applications include electronic substrates, packaging, instrument racks, IGBT base plates, heat spreaders and heat sinks and electro-optical housings. Some of the key applications for such high volume fraction MMCs are presented in Ref 1, and a summary of relevant commercially available materials is given in Table 1 [1]. It is interesting to note that relevant thermal property data - particularly the thermal conductivity - is not readily available for many of the materials included.

For many applications the material properties such as thermal diffusivity, thermal conductivity and heat capacity are important inputs to finite element and analytical models, but it is interesting to note that the full data is not always presented on the manufacturer's datasheets, and potential users are recommended to carry out their own assessments of material properties, particularly in critical design applications. A short summary of thermophysical properties and test methods for MMC is presented in Ref 2.

Work carried out as part of this study has focused on measuring the thermal properties of three candidate materials. As noted above, CTE mismatch is a key driver for component failure, particularly where thermal cycling exists, as is the case in most electronic systems. Heat loss and cooling are also important factors, so the heat capacity and thermal conductivity are key parameters also. The devices must also remain stable over the temperature cycle, so dimensional stability and the evaluation of damage development and degradation of the interface properties under operating conditions is also important. In the following sections results are presented describing the test methods used for measuring thermal diffusivity, specific heat, thermal conductivity and expansion. Comments and advice

are given regarding the suitability of the methods, together with recommendations for improving the quality of the data and measurements.

Although the emphasis within this study is on thermal performance, in many applications the mechanical properties are still important. Difficulties have been experienced in obtaining reliable mechanical property data, particularly with the materials containing very high levels of reinforcement, and some of the issues dealing with the measurement of tensile properties, modulus, fracture toughness and ductility are covered in an accompanying Measurement Note on mechanical properties [3].

Supplier	MMC type	Volume fraction (%)	CTE ($x10^{-6}K^{-1}$)	Density (gcm^{-3})	k ($Wm^{-1}K^{-1}$)
Advanced Materials Lanxide	Al + Al2O3	25 - 38	14.5 - 18		
Aerospace Metal Composites Ltd	Al + SiC	25 - 40	13.4 - 15.5	2.8 - 2.9	130
Aerospace Metal Composites Ltd	Fe + TiB2	15 - 30		6.8 - 7.4	
Alloy Technology Intern Inc.	Steel + TiC	25 - 45	5.1 -7.3	6.5 - 7.0	
Alyn Corperation	Al + B4C	5 - 35	8.1 - 11.4	2.7	
Alyn Corperation	Al + SiC	15 - 35	7.9 - 10.5	2.8 - 2.9	
Ametek Specially Metal Products	Al + SiC	68	6.8 - 7.5	3	
Brush Wellman Inc	Be + BeO	20 - 60	6.1 - 8.7	2.0 - 2.5	
Ceramics Process Systems	Al + SiC	37 - 70	6.8 - 11.5	2.9 - 3.0	170 - 180
Cercast	Al + SiC	65	8.1		
Chesapeake Composites Corp	Al + Al2O3	30 - 40	15.7 - 16.5	3.0 - 3.2	
Creuzet Aeronautique	Al + SiC	17 - 25		2.6 - 2.9	
dmc2 Electronic Components Corp	Al + SiC		6.4 - 7.3	2.9 - 3.0	
Duralcan USA	Al + Al2O3	10 - 20	11 - 16	2.8 - 2.9	
Duralcan USA	Al + SiC	10 - 20	16.6 - 21.4	2.7 - 2.8	
DWA Aluminum Composites	Al + SiC	15.5 - 50			
Electrovac	Al + SiC	70	6.8	3	180 - 200
Goodfellow Corporation	Al + SiC	13 - 17		2.6 - 2.9	
Metal Matrix Cast Composites	Al + Al2O3	55	11.8 - 13.2	3.0 - 3.5	
Metal Matrix Cast Composites	Al + graphite		4 – 7.5	2.4 – 2.5	200 - 230
Millenium Materials	Al + SiB6	10 - 70	7 - 17		65 - 194
Osprey Metals (Si-Al alloys)	Si + Al	27 - 70	7 - 17	2.4 - 2.7	120 -180
PCC Advanced Forming Technology	Al + SiC	68 - 69	6.9 - 7.4	3.0	
Polese Company	Al + SiC		13	2.79	

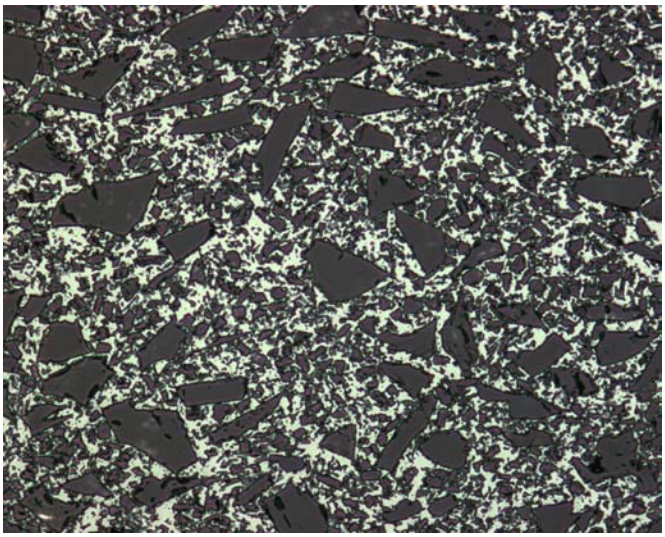
Table 1: Thermal properties of relevant MMCs [1]

