

National Physical Laboratory

Centre for Materials Measurement and Technology

Thermal Properties of Composite Materials - an Interlaboratory Study

by

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Summary

An interlaboratory study of the measurement of thermal diffusivity and thermal expansion characteristics of metal and polymer matrix composites has been conducted with the purpose of determining the effect of measurement technique on the result and the likely reliability of data.

Reasonable agreement was obtained in most of the tests, although there remain some questions of reproducibility of thermal diffusivity results despite the use of "reference materials" with "recommended data" to determine apparatus performance. PMCs proved difficult to test in representative thicknesses because of their low thermal diffusivity. Reducing the thickness to a level where the rear-face thermal transient is more readily measured would result in testing only one or two plies which may not be representative of the full normal layup thickness.

In thermal expansion measurement, composite materials are shown to exhibit progressive size changes with thermal cycling. In part this is due to annealing effects on the first cycle as the test-piece adjusts to the heating/cooling rate of the tests, and to ratchetting effects due to plastic deformation in the matrix phase. When thermal expansion tests are made with a push-rod dilatometer, the added push-rod force appears to result in additional contraction effects, especially during the first thermal cycle, compared with interferometric tests in which the applied stress is much lower.

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1. INTRODUCTION

Composite materials often show variable effects in thermal properties resulting from thermal expansion mismatch between the matrix and the reinforcement. On cooling from the original fabrication temperature at which the matrix is stress-free, internal stresses are built up which depend on the cooling schedule and the type of reinforcement and its dispersion. On subsequent reheating to determine thermophysical properties, changes in the internal stress field occur as the matrix or its reinforcement undergo stress relaxation. In some cases these changes lead to new stable dimensions, but in others, thermal cycling leads to strain ratchetting, with progressive increases or decreases in length as the material progressive fatigues. Characterising the stability of the materials by measuring thermophysical characteristics is thus a key aspect of determining overall materials performance.

In many instruments designed to measure thermophysical properties, the form of the test-piece and the constraints applied to it vary considerably. In mechanical dilatometers for thermal expansion measurement, a push-rod measures the dimensional change as the test-piece is heated or cooled. The push-rod exerts some force in order to maintain constant contact with the test-piece, and this can induce deformations in the test-piece, particularly if the test-piece becomes soft. In addition, this force can result in irreversible movements of the test-piece during thermal cycling as the latter beds down stably in the supporting apparatus. Optical systems using interferometry to record displacement are an alternative to mechanical systems. No force is applied, apart from the mass of a small optical flat (< 1 g) placed on the upper surface of the test-piece. For materials which deform readily, such a system should show results closer to the true expansion behaviour than ones in which a mechanical force is applied.

In thermal diffusivity measurement, a small disc test-piece is subjected to a laser pulse on one face, and the transient temperature rise curve on the opposite face is recorded, from which the thermal diffusivity can be calculated. Although the technique has cost and time advantages over conventional thermal conductivity measurement, and requires only a small test-piece, it is restricted to measurements over a narrow range of test-piece thickness, which is ideally related to the thermal diffusivity being measured. There are therefore questions of conflict between representative material microstructures in the test-piece and the size requirements for the apparatus. In addition, even if the procedures in EN 821-2 or BS 7134:4.2 are followed, the software used to analyse the detected transients varies in detail from establishment to establishment, in particular the manner in which trace noise is dealt with.

As part of the Design with Composite Components (DCC) programme, Project 2 on Thermal Properties of Composites, an interlaboratory study was conducted between NPL, UMIST Materials Science Centre and AEA Technology, Risley with the purpose of establishing the repeatability and reproducibility of measurements on representative fibre-reinforced PMCs and particulate reinforced MMCs.

2. MATERIALS

The materials selected for this interlaboratory study were:

MMC: Alcan, "Duralcan W6A20A", 6061 Al alloy with 20% Al_2O_3 particulate

reinforced, solution treated and aged condition T5 (NPL code AAP105), thermal diffusivity and expansion measured in the axial direction. The billet was roughly rectangular in cross-section with rounded corners.

MMC: Advanced Metal Composites "AMC225", 2124 Al alloy with 25% SiC particulate reinforcement, as manufactured (extruded), condition T1 (NPL code ASP122), thermal diffusivity and expansion to be measured in the axial direction. The billet was approximately 35 mm diameter.

PMC: ACG woven 0-90° crossply carbon/epoxy mould material LTM10, 2/2 twill carbon fabric with 1-8-1 layup, outer plies with a finer weave than inner plies, thickness 6 mm with as-moulded surfaces (NPL code CFS003), thermal diffusivity measured through thickness, thermal expansion measured in plane and through thickness.

PMC: ACG woven 0-90° crossply glass/epoxy mould material LTM10, glass fabric with 1-8-1 layup, outer plies with a finer weave than inner plies, thickness 6 mm with as-moulded surfaces (NPL code KNC), thermal diffusivity measured through thickness, thermal expansion measured in-plane and through thickness.

Test-pieces were prepared in appropriate orientations by diamond machining. Thermal expansion test-pieces for mechanical dilatometry were in the form of small bars, 6 x 6 x 25 mm, while for the laser interferometry, rings were cut approximately 12 mm diameter and 6 mm thick with a 6 mm diameter central hole as required by the AEA Technology apparatus. Thermal diffusivity test-pieces were machined as small discs, 12 mm diameter for NPL and AEA Technology, and 10 mm diameter for UMIST. Normally, the thickness of thermal diffusivity test-pieces is controlled to a level such that the transient half-rise time is within a convenient window which acts as a compromise between, at shorter times, significant finite pulse time errors, and at longer times, high laser power requirements and heat loss errors. While both of these types of error can be corrected for using theoretical assessments, half-rise times between 50 and 200 ms are preferred to minimise the magnitude of the corrections. Thus for the MMCs with expected high thermal diffusivity, a thickness of 3 mm was prepared. For the PMCs, ideal thicknesses were of the order 1 mm or less, which was typical of the thickness of two plies. Using such thin test-pieces, results representative of the full thickness of a balanced weave would therefore not be obtained. Consequently, it was decided to machine the test-pieces from one side to a thickness about 3.2 mm, or about 50% of the overall thickness so that one-half of the structure would be present at the risk of making measurement difficult.

3. TESTING

3.1 Testing protocols

The test protocols were straightforward and simple. Each collaborator was to undertake the tests in their normal manner, providing data at a series of temperatures as follows:

MMC test-pieces: Thermal expansion: 25 °C, 100 °C, 200 °C, 300 °C, 400 °C
Thermal diffusivity: 25 °C, 200 °C, 400 °C

PMC test-pieces: Thermal expansion: 25 °C, 50 °C, 100 °C, 150 °C, 200 °C
 Thermal diffusivity: 25 °C, 100 °C, 200 °C

reflecting their respective potential use ranges. Measurements were to be performed over at least two thermal cycles for thermal diffusivity, recording at least three laser pulses at each temperature, and three thermal cycles for thermal expansion, in both cases with data recorded on both heating and cooling.

Collaborators were also asked to supply information concerning their calibration routines and any recent checks performed on their apparatus.

3.2 Test facilities

For thermal diffusivity measurement, the laboratories had different facilities, and the relevant details are summarised in Table 1. All had been constructed from various bought-in parts, rather than complete commercial systems.

For thermal expansion measurement, NPL's mechanical dilatometers are of commercial type (Linseis), but with modified test-piece holders, and recording and computational software developed in-house. UMIST's mechanical system is in-house constructed. AEA Technology's system is based on interferometry, and is in-house constructed.

4. RESULTS AND DISCUSSION

4.1 Thermal diffusivity

4.1.1 MMCs

The results are shown in Figures 1 and 2, and are summarised in Table 2, in which the individual determinations are averaged and rounded to two significant figures for clarity. Results were not recorded at all temperatures by all laboratories. They show some surprising differences between the laboratories. The MMCs showed high values of thermal diffusivity, and all laboratories demonstrated a fall with increasing temperature. However, average results at each temperature, the magnitude of the fall with increasing temperature, and the extent of hysteresis in temperature cycling varied significantly. Differences between laboratories were up to 20%, but more typically 10%. This is larger than any individual organisation proposed as their likely absolute measurement error, typically $\pm 4\%$ (EN 821-2 and BS 7134:4.2 quote $< 5\%$ as an expectation). In part this can be attributed to different test-piece support geometries, arrangements for detecting and recording transient temperature changes, and calculation and correction methods, and in part to potential small variations from test-piece to test-piece. AEAT provided recent data correlating thermal diffusivity measurements with data based on thermal conductivity for certified NIST reference materials (SRM 1461, stainless steel, and SRM 8421 electrolytic iron), Figure 3 apparently indicating agreement well within $\pm 5\%$.

However, the recording of different temperature dependences, which should be mostly independent of the details of the method, cannot readily be explained. Care was taken to ensure that the test-pieces were cut in the same orientation from the same billet of material,

and were surface finished in the same grinding operation. Although in principle the level of reinforcement could vary a little spatially, this is unlikely radically to affect the temperature dependence. One is forced to conclude that the reliability of the measurements is less than that required to identify any slight changes in the materials as a result of thermal cycling.

Following discussions with the participants, one (UMIST) felt that their facility was not operating under optimum conditions (a damaged mirror) at the time, and this may have influenced the results. Some repeat measurements on one of the MMCs (AMC225) were made later by a different operator after facility overhaul, and the results fell closer to those of other laboratories with a similar temperature dependence.

4.1.2 PMCs

The results from the **LTM10/carbon** PMC material (Table 2, Figure 4) show similar apparent marked differences in magnitude and temperature dependence between collaborators. The low values of thermal diffusivity posed some problems for the operators, who felt that the supplied test-piece was thicker (at 3 mm) than ideal (around 1 mm based on conventional $t_{1/2}$ ranges) for the measurement. As a result there was a risk that to obtain a detectable and reasonably low-noise transient for analysis the laser power might need to be raised to such a level that there was a risk of damaging the incident face of the test-piece. This was investigated by UMIST. Severe incident face damage was noted after three laser pulses (Figure 5), and the test-piece had to be ground down for subsequent measurements. All three collaborators noted a decline in thermal diffusivity with temperature cycling, but it is unclear whether this is a genuine change in the material bulk or an effect of progressive laser damage.

To reduce the test-piece thickness to an ideal level would reduce the number of plies to a level which might then no longer be representative for the material, so this was not done.

Of the collaborators, only AEA Technology was able to measure the **LTM10/glass** material, in this case not with the laser facility, which proved inappropriate, but with a xenon flash-lamp system using a thermocouple bonded to the centre of the rear test-piece face. Thermal diffusivity values approximately 60% of those of LTM10/carbon were obtained (Figure 6), although because of the use of a thermocouple to detect the temperature transient, such results risk not being representative because of the lateral structural inhomogeneity of woven PMCs possibly producing local surface transient temperature variations. Of the other collaborators, UMIST said the test-piece was translucent, but did not attempt to graphite coat it, and NPL could not obtain a suitable transient using the normal laser method.

NPL's test-piece was then thinned from 3.2 mm to about 1.3 mm to obtain the results shown in Figure 7, at the risk of them no longer being representative of the as-moulded full number of plies.

4.1.3 Discussion

It is therefore clear that the laser flash method of determining the thermal transport behaviour of PMC through a representative thickness is appropriate only if the thickness is compatible with the technique employed. For larger thicknesses other appropriate heat sources and detectors may be required. Alternatively, thermal conductivity measurement using a steady

state disc or panel method could be used. On the other hand, the laser-flash method works well for high thermal conductivity materials such as MMCs, but there remain questions of the reproducibility between laboratories.

The laser flash method is said not to need calibration as such because thermal diffusivity is determined from the shape of a time transient and the test-piece thickness. Calibration is required only of the recording system time-base and a micrometer for measuring test-piece thickness. However, this assumes that the conditions used in the theoretical solution of the heat-flow equation are upheld in practice. Possible departures from the ideal situation include:

1. Non-uniform laser irradiation of the receiving face, resulting in some lateral heat flow. This can be minimised by use of a beam spreader, but can be difficult to control with a given laser source. The larger the test-piece diameter the more inhomogeneous the incident beam is likely to be.
2. Lateral conductive heat losses into the test-piece holder, usually minimised by use of point contact clamping.
3. Radiation losses, normally minimised by operating with the appropriate thickness. The proportion due to lateral losses is likely to be greater for smaller diameter thicker test-pieces.
4. Non-zero width input pulse. Lasers using a pulse duration of less than 1 ms normally do not pose any problems.
5. Unstable background temperature. If, for example, the test-piece temperature is rising when the flash is made, such that over the analysis period the rise is more than a few percent of the laser induced back-face transient, the transient rise time is apparently lengthened, and the thermal diffusivity underestimated.

In the present work, these factors are different for each of the sets of equipment used. In addition, there are different methods of analysis, involving curve-smoothing and fitting routines, either of the first part of the transient or the whole curve. Usually these are based on special software packages to speed the evaluation process. In some cases, thermal expansion of the test-piece thickness was corrected for, although this accounts only for differences of a percent or so. The differences in experimental set-up and analysis techniques are summarised in Table 1.

All laboratories involved occasionally tested one or more "reference materials" to check on apparatus function, and claimed their results to be within a 2-3% of the "recommended values". One provided such evidence. All reported their systems to be functioning normally (except for a faulty mirror in one case) and to have been recently checked using such references. With hindsight it might have been useful to include one of these "reference materials" as a blind test-piece in the interlaboratory study.

Thermal expansion

4.2.1 MMCs

Thermal expansion data for the MMCs recorded using mechanical dilatometry are shown in Table 3. For the three **W6A20A** test-pieces evaluated, there is reasonable agreement between the two laboratories involved (Figures 8, 9). There is a small annealing effect in the first thermal cycle, leading to a moderate but variable offset between heating and cooling apparent lengths. In the **AMC225** material, the annealing effect is larger and more variable between test-pieces (Figures 10, 11). (UMIST found a large low temperature movement in the first cycle, but this author attributes this mainly to settlement of the test-piece in the apparatus. True annealing effects set in gradually above about 200 °C.)

For these measurements, NPL used a horizontal silica dilatometer, whereas UMIST used a vertical alumina dilatometer for which the apparatus correction is a much larger proportion of the result. Despite this, the results are in quite reasonable agreement. It will be noted that the net offsets over the second and third cycles are small but fairly consistent. This represents the material ratchetting effect. Consistent determination of this effect requires the mechanical position of the test-piece in the apparatus to be very stable, and this for the most part appears to have been achieved. Expansion coefficients calculated from these data would be consistent to within $1 \times 10^{-6} \text{ K}^{-1}$ (25-400 °C), which is reasonable considering that the different test-pieces came from different cross-sectional positions within the original billets, and there could be some spatial variation of reinforcement.

The results on the **W6A20A** MMC from the laser interferometer at AEA Technology (Table 4, Figure 12) are in fairly good agreement with the mechanical dilatometry results. They too show some hysteresis between heating and cooling, and a net offset in each cycle, although this is considerably smaller than found by mechanical dilatometry, suggesting that the small push-rod forces are sufficient to exaggerate offsets on cycling.

In order to check on billet homogeneity and isotropy, NPL also undertook some mechanical dilatometer measurements on test-pieces cut perpendicular to the billet axis. The results (Table 5) show again that the annealing transient is variable, leading to a variable offset on the first thermal cycle, but in subsequent cycles, behaviour is uniform, and the results are similar to those on axial test-pieces given in Table 2.

PMCs

The mechanical dilatometry results for the **in-plane** test-pieces are shown in Table 6. NPL used a vertical silica system, and UMIST a vertical alumina system. There are significant differences between the detected results, but these are ascribable to likely small calibration differences which are especially noticeable with low thermal expansion materials. In the case of near zero expansion materials such as CFRP, the correction factors for a silica dilatometer system are rather less than for an alumina system, so the potential errors are considered as likely to be smaller. In both cases, the expansion behaviour on the first cycle was found to be different from subsequent cycles with a significant shrinkage in both types of material. This is attributable to annealing effects. In the second, third and fourth thermal cycles, further slight shrinkage occurred, but in all cases this was rather less than 100 ppm per cycle (see

Figures 13, 14).

NPL conducted **through-thickness** mechanical dilatometry measurements on the same materials. The results (Table 7) show that in the as-received condition, the LTM10/carbon material displays a higher expansion coefficient, and this reduces progressively with subsequent cycling. In contrast, the LTM10/glass material shows a reduced expansion coefficient on first heating and a much larger net offset over the first cycle. Subsequent cycles showed a raised expansion coefficient, with fairly consistent behaviour, continuing to show a net shrinkage over each cycle. A somewhat surprising feature of these results is that after stabilising the expansion behaviour with cycling, the through-thickness expansion coefficients of the carbon-containing material are greater than those of the glass-containing material, despite the fact that the carbon reinforcement has a lower expansion coefficient than the glass.

AEA Technology also made **through-thickness** measurements by laser interferometry and obtained results similar to those from mechanical dilatometry. The data are plotted in Figures 15 and 16. Note that on the first cycle, the test-piece was held at 100 and 150 °C, and a progressive shrinkage was noted, with an expansion that is reduced compared with the second and third cycles performed at constant heating rate. The numerical data are given in Table 8 which shows that the net offset over the first cycle is reduced compared with the mechanical dilatometry results, but there is otherwise quite good agreement between mechanical and interferometric data.

4.2.3 Discussion

All these data were obtained using a slow temperature ramping technique. This tends to result in some temperature variation in the test-piece, and a risk that the measuring thermocouple, which may only be touching the test-piece surface, is not recording the true average test-piece temperature. The consequence is a slight difference in apparent behaviour on heating and cooling (the average test-piece temperature probably lags the external thermocouple temperature on heating, but may be hotter on cooling). However, in principle, much of this can be removed by appropriate calibration, but there still remains a tendency for the transient on starting a thermal cycle to be inadequately catered for compared with the equivalent cooling. This is reflected in the dilatometer results over the range 25 to 100 °C which are slightly lower than those obtained during the subsequent cooling 100 to 25 °C.

For PMC materials, the potential for push-rod pressure to cause distortion of the test-piece is evident in comparing the mechanical and interferometric data for the through-thickness test-pieces. The results at temperatures up to 150 °C, where the resin component is rigid, are similar for the two techniques. This was the limit used for the NPL tests, which induced a considerable offset in each cycle. The AEA Technology interferometric tests were taken to over 200 °C, but resulted in smaller offsets than the mechanical tests.