2 MATERIALS EXAMINED

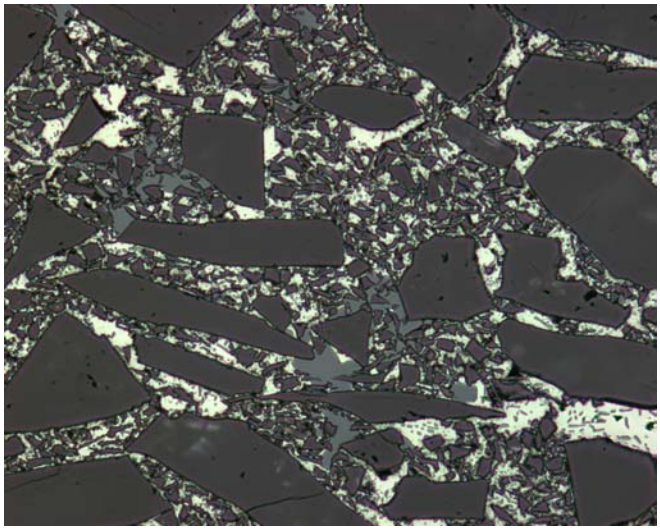
Three commercially available materials have been examined in this study, with the volume fraction of reinforcement ranging from 40-70%. The materials are detailed in Table 2 and micrographs presented in Fig 1 below.

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

Table 2: Details of materials examined



CPS AlSiC-9 (x200)



Denka (x200)

AMC 640xa (x 500)

Fig 1: Micrographs of the 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).

3 HEAT TRANSFER AND THERMAL PROPERTIES

The most important heat transfer mechanism in metals is conduction, which involves the transfer of energy within the material by a combination of electrons and lattice waves or phonons. The rate of heat transfer depends upon the temperature gradient and the thermal conductivity of the material. The thermal conductivity, λ, is a property of a material that expresses the heat flux that will flow through if a temperature gradient exists through the material. Because the transport mechanisms involved are similar there is a good correlation between the thermal conductivity and electrical conductivity of a metal. The direct measurement of thermal conductivity is difficult because it requires the establishment of a well-defined, constant temperature gradient that is difficult to achieve practically. It is common therefore to use non-steady state methods, such as the laser flash technique, to measure thermal diffusivity, which is a measure of the rate at which a material reaches equilibrium with it surroundings. The thermal conductivity, λ, can then be calculated from the relationship:

$$\lambda = a.\rho.C_p$$

… where a is the thermal diffusivity, ρ the density and C_p the specific heat capacity.

Typical values of thermal conductivity for aluminium alloys are of the order of 170-240 $Wm^{-1}K^{-1}$, whilst those from the literature for SiC vary from ~4-20 $Wm^{-1}K^{-1}$ for polycrystalline SiC to ~100 $Wm^{-1}K^{-1}$ for single crystal SiC [4], with similar values for Al_2O_3. Most MMCs use relatively low-grade SiC particulate so the lower figure for the polycrystalline material is likely to be relevant, although some datasheets do specify the use of "aerospace grade" SiC.

The introduction of a ceramic reinforcement with a lower thermal conductivity relative to the metal matrix would therefore be expected to have a significant effect on reducing the overall composite thermal performance, but thermal conductivity is not the only parameter of interest. Thermal transport in Al-MMCs is a complex behaviour depending on material composition, microstructure and heat treatment condition. For many applications some compromise on thermal performance must be made. Many of the materials have been developed to give a particular thermal expansion behaviour, and indeed the CPS AlSiC-9 material examined as part of this study has been designed to give an average CTE of 9 x 10^{-6} $^oC^{-1}$ (see measurements later). For electronic applications, when choosing potential composite materials, the key design criteria are achieving a combination of matched CTE, reduced weight and good thermal conductivity. It is a challenge that the materials manufacturers are meeting through appropriate selection of reinforcement type and size, interface chemistry, different matrix alloys, processing conditions and heat treatments. In many cases however, the data is not always quoted in the manufacturers material datasheet, and often only a single value, calculated at a specific temperature, is quoted. Care must be taken if accurate modelling and design calculations are employed for a range of temperatures.

In this study the thermal diffusivity was measured by the Laser Flash technique; separate specific heat and density measurements were made respectively using Differential Scanning Calorimetry (DSC) and the Archimedes principle and the thermal conductivity calculated from these.

4 THERMAL DIFFUSIVITY MEASUREMENTS USING THE LASER FLASH TECHNIQUE

The thermal diffusivity of the three materials was measured using a Netzsch LFA 427 laser flash equipment, according to the standard NPL procedure [5]. In the laser flash thermal diffusivity measurement, one side of a relatively thin, parallel-sided disc is subjected to a short pulse of laser energy and the subsequent temperature response on the opposite side of the sample is measured, usually with an infrared sensor or pyrometer. A schematic of the apparatus is given in Fig 2 below, and with the typical output shown in Fig 3.