The difference in through-thickness behaviour of the two PMC materials, with the thermal expansion of the carbon-reinforced material being apparently higher than that of the glass-reinforced material in both mechanical and interferometric tests, may appear surprising in view of the lower expansion coefficient of the carbon reinforcement than the glass. Leaving aside potential small differences in volume fraction, this effect also results when estimates of thermal expansion behaviour are made using a thermoelastic model for uncracked planar

balanced cross-ply materials¹. The explanation appears to be that, compared with glass-fibre reinforced material, the greater thermal expansion and elastic mismatches between fibre and resin in the case of carbon fibres transfers to the resin component more of the thermal contraction strains built up on cooling. These strains are tensile in both axial and hoop directions around each fibre, and result in a strong Poisson contraction effect in a radial direction. With no through-thickness reinforcement, this means that the apparent thermal expansion behaviour does not obey a simple rule of mixtures, but is influenced considerably by Poisson contraction effects, which for a given volume fraction are greater with carbon fibres than with glass fibres. Clearly, matrix cracking relieves stresses to a significant extent, and the differences found in practice are rather less than the predictions for a crack-free matrix.

5. CONCLUSIONS

In an interlaboratory measurement study of the thermal properties of composite materials, it has been shown that:

1. The range of results between laboratories in thermal diffusivity measurement (10-20%) is not as small as the claimed individual accuracies ($\pm 4\%$ or better), despite the calibration of electronic equipment and the use of "reference materials" with "recommended data". Further consideration needs to be given to determine specific causes of variations, but these may be both operator and calculation algorithm dependent.
2. Thermal diffusivity of polymer matrix composites is difficult to measure reliably by the laser flash/i.r. detector method using representative thicknesses of layup. The low values obtained ($< 0.5 \text{ mm}^2 \text{ s}^{-1}$) mean that the optimum thickness is less than 1 mm, encompassing only one or two plies. Results obtained on such a thickness would be prone to spatial variation as a result of weaves and inter-ply resin layers, and could not therefore reliably be extrapolated to the full layup thickness. A flash-lamp/thermocouple method appears more capable of coping with several millimetres test-piece thickness. Direct thermal conductivity measurements could be more appropriate.
3. A comparison of thermal expansion measurements shows that results for the MMCs have a spread of about $\pm 0.5 \times 10^{-6} \text{ K}^{-1}$ in mean coefficient, but there are differences which can probably be ascribed to the test-piece shifting in the apparatus and calibration corrections, the latter being much larger for alumina dilatometers than for those based on fused silica. Some spatial variation in the test materials cannot be ruled out.
4. Thermal expansion measurements made by mechanical dilatometer show hysteresis effects, and permanent offsets due to annealing (on the first cycle) and ratchetting (on subsequent cycles). The permanent offsets tend to be greater than in comparable interferometric measurements in which the applied force is negligible. This applied

¹ Using thermoelastic models developed at NPL by Dr L N McCartney.

particularly to PMCs.

5. The through-thickness thermal expansion coefficients for two similarly constructed PMCs show the surprising situation of being greater for the carbon fibre reinforced material than for the glass-reinforced material, whereas for the reinforcements themselves the carbon has a lower expansion coefficient than the glass. This result can be explained by a cross-ply laminate model in which the high stiffness and greater thermal expansion mismatch between the resin and the carbon fibre leads to enhanced Poisson contraction effects compared with glass reinforcement.

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Table 1 - Comparison of test protocols for thermal diffusivity measurement

Test feature	NPL	UMIST	AEA Technology
Test-piece diameter	12 mm	10 mm	12 mm
Coating test-piece	Graphite, both faces	None	MMC: graphite, receiving face PMC: graphite both faces
Mounting, heating system	Tantalum three-point ring clamp, tantalum tube furnace	Mounted in graphite susceptor, r.f. heating	Molybdenum three-point ring clamp in a graphite tube furnace
Pulse source	Nd-glass class 4, 40 J setting, 0.8 ms pulse width	Nd-glass class 4, 100 J, 0.67 ms pulse width	Nd-glass laser and xenon flash lamp
Illumination area	11.5 mm diameter disc	Normally 9 mm diameter; for PMC, restricted to ~ 6 mm diameter by an annulus placed over the test-piece so that laser damage could be assessed	11 mm diameter disc
Atmosphere	Vacuum	Vacuum	Vacuum
Temperature measurement	Type R thermocouple, close to but not touching test-piece	Type R thermocouple	Type E thermocouple
Radiation sighting area, focusing and transient detector	11.0 mm diameter circle, CaF ₂ lens, InSb detector	Approx. 9 mm diameter circle, CaF ₂ lens, InSb detector.	MMC: Liquid N ₂ cooled InSb detector viewing 2 mm diameter circle of back face PMC: Thermocouple detection using flash tube source rather than laser.
Calculation	Polynomial fits to rise and peak regions, radiation correction using $5t_{1/2}$ and $10t_{1/2}$ method (Cowan), finite pulse time correction (Taylor/Clark).	BS 7134:4.2. Details not given, but based on $t_{1/2}$ values, corrected for finite pulse time (Taylor/Clark) and heat losses (Cowan).	BS 7134:4.2, whole curve fitted, finite pulse time (Taylor/Clark), heat loss (Cowan) and thermal expansion correction.
Corrected for thermal expansion	No	No	Yes, $20 \times 10^{-6} \text{ K}^{-1}$ assumed (effect of correction small)
Calibration	Annual check on time-base of oscilloscope system, micrometer, etc.	No details	Annual check on frequency response, linearity of amplifier/oscilloscope system
Reference material checks	"Within 2% of recommended data for Poco AXM5Q1 graphite."	"Within 1% of recommended values", 24 May 1996 (Poco Graphite)	NIST electrolytic iron, Jan 96 Stainless steel, Jan 96, both within 3% of "reference" values up to 600 °C

Table 2 - Thermal diffusivity
(averaged and rounded data for three laser pulses)

Material	Laboratory	Thermal diffusivity, 10 ⁻⁶ m ² s ⁻¹							
		RT	200 °C	400 °C	200 °C	RT	200 °C	400 °C	RT
W6A20A axial	NPL	57	52	48	-	57	52	48	-
	UMIST 1	63	63	62	61	65	62	58	65
	AEA	59	56	52	58	62	56	53	-
AMC225 axial	NPL	61	54	47	-	61	54	47	-
	UMIST 1	69	65	61	67	69	65	62	68
	UMIST 2*	61	57	43	51	55 (120°C)			
	AEA	60	58	51	58	63	60	52	63
		RT	100 °C	200 °C	100 °C	RT	100 °C	200 °C	RT
LTM10/ carbon through thickness	NPL	0.40	0.36	0.297	-	0.392	0.35	0.297	-
	UMIST 1**	0.37	0.35	0.35	0.35	0.35	0.34	0.32	0.33
	AEA	0.37	0.32	0.28	-	0.40	0.37	0.31	-
LTM10/ glass through thickness	NPL***	0.12	0.14	0.14	-	0.07	0.10	0.13	-
	UMIST****	-	-	-	-	-	-	-	-
	AEA	0.27	0.24	0.21	-	0.28	0.26	0.23	-

* Second operator at a later data after facility overhaul ** After removing laser damage from front face
*** Thin test-piece, 1.3 mm instead of full 3.2 mm **** For comments, see text

Table 3A - Thermal expansion - mechanical dilatometry - axial - metal matrix composites
(Heating rate: NPL 2 K min⁻¹, UMIST 2 K min⁻¹. Data: rounded to nearest 10 ppm)

Material	Laboratory	Run	Thermal expansion, ppm over range									
			25-100	25-200	25-300	25-400	400-25	300-25	200-25	100-25	Offset	
W6A20A axial	NPL1	1	1250	3210	5430	7590	7740	5500	3350	1370	-310	
		2	1250	3180	5280	7480	7670	5450	3310	1330	-200	
		3	1250	3200	5350	7570	7640	5430	3290	1320	-30	
	NPL2	1	1280	3290	5510	7510	7830	5520	3330	1320	-770	
		2	1300	3310	5470	7630	7680	5420	3260	1260	-160	
		3	1280	3250	5410	7590	7780	5510	3340	1320	-250	
	UMIST	1	1260	3200	5240	6740	7700	5350	3230	1260	-1160	
		2	1130	3080	5160	7180	7500	5244	3170	1200	-400	
		3	1230	3180	5260	7340	7570	5310	3220	1260	-380	
AMC225 axial	NPL1	1	1170	2860	4560	6610	7080	4970	2880	1130	-490	
		2	1160	2870	4690	6800	7040	4940	2850	1100	-100	
		3	1190	2950	4810	6950	7240	5160	2770	1090	-90	
	NPL2	1	1160	2780	3970	5660	7250	5090	2940	1160	-2290	
		2	1170	2920	4790	6950	7000	5050	2920	1140	-220	
		3	1180	2930	4820	6980	7190	5050	2940	1170	-160	
	UMIST	1	-290	1180	2650	4500	6920	4840	2820	1100	-2900	
		2	1010	2680	4530	6730	6900	4810	2760	1050	-160	
		3	1110	2800	4630	6860	7010	4950	2800	1110	-180	

Table 3B - Thermal expansion coefficients - mechanical dilatometry - axial - metal matrix composites
(Heating rate: NPL 2 K min⁻¹, UMIST 2 K min⁻¹. Data: rounded to nearest 0.1 x 10⁻⁶ K⁻¹)