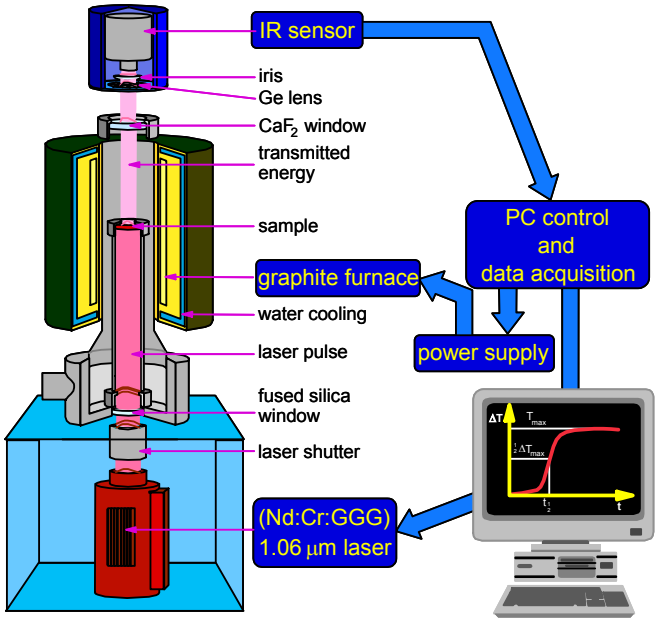


Fig 2: Schematic of Laser Flash Apparatus

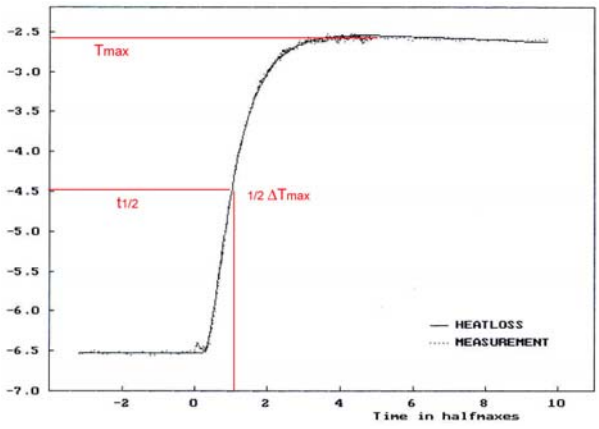


Fig 3: Typical temperature response from the laser flash experiment

The thermal diffusivity is calculated from the value of the half rise time ($t_{1/2}$), which is determined by measuring the point at which the sample reaches half of the maximum temperature rise (½ ΔT_{max}). The part of the curve beyond this point includes the cooling of the sample and, in the method of calculation used at NPL, correction for the heat loss has been made according to the method developed by Dursza [6]. It should be noted that the measured changes in temperature are small, typically ~ 1-2 ºC. The plot above shows a fit to the experimental data, based on theoretical curves, and this dramatically reduces the problems due to noise, drift and spikes, and greatly improves the accuracy and reliability of the results.

For the initial measurements on the materials in this study, a standard specimen with a thickness of 1.5 mm was used. The samples were heated to temperature in a graphite furnace under an argon atmosphere and held at temperature for at least 10 minutes prior to making the measurements. Measurements were made in 100 ^{o}C steps throughout the heating and cooling cycle, up to a maximum temperature of ~500 ^{o}C. Fig 4 shows the results from one of the

initial tests on the CPS material, where the maximum temperature is difficult to define precisely, leading to unacceptable levels of scatter.

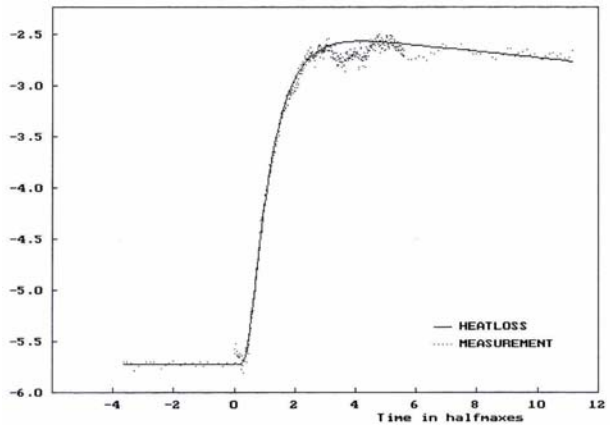


Fig 4: Example of poor data caused by thin specimen

This particular response is typical of a test on a thin specimen with high thermal conductivity, where there is insufficient separation of the laser pulse width and the half rise time, leading to considerable uncertainty in the measured values. In practice the optimum conditions give a half rise time that is at least 20x larger than the pulse width, but with the standard specimen thickness used in the initial study, the conditions resulted in a separation of only 5x.

Experiments were repeated using 3mm thick samples, to give the optimum test conditions and the required difference in the size of the laser pulse width and the half rise time. Summary results from the tests carried out under these conditions are plotted in Fig 5 and summarised in given in Table 4.