Material	Labora- tory	Run	Thermal expansion coefficient, 10 ⁻⁶ K ⁻¹ , over range								Offset, ppm
			25-100	25-200	25-300	25-400	400-25	300-25	200-25	100-25	
W6A20A axial	NPL1	1	16.7	18.3	19.7	20.2	20.6	20.0	19.1	18.3	-310
		2	16.7	18.2	19.2	19.9	20.5	19.8	18.9	17.7	-200
		3	16.7	18.3	19.5	20.2	20.4	19.7	18.8	17.6	-30
	NPL2	1	17.1	18.8	20.0	20.0	20.9	20.1	19.0	17.6	-770
		2	17.3	18.9	19.9	20.3	20.5	19.7	18.6	16.8	-160
		3	17.1	18.6	19.7	20.2	20.7	20.0	19.1	17.6	-250
	UMIST	1	16.8	18.3	19.1	18.0	20.5	19.5	18.5	16.8	-1160
		2	15.1	17.6	18.7	19.1	20.0	19.1	18.1	16.0	-400
		3	16.4	18.2	19.1	19.6	20.2	19.3	18.4	16.8	-380
AMC225 axial	NPL1	1	15.6	16.3	16.6	17.6	18.9	18.1	16.5	15.1	-490
		2	15.5	16.4	17.1	18.1	18.8	18.0	16.3	14.7	-100
		3	15.9	16.9	17.5	18.5	19.3	18.8	15.8	14.5	-90
	NPL2	1	15.5	15.9	14.4	15.1	19.3	18.5	16.8	15.5	-2290
		2	15.6	16.7	17.4	18.5	18.7	18.4	16.7	15.2	-220
		3	15.7	16.7	17.5	18.6	19.2	18.4	16.8	15.6	-160
	UMIST	1	-3.9	6.7	9.6	12.0	18.5	17.6	16.1	14.7	-2900
		2	13.5	15.3	16.5	17.9	18.4	17.5	15.8	14.0	-160
		3	14.8	16.0	16.8	18.3	18.7	18.0	16.0	14.8	-180

Table 4A - Thermal expansion laser interferometry - metal matrix composites

Material	Labora- tory	Run	Thermal expansion, ppm over range								
			25-100	25-200	25-300	25-400	400-25	300-25	200-25	100-25	Offset
W6A20A axial	AEA	1	1382	-	-	-	-	-	-	-	-
		2	1372	3399	5510	7668	7749	5460	3335	1372	-115
		3	1371	3395	5601	7692	7733	5475	3336	1392	-101

Table 4B - Thermal expansion coefficients - laser interferometry - metal matrix composites

Material	Labora- tory	Run	Thermal expansion, 10^{-6} K^{-1} , over range								Offset, ppm
			25-100	25-200	25-300	25-400	400-25	300-25	200-25	100-25	
W6A20A axial	AEA	1	18.43	-	-	-	-	-	-	-	-
		2	18.29	19.42	20.04	20.45	20.66	19.85	19.06	18.29	-115
		3	18.43	19.40	20.37	20.51	20.62	19.91	19.06	18.56	-101

Table 5A - Thermal expansion - mechanical dilatometry - transverse - metal matrix composites
(Data rounded to nearest 10 ppm)

Material	Laboratory/ specimen	Run	Thermal expansion, ppm over range								Offset
			25-100	25-200	25-300	25-400	400-25	300-25	200-25	100-25	
W6A20A	NPL3, perp. to axis, longer side	1	1360	3430	5530	7700	8280	5860	3580	1430	-800
		2	1350	3470	5770	8210	8270	5830	3550	1400	-180
		3	1360	3480	5760	8200	8310	5870	3590	1460	-190
		4	1350	3480	5790	8230	8390	5930	3650	1500	-190
	NPL4, perp. axis, shorter side	1	650	2040	4070	5940	8340	5900	3620	1480	-3160
		2	1360	3510	5800	8220	8310	5850	3580	1450	-280
		3	1340	3460	5760	8280	8310	5850	3450	1410	-260
		4	1360	3480	5750	8200	8270	5820	3560	1450	-160
AMC225	NPL1, perp. to length, diametral	1	1060	2650	4390	6310	6480	4520	2650	1100	-340
		2	1090	2720	4560	6550	6570	4590	2720	1120	-55
		3	1090	2710	4540	6540	6450	4510	2710	1110	+50
		4	1100	2710	4530	6520	6460	4500	2710	1090	+30

Table 5B - Thermal expansion coefficients - mechanical dilatometry - transverse - metal matrix composites
(Data rounded to nearest 0.1 x 10⁻⁶ K⁻¹)

Material	Laboratory/ specimen	Run	Thermal expansion, 10 ⁻⁶ K ⁻¹ , over range								Offset, ppm
			25-100	25-200	25-300	25-400	400-25	300-25	200-25	100-25	
W6A20A	NPL3, perp. to axis, longer side	1	18.1	19.6	20.1	20.5	22.1	21.3	20.5	19.1	-800
		2	18.0	19.8	21.0	21.9	22.1	21.2	20.3	18.7	-180
		3	18.1	19.9	20.9	21.9	22.2	21.3	20.5	19.5	-190
		4	18.0	19.9	21.1	21.9	22.4	21.6	20.9	20.0	-190
	NPL4, perp. axis, shorter side	1	8.7	11.7	14.8	15.8	22.2	21.5	20.7	19.7	-3160
		2	18.1	20.1	21.1	21.9	22.2	21.3	20.5	19.3	-280
		3	17.9	19.8	20.9	22.1	22.2	21.3	19.7	18.8	-260
		4	18.1	19.9	20.9	21.9	22.1	21.2	20.3	19.3	-160
AMC225	NPL1, perp. to length, diametral	1	14.1	15.1	16.0	16.8	17.3	16.4	15.1	14.7	-340
		2	14.5	15.5	16.6	17.5	17.5	16.7	15.5	14.9	-55
		3	14.5	15.5	16.5	17.4	17.2	16.4	15.5	14.8	+50
		4	14.7	15.5	16.5	17.4	17.2	16.4	15.5	14.5	+30

Table 6A - Thermal expansion - mechanical dilatometry - polymer composites - in plane
(Data rounded to nearest 10 ppm)

Material	Laboratory	Run	Thermal expansion, ppm over range									
			25-50	25-100	25-150	25-200	200-25	150-25	100-25	50-25	Offset	
LTM10/ carbon in plane	NPL1	1	40	0	90	-	-	280	140	50	-430	
		2	50	190	320	-	-	300	130	50	+30	
		3	50	180	310	-	-	260	140	50	-20	
	UMIST	1	80	250	330	410	700	460	200	90	-290	
		2	110	310	510	670	710	480	290	90	-30	
		3	90	280	430	620	670	470	270	90	-40	
LTM10/ glass in plane	NPL1	1	250	680	1080	-	-	1350	710	210	-500	
		2	240	740	1290	-	-	1300	700	210	-70	
		3	250	770	1340	-	-	1300	680	210	-40	
	UMIST	1	80	260	700	1030	2100	1520	850	290	-1020	
		2	300	870	1540	2030	2070	1520	850	280	-50	
		3	270	860	1520	2050	2120	1560	880	310	-90	

Table 6B - Thermal expansion coefficients - mechanical dilatometry - polymer composites - in plane
(Data rounded to nearest 10⁻⁶ K⁻¹)

Material	Laboratory	Run	Thermal expansion, ppm over range								Offset, ppm
			25-50	25-100	25-150	25-200	200-25	150-25	100-25	50-25	
LTM10/ carbon in plane	NPL1	1	1.6	0.0	0.7	-	-	2.2	1.9	2.0	-430
		2	2.0	2.5	2.6	-	-	2.4	1.7	2.0	+30
		3	2.0	2.4	2.5	-	-	2.1	1.9	2.0	-20
	UMIST	1	3.2	3.3	2.6	2.3	4.0	3.7	2.7	3.6	-290
		2	4.4	4.1	4.1	3.8	4.1	3.8	3.9	3.6	-30
		3	3.6	3.7	3.4	3.5	3.8	3.8	3.6	3.6	-40
LTM10/ glass in plane	NPL1	1	10.0	9.1	8.6	-	-	10.8	9.5	8.4	-500
		2	9.6	9.9	10.3	-	-	10.4	9.3	8.4	-70
		3	10.0	10.3	10.7	-	-	10.4	9.1	8.4	-40
	UMIST	1	3.2	3.5	5.6	5.9	12.0	12.2	11.3	11.6	-1020
		2	12.0	11.6	12.3	11.6	11.8	12.3	11.3	11.2	-50
		3	10.8	11.5	12.2	11.7	12.1	12.5	11.7	12.4	-90

Table 7A - Thermal expansion - mechanical dilatometry - polymer matrix composites - through thickness
(Data rounded to nearest 10 ppm)

Material	Laboratory	Run	Thermal expansion, ppm over range									
			-25 to 25	0 to 25	25 to 50	25 to 100	25 to 150	150 to 25	100 to 25	50 to 25	Offset	
LTM10/ carbon through thickness	NPL	1	-2540	-1250	1460	5200	8510	8290	4260	1480	-1900	
		2	-2420	-1180	1420	4720	8000	7630	3950	1200	-550	
		3	-2460	-1180	1300	4320	7440	7260	3710	1320	-630	
		4	-2540	-1180	1220	4280	7490	6830	3430	1120	-320	
LTM10/ glass through thickness	NPL	1	-	-880	880	-1425	150	o/s	o/s	o/s	-6070	
		2	-1810	-930	1120	3860	o/s	o/s	3550	1060	-710	
		3	-	-960	1110	3750	6560	6560	3560	1100	-430	
		4	-1800	-930	1070	3760	6600	6450	3500	(1060)	-120	