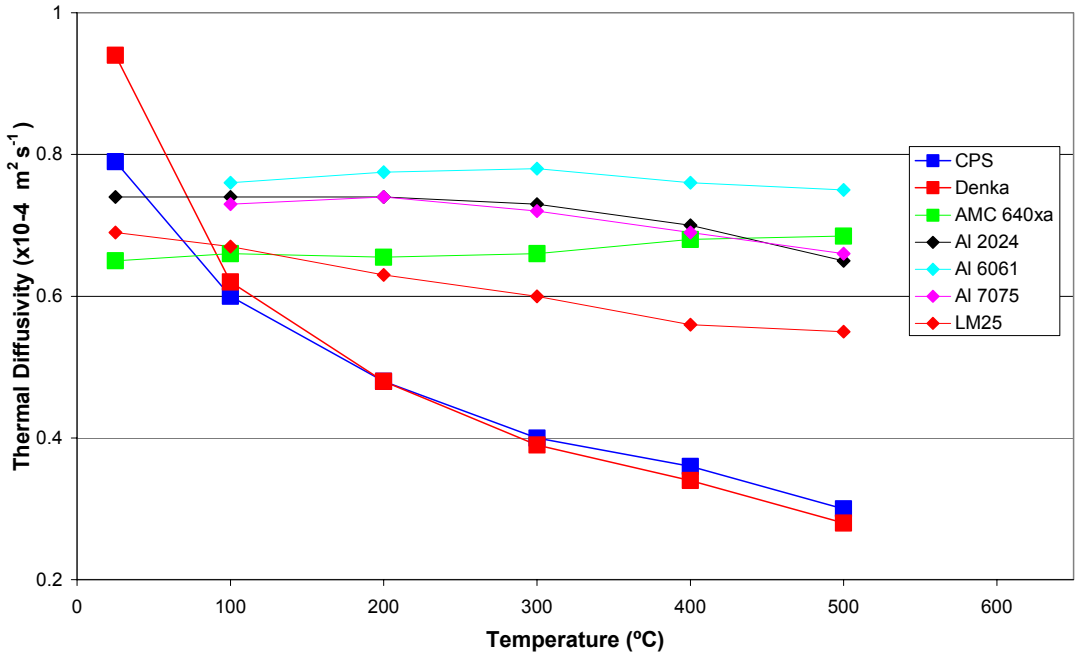


Fig 5: Laser flash thermal diffusivity data for the 3 materials examined

Fig 5 also includes representative handbook data [7] for a range of aluminium alloys for comparison, showing considerable differences in the behaviour of the wrought and cast alloys. Results calculated using the laser flash technique are estimated to have an uncertainty of ~5%.

The values of diffusivity for the CPS and Denka materials are very different than the AMC 640xa and this is a reflection of the considerable differences in composition, microstructure, metallurgical condition and heat treatment. Room temperature values for the Denka MMC are highest, but drop sharply on heating, and above ~ 100 ºC the response of the CPS and Denka materials are very similar. This behaviour is similar to that observed elsewhere [8-11] on HVMC. The AMC 640xa material shows a similar response to the unreinforced wrought aluminium alloys, albeit with lower values, and the diffusivity remains relatively constant over the temperature range examined. The thermal diffusivity has been seen to increase with ageing [9], and this could be the case with the AMC640xa, which was tested in the T1 (as-received) condition, although the results represent the average of 3 heating cycles and no significant difference was observed between the repeat measurements in this case.

The laser diffusivity data is not readily used by engineers and designers, who are used to dealing with thermal conductivity. Additional measurements of specific heat and density were made over the same temperature range to enable thermal conductivity response of these materials to be calculated. Details are presented in the following sections.

5 SPECIFIC HEAT MEASUREMENTS

The specific heat capacity is the the quantity of heat energy needed to raise the temperature of a unit quantity of material by 1 ºC. Measurements in this study have been made on a Perkin Elmer DSC7 differential scanning calorimeter, using small flat discs, approximately 5mm in diameter and 1mm thick machined from each material. For the measurement two small aluminium pans were heated in a cylindrical furnace in a nitrogen atmosphere. The power given to each of the pans was monitored over the temperature range, and recorded while the furnace temperature rose at 10 ºC min^{-1}.

Three separate temperature scans were made with one pan empty and the other respectively: empty (baseline scan), containing sapphire, a heat capacity reference material (reference scan), and containing the sample (measurement scan). The required specific heat capacity was obtained by comparing the measurement and reference scans, using the baseline scan to correct for departures of the instrumental baseline from true zero. Data is plotted in Fig 6 and summarised in Table 4. Again, for comparison representative handbook data [7] for a range of aluminium alloys is included.

For all the materials examined the specific heat capacity was found to increase as a function of temperature. Up to ~200-300^{o}C, the heat capacity of the MMC materials is a little lower than that of the unreinforced aluminium alloys, but generally there is little difference in behaviour of all the materials examined, and the standard test method did not cause any problems or issues for these materials.