Table 7B - Thermal expansion coefficients - mechanical dilatometry - polymer matrix composites - through thickness
(Data rounded to nearest $0.1 \times 10^{-6} \text{ K}^{-1}$)

Material	Laboratory	Run	Thermal expansion, ppm over range								
			-25 to 25	0 to 25	25 to 50	25 to 100	25 to 150	150 to 25	100 to 25	50 to 25	Offset
LTM10/ carbon through thickness	NPL	1	50.8	50.0	58.4	69.3	68.1	66.3	56.8	59.2	-1900
		2	48.4	47.2	56.8	62.9	64.0	61.0	52.7	48.0	-550
		3	49.2	47.2	52.0	57.6	59.5	58.1	49.5	52.8	-630
		4	50.8	47.2	48.8	57.1	59.9	54.6	45.7	44.8	-320
LTM10/ glass through thickness	NPL	1	-	35.2	35.2	-19.0	1.2	o/s	o/s	o/s	-6070
		2	36.2	37.2	44.8	51.5	o/s	o/s	47.3	42.4	-710
		3	-	38.4	44.4	50.0	52.4	52.5	47.5	44.0	-430
		4	36.0	37.2	42.8	50.1	52.8	51.6	46.7	42.4	-120

Table 8A - Thermal expansion - interferometric dilatometry - polymer matrix composites - through-thickness
(Data rounded to nearest 1 ppm)

Material	Laboratory	Run	Thermal expansion, ppm over range								
			25 to 50	25 to 100	25 to 150	25 to 200	200 to 25	150 to 25	100 to 25	50 to 25	Offset
LTM10/ carbon through thickness	AEA	1	1430	4244	5919	10125	12249	6932	3798	1176	-989
		2	1124	3970	7221	11271	11801	6886	3792	1175	-23
		3	1248	3999	7222	11203	10897	6762	3668	1132	-213
LTM10/ glass through thickness	AEA	1	1294	3527	4893	8524	10358	6372	3622	(1294)	-1992
		2	(1294)	3578	6406	10293	10197	6348	3597	(1294)	0
		3	(1294)	3546	6404	10189	10111	6335	3567	(1294)	1

Note: For LTM10/glass, the test-piece was not cooled to 25 °C between runs. The data in the above table assume a value of 1294 ppm to extrapolate results to 25 °C, assuming that there is no influence of cycling on thermal expansion over the range 25°C to 50 °C.

Table 8B - Thermal expansion coefficients - interferometric dilatometry - polymer matrix composites - through-thickness
(Data rounded to nearest $0.1 \times 10^{-6} \text{ K}^{-1}$)

Material	Laboratory	Run	Thermal expansion, ppm over range								Offset, ppm
			25 to 50	25 to 100	25 to 150	25 to 200	200 to 25	150 to 25	100 to 25	50 to 25	
LTM10/ carbon through thickness	AEA	1	57.2	56.6	47.4	57.9	70.0	55.5	50.6	47.0	-989
		2	45.0	52.9	57.8	64.4	67.4	55.1	50.6	47.0	-23
		3	49.9	53.3	57.8	64.0	62.3	54.1	48.9	45.3	-213
LTM10/ glass through thickness	AEA	1	51.8	47.0	39.1	48.7	59.2	51.0	48.3	(51.8)	-1992
		2	(51.8)	47.7	51.2	58.8	58.3	50.8	48.0	(51.8)	0
		3	(51.8)	47.3	51.2	58.2	57.8	50.7	47.6	(51.8)	1

Note: For LTM10/glass, the test-piece was not cooled to 25 °C between runs, The data in the above table assume a value of $51.8 \times 10^{-6} \text{ K}^{-1}$ to extrapolate results to 25 °C, assuming that there is no influence of cycling on thermal expansion over the range 25°C to 50 °C.

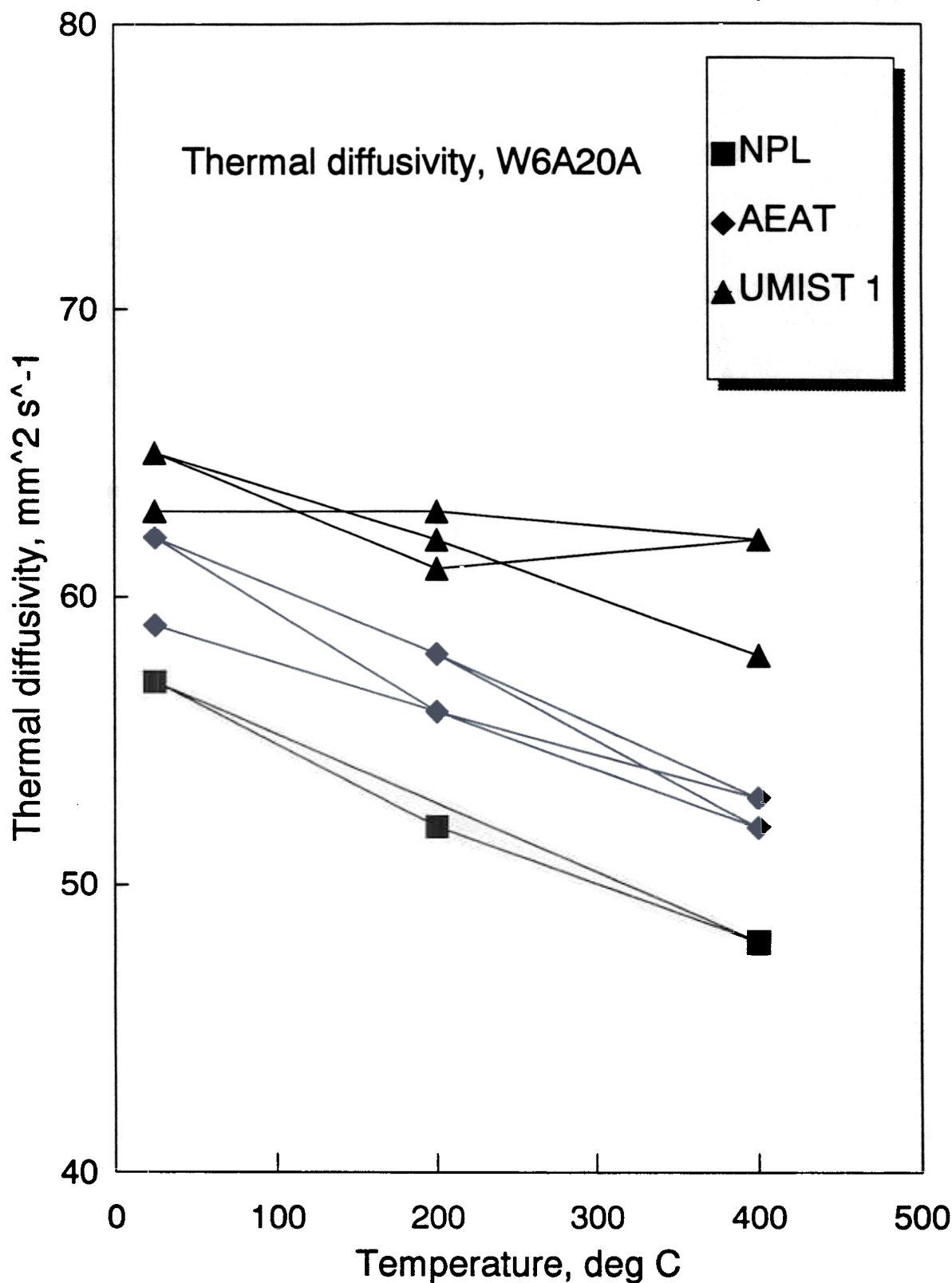


Figure 1 Thermal diffusivity of W6A20A MMC as measured by each of the collaborators. Differences may be due in part to spatial inhomogeneity of reinforcement or to measurement procedures.

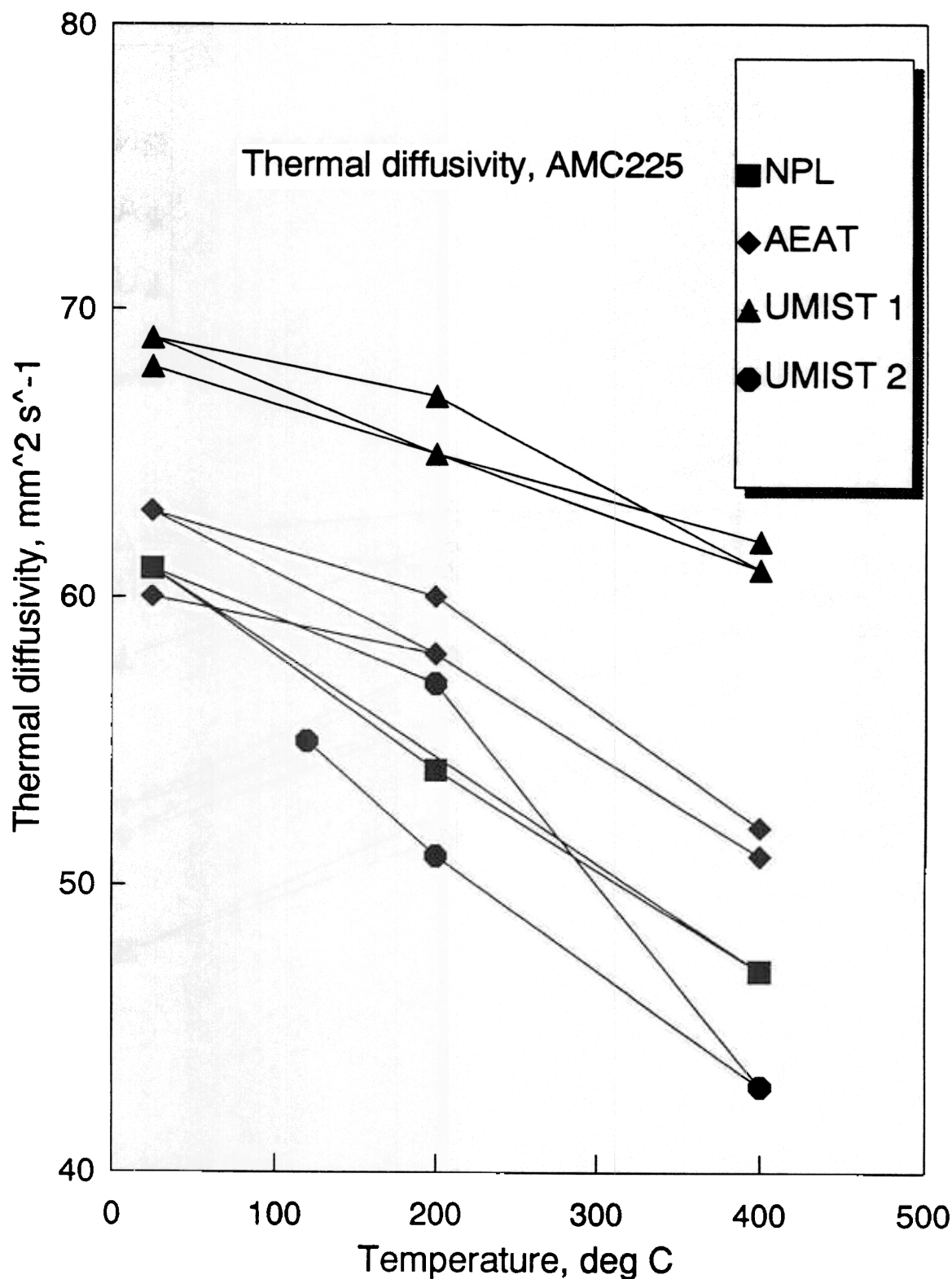


Figure 2 Thermal diffusivity of AMC225 MMC as measured by each of the collaborators. Repeat measurements by UMIST are more closely aligned to the other results than the first set.

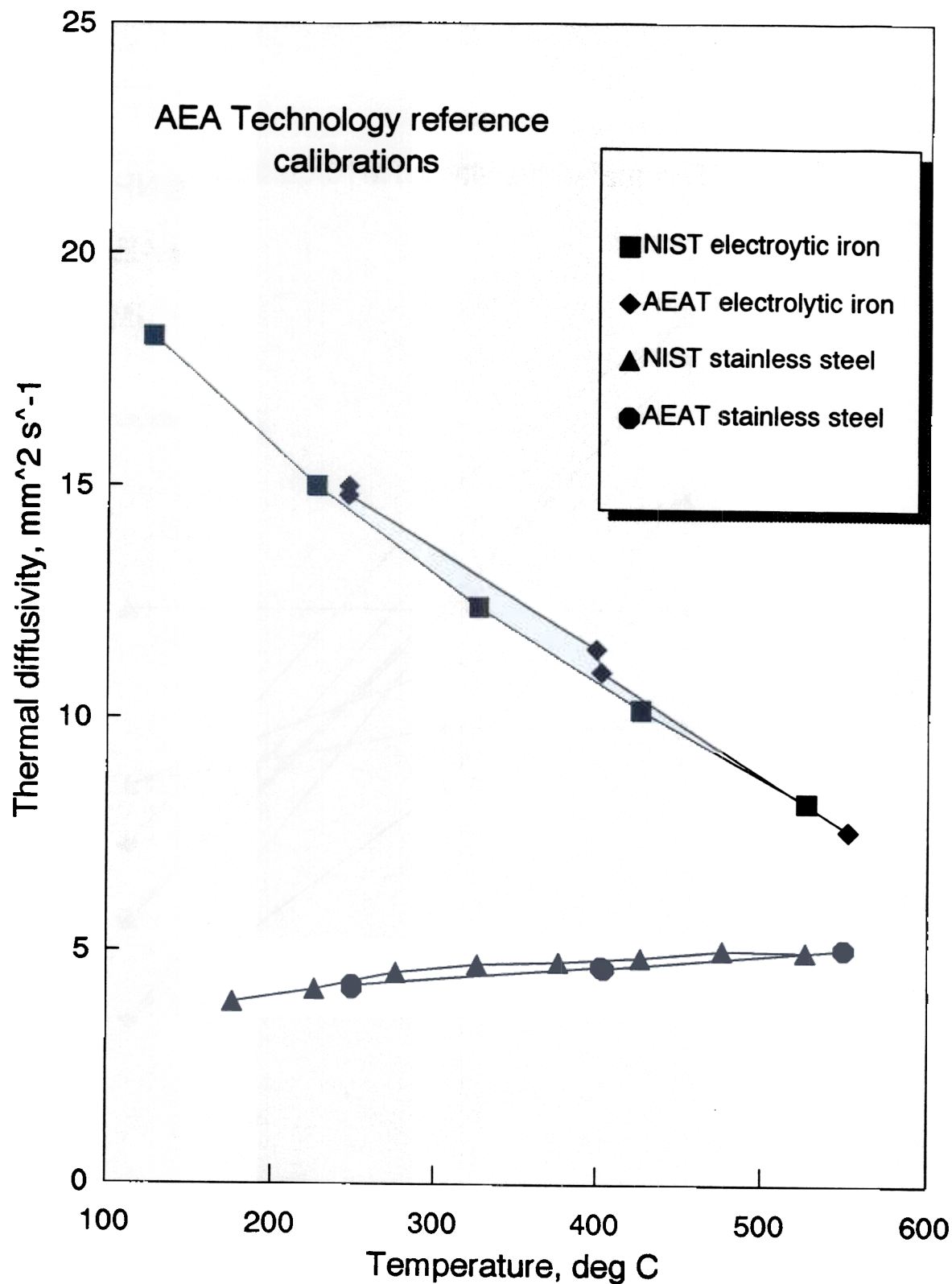


Figure 3 Recent AEA Technology data on NIST thermal conductivity reference materials (reference data converted to thermal diffusivity) showing a close match of experimental to reference data.

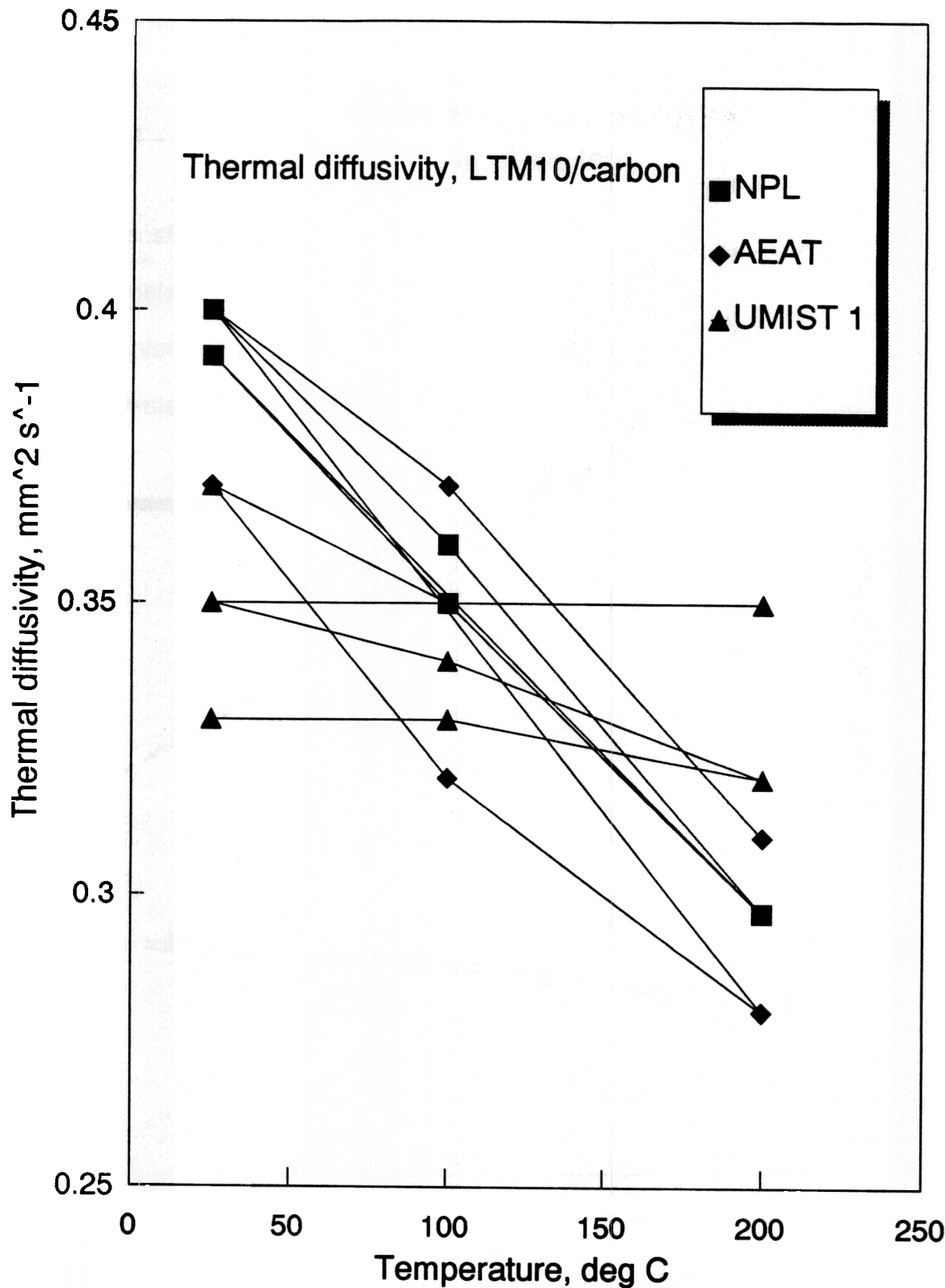


Figure 4 Thermal diffusivity of LTM10/carbon through thickness. NPL and UMIST data were obtained by laser flash/i.r. detector, and AEAT data by xenon flash lamp/thermocouple.