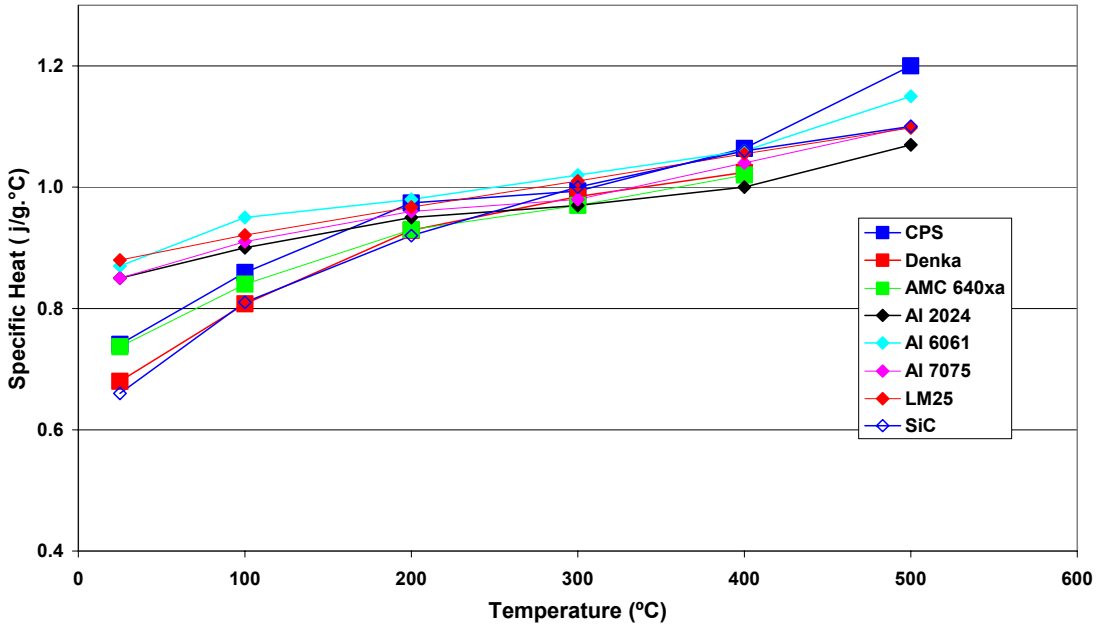


Fig 6: Specific Heat (DSC) measurements

6 THERMAL EXPANSION MEASUREMENTS

Thermal expansion mismatch is probably the most critical factor in many of the applications for high volume fraction MMCs in the electronics sector, and is a key driver for component failure, particularly where thermal cycling exists. As the power handling requirements, operating temperatures and packing densities of electronic systems increase, the task of reducing thermal mismatch strains and improving the thermal management capability of these devices poses an increasingly important challenge. It is crucial that the CTE is accurately determined, and any CTE mismatch reduced, because electronic packages consist of various conducting and insulating materials, all of which have different values of CTE.

In this study measurements of thermal expansion have been made using a variety of techniques, including conventional dilatometry, strain gauges and image correlation methods. A summary of the results is presented below but further information on the various methods are described in greater detail in NPL Measurement Note MATC(MN)45 [12]. Dilatometry is the most well-established technique for measuring CTE, but making measurements with the dilatometer is still a delicate, demanding task**.** The test uses a testpiece in the form of a simple round or flat bar, which is held against a stop in a support tube, with its free end contacted by a push rod. In this study, specimens machined from each material approximately 40 mm long x 5mm x 5mm, were heated at 3 ^{o}C min^{-1} between room temperature and ~200 ^{o}C. Liquid nitrogen cooling extended the lower range to -10 ^{o}C, following which a further 3 repeat cycles were made. Changes in length were transmitted by a push rod in contact with the specimen to a calibrated LVDT, and tests carried out according to a validated NPL procedure [13].

Representative data from the three materials is presented in Fig 7 below. Typically the measurements have a reproducibility of about ± 0.2 x 10^{-6} ^{o}C^{-1} measured over a range of 100

^{o}C, with an absolute accuracy of about ± 0.2 x 10^{-6} $^{o}C^{-1}$. Over larger temperature ranges the absolute accuracy can be better than this, but the range 100-200 ^{o}C is typical of that encountered in the electronics applications being considered.