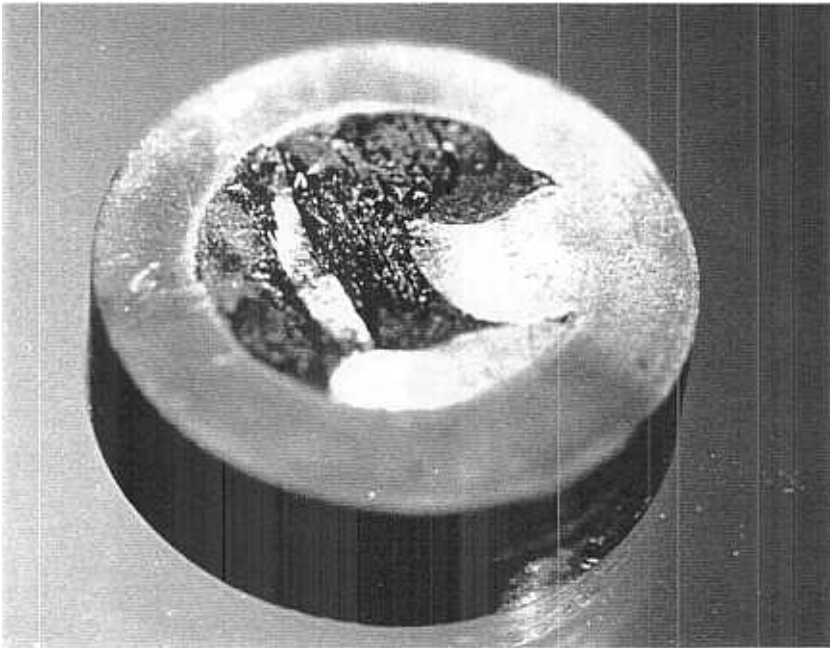


Figure 5 Damage to UMIST sample of LTM10/carbon by three laser pulse, resulting in inability to make consistent measurements. Note that a restricting annulus was placed over the test-piece so that the effects of laser irradiation could be more clearly seen.

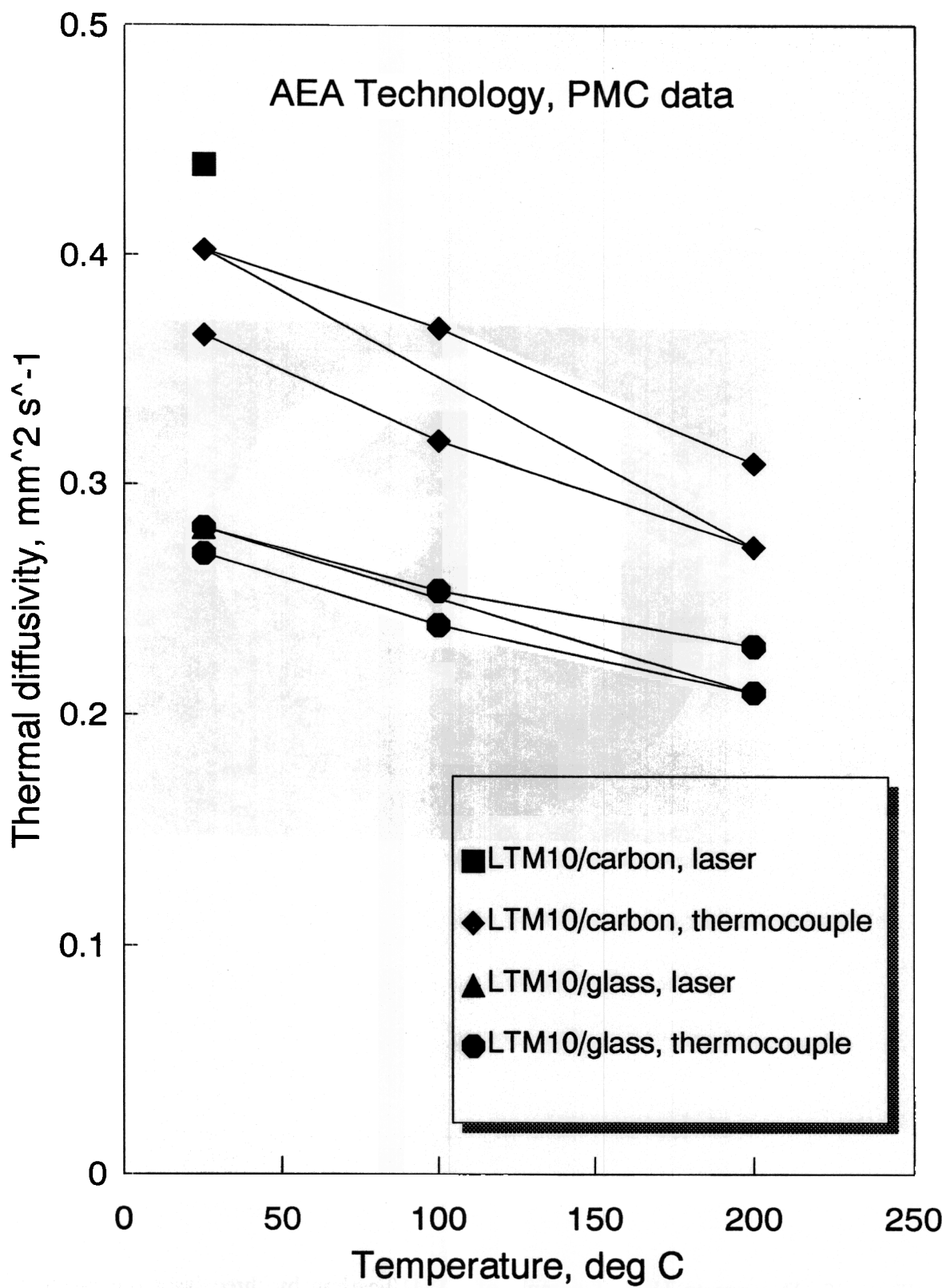


Figure 6 Thermal diffusivity of LTM10/carbon and LTM10/glass by AEAT comparing laser and xenon lamp flash methods.