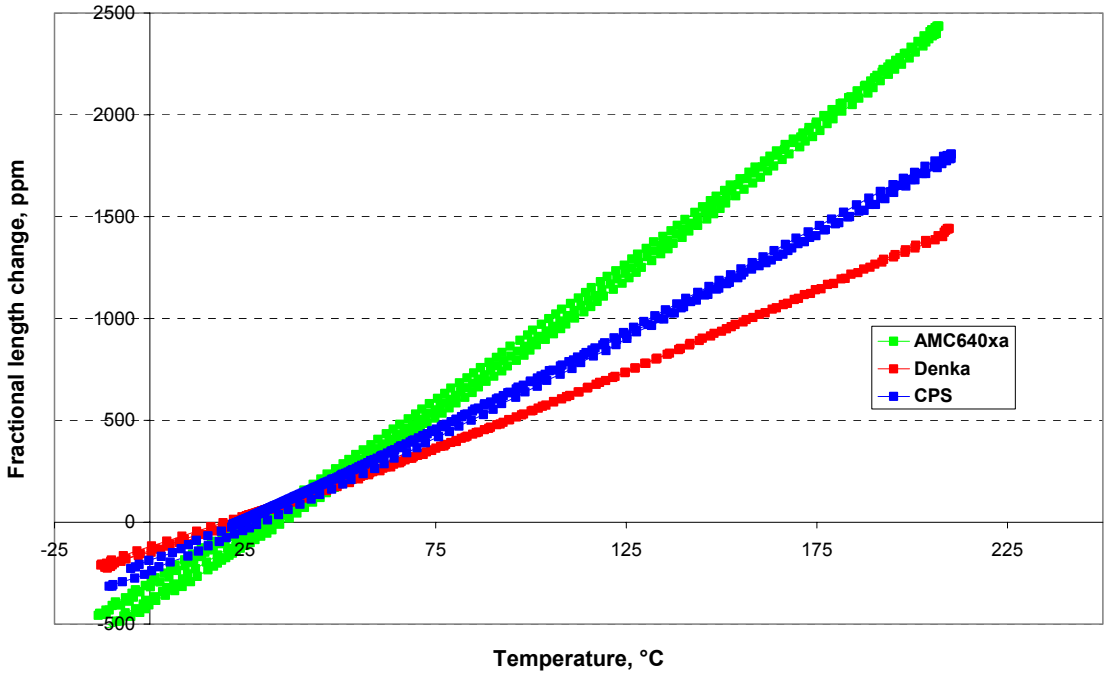


Fig 7: Typical thermal expansion behaviour of the 3 materials examined

For all the materials examined the curves above show very little hysteresis and this is an indication that the microstructure is relatively stable over the temperature range examined and that no significant residual stresses are present. Values for the CTE calculated from the slopes of these curves, together with measurements made using other techniques – strain gauge and image correlation methods - reported in Ref 12 and additional calibration measurements on aluminium and copper "reference" samples are given in Table 3. It is important to note that the values in Table 3 are not expected to be identical, as the tests have been carried out over different temperature ranges.

Material	Dilatometry *(0-200 ℃)*	Strain Gauges *(0-150 ℃)*	Image Correlation *(50-200 ℃)*
CPS	9.5	9.1	10.1-10.5
Denka	7.4	6.5	6.8-7.4
AMC640	13.5	12.6	11.2-12
Al	23.4	24.0	23.0-26.1
Copper	16.6	16.8	16.1–18.3

Table 3: Mean CTE values (x 10^{-6} $^{o}C^{-1}$) for the 3 MMCs and reference materials tested

As with the thermal diffusivity data presented above, the variation with temperature over specific range relative to the operating conditions of a device is often of interest rather than a single average CTE value for the whole temperature range. This is not always available, but sometimes "instantaneous CTE values", calculated over a small number of points and small temperature range are presented [8,11]. This is generally not the case for thermal diffusivity and conductivity where a single value (often calculated only at room temperature) is normally presented. Instantaneous CTE values are important because designers are often interested in the CTE behaviour over a small temperature range, representative of the temperature cycle of a particular component or package. The data from the tests shown in Fig 8 has been re-plotted in Fig 9 below to represent the "instantaneous CTE". A 5-point sliding average was used to smooth the data and reduce large fluctuations that might mask the true material response.

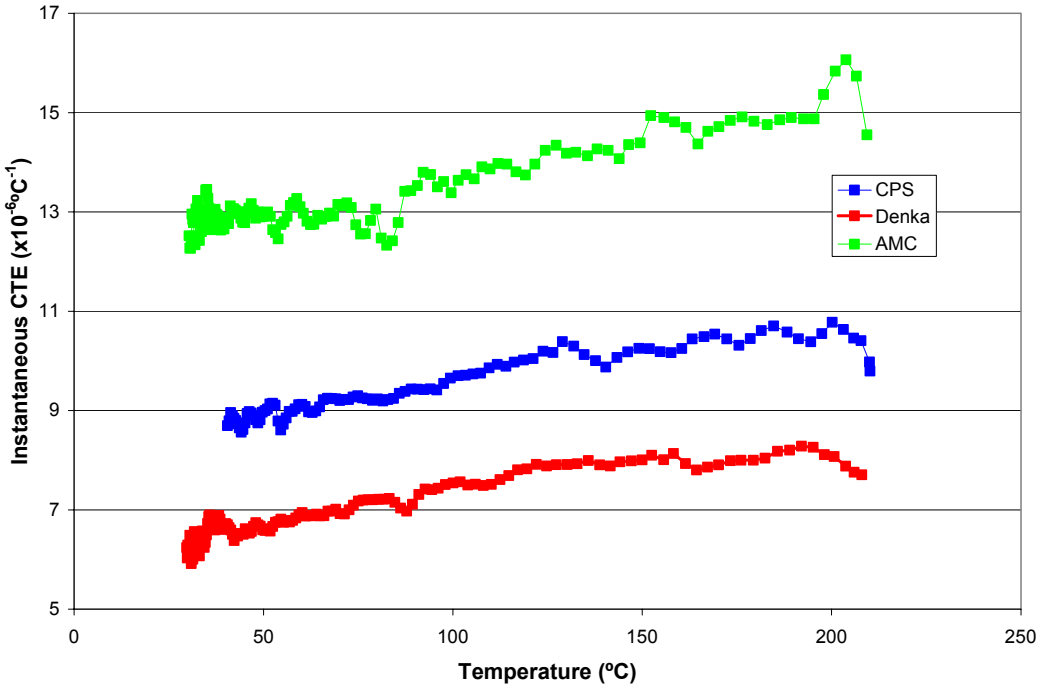


Fig 8: Variation of CTE value with temperature

7 DENSITY MEASUREMENTS

To calculate the values for thermal conductivity from the thermal diffusivity and specific heat measurements, the room temperature density of each material was measured using small rectangular bar specimens approximately 60mm x 4mm x 4mm. Each testpiece was first weighed in air to determine their mass, and then individually weighed in distilled water. The density was then calculated from the difference between the weight in air and the weight in water and from the density of pure water. Measurements were only made at room temperature, but are extrapolated to higher temperatures by considering the thermal expansion behaviour. Using the linear CTE values measured by dilatometry as part of this study (see later), the volumetric thermal expansion was calculated (for most materials this is approximately 3x the linear CTE) and the variation of density with temperature for the 3 materials predicted, as shown in Fig 10 and Table 4.

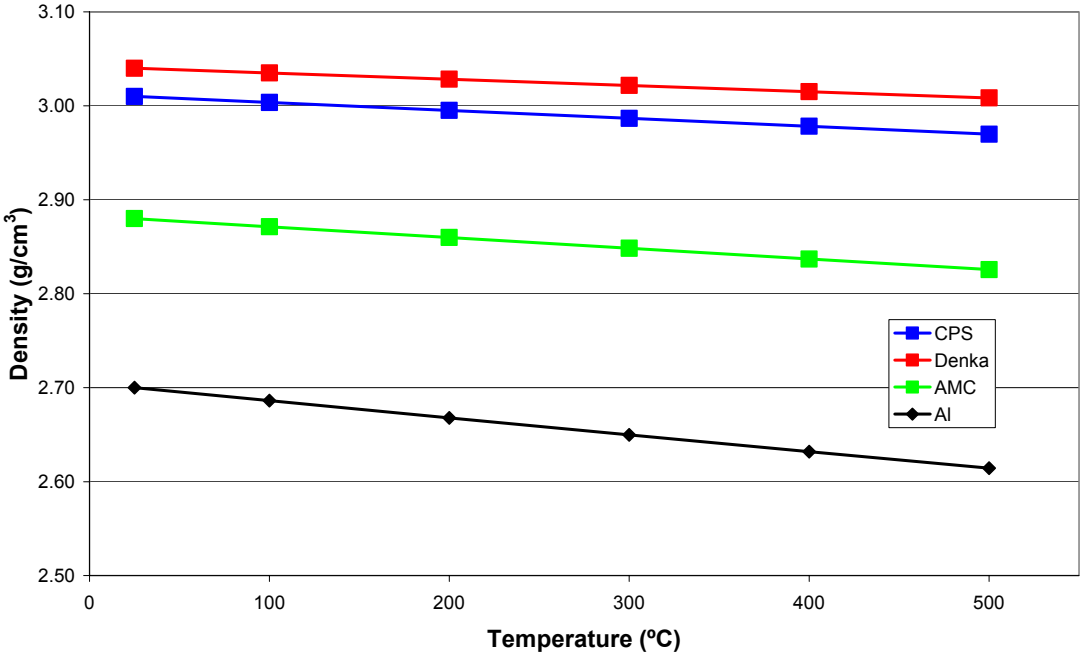


Fig 9: Predicted variation of density with temperature

8 THERMAL CONDUCTIVITY CALCULATIONS

As noted previously, the thermal conductivity, λ can be calculated directly from the relationship:

$$\lambda = a.\rho.C_p$$

… where a is the thermal diffusivity, ρ the density and C_p the specific heat capacity.

The results from the separate measurements of diffusivity, specific heat and density are summarised in Table 4 together with the calculated values for thermal conductivity. The variation of thermal conductivity with temperature for the three composites is plotted in Fig 10. Where possible, measurements were made at the same temperatures to enable direct reading of results, but in some cases interpolation of values was necessary to estimate the value of the parameter at the specified temperature. Again, for comparison, representative handbook data [7] for a range of aluminium alloys is also included.

Results show a similar trend to the thermal diffusivity data presented previously. At room temperature the CPS and Denka materials have the highest thermal conductivity values (of 176 $Wm^{-1}K^{-1}$ and 168 $Wm^{-1}K^{-1}$ respectively) but these fall to about two thirds of this value at 400 ºC. The thermal conductivity of the AMC640xa increases with temperature, following a similar behaviour to the unreinforced 6061 aluminium matrix alloy.

Thermal Diffusivity ($x10^{-4}$ m^2 s^{-1})						
	Temperature ºC					
	25	100	200	300	400	500
CPS AlSiC9	0.79	0.6	0.48	0.4	0.36	0.3
Denka MMC	0.94	0.62	0.48	0.39	0.34	0.28
AMC 640xa	0.65	0.66	0.655	0.66	0.68	0.685
Al 2024*	0.74	0.74	0.74	0.73	0.7	0.65
Al 6061*		0.76	0.775	0.78	0.76	0.75
Al 7075*		0.73	0.74	0.72	0.69	0.66
LM25*	0.69	0.67	0.63	0.6	0.56	0.55
Density (g/cm^3)						
	Temperature ºC					
	25	100	200	300	400	500
CPS AlSiC9	3.01	3.00	3.00	2.99	2.98	2.97
Denka MMC	3.04	3.03	3.03	3.02	3.01	3.01
AMC 640xa	2.88	2.87	2.86	2.85	2.84	2.83
Al 2024*	2.79	2.77	2.75	2.73	2.71	2.68
Al 6061*	2.71	2.70	2.68	2.66	2.64	2.61
Al 7075*	2.81	2.80	2.77	2.75	2.73	2.70
LM25*	2.68	2.66	2.64	2.62	2.60	2.58
Specific Heat (j/g.K)						
	Temperature ºC					
	25	100	200	300	400	500
CPS AlSiC9	0.74	0.86	0.97	0.99	1.06	1.20
Denka MMC	0.68	0.81	0.93	0.98	1.02	
AMC 640xa	0.74	0.84	0.93	0.97	1.02	
Al 2024*	0.85	0.90	0.95	0.97	1.00	1.07
Al 6061*	0.87	0.95	0.98	1.02	1.06	1.15
Al 7075*	0.85	0.91	0.96	0.98	1.04	1.10
LM25*	0.88	0.92	0.97	1.01	1.06	1.10
Thermal Conductivity (W m^{-1} K^{-1})						
	Temperature ºC					
	25	100	200	300	400	500
CPS AlSiC9	176	155	140	119	114	
Denka MMC	168	152	136	116	105	
AMC 640xa	138	159	174	182	196	
Al 2024*	175	185	193	193	190	188
Al 6061*		195	203	211	212	225
Al 7075*		186	197	194	196	196
LM25*	163	165	162	155	153	145

*** Handbook Data from Ref [7]**

Table 4: Summary of thermal property measurements and calculations

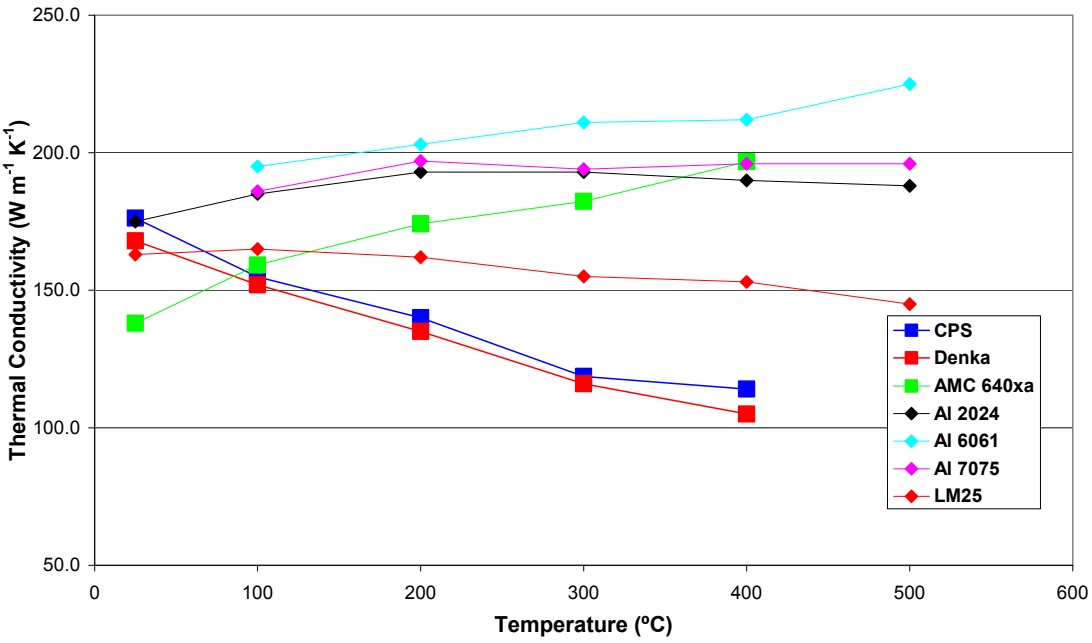


Fig 10: Thermal conductivity measurements

The main focus of this study is on the test methods used to measure thermal properties. Work examining the effect of changes in heat treatment, precipitation kinetics and the metallurgical state of the matrix on thermal properties are discussed in Refs 8-11 for some of the candidate materials being developed, and examined in this study. The reader is referred to these for detailed metallurgical analysis of the mechanisms involved. Ref 14 also includes further work examining the creep, dimensional stability and thermo-mechanical fatigue behaviour of the materials examined in this study.

9 SUMMARY

Unlike the measurements on mechanical properties reported in the accompanying Measurement Note [3], the work carried out as part of this study examining the thermal properties of high volume fraction MMCs have shown no significant practical issues and difficulties in using the standard techniques developed for metals. The thermal properties are clearly influenced by the composition, microstructure, matrix alloy, type, size and volume fraction of reinforcement and heat treatment condition, though from this study it is not possible to make statements about their individual effects.

Generally the quality and range of manufacturer's data available is not as comprehensive as required, and potential users are recommended to carry out their own measurements under test conditions appropriate to the application. This is particularly recommended for critical applications, because there can be significant variations with temperature, particularly with the thermal diffusivity and conductivity data.

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