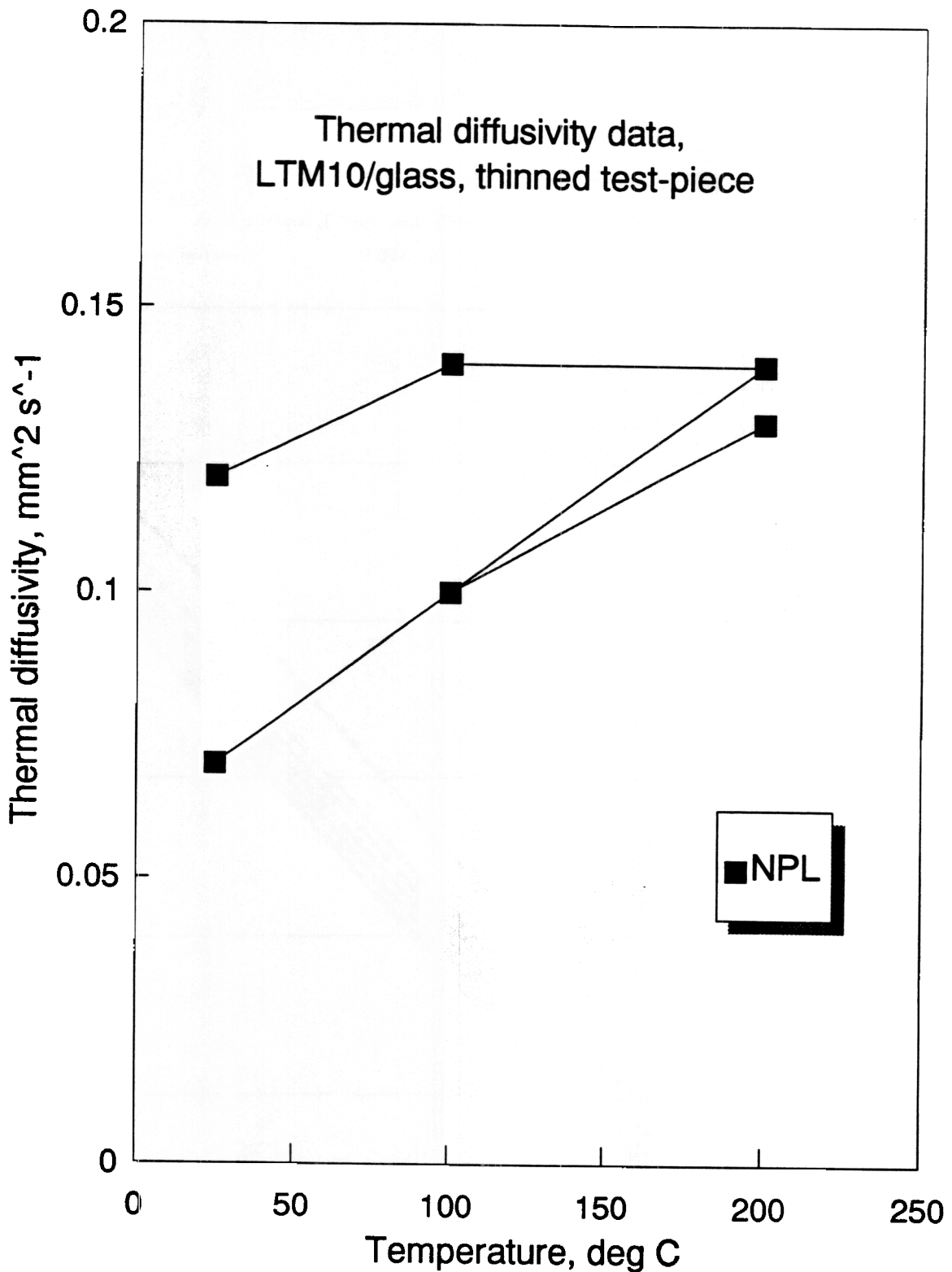


Figure 7 Thermal diffusivity results for LTM10/glass obtained by NPL by the laser flash method after thinning the test-piece to 1.3 mm from the initial 3.2 mm. These data are significantly lower than the full thickness data from AEAT obtained by xenon flash lamp.

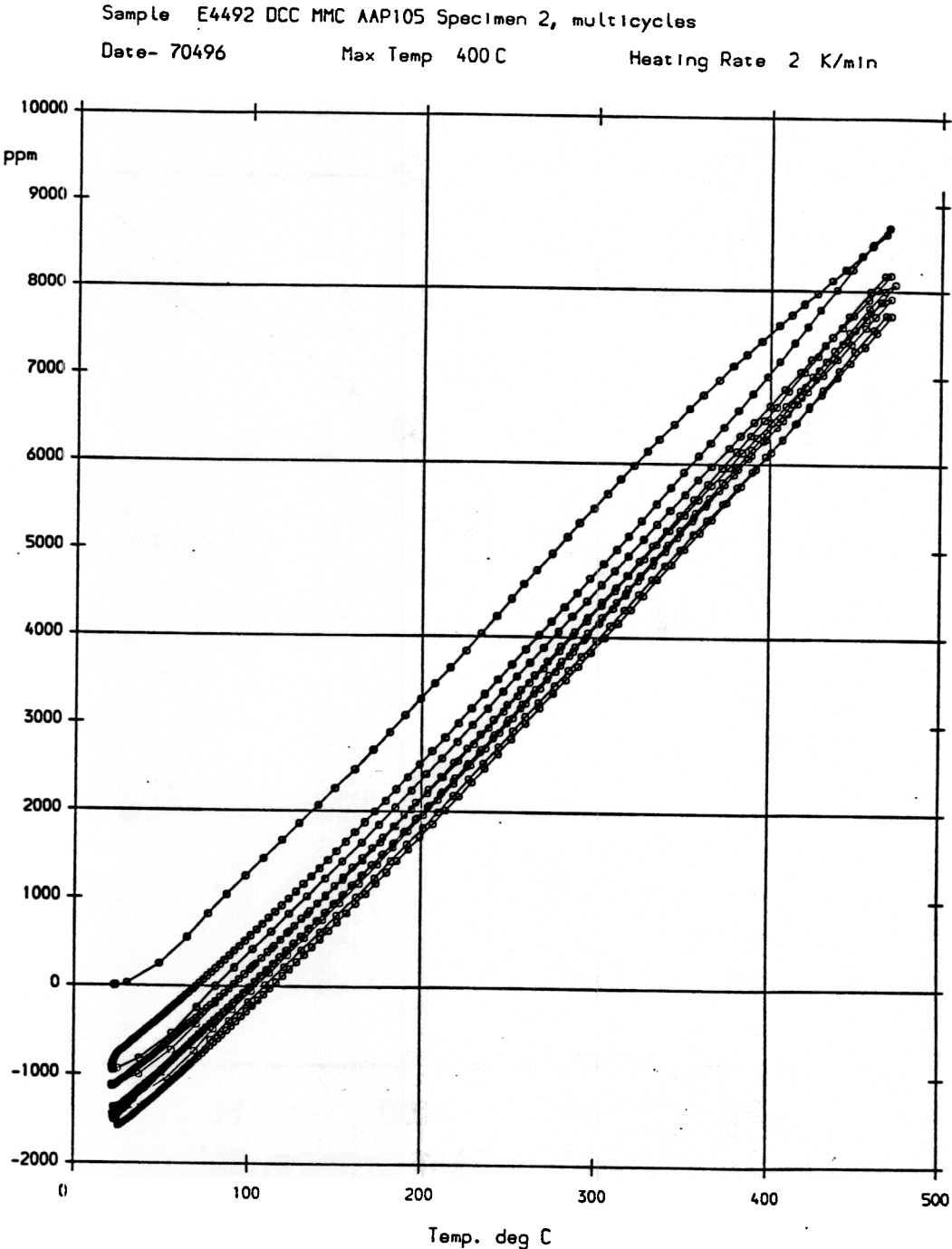


Figure 8 Thermal expansion behaviour of W6A20A (AAP105) in multiple cycles (NPL results).

Thermal Linear Expansion of Sample AAP105

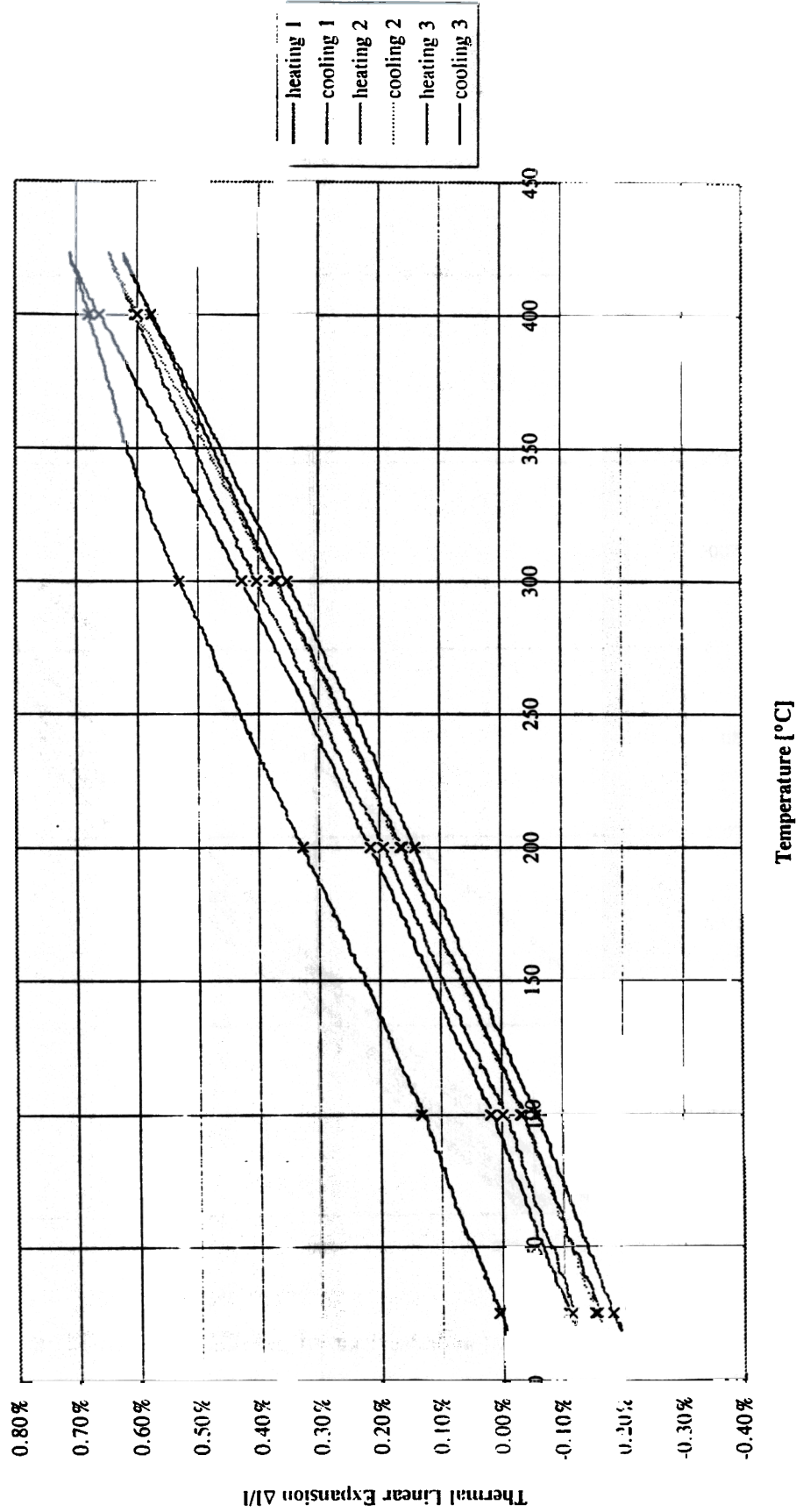


Figure 9 Thermal expansion behaviour of W6A20A (AAP105) in multiple cycles (UMIST results).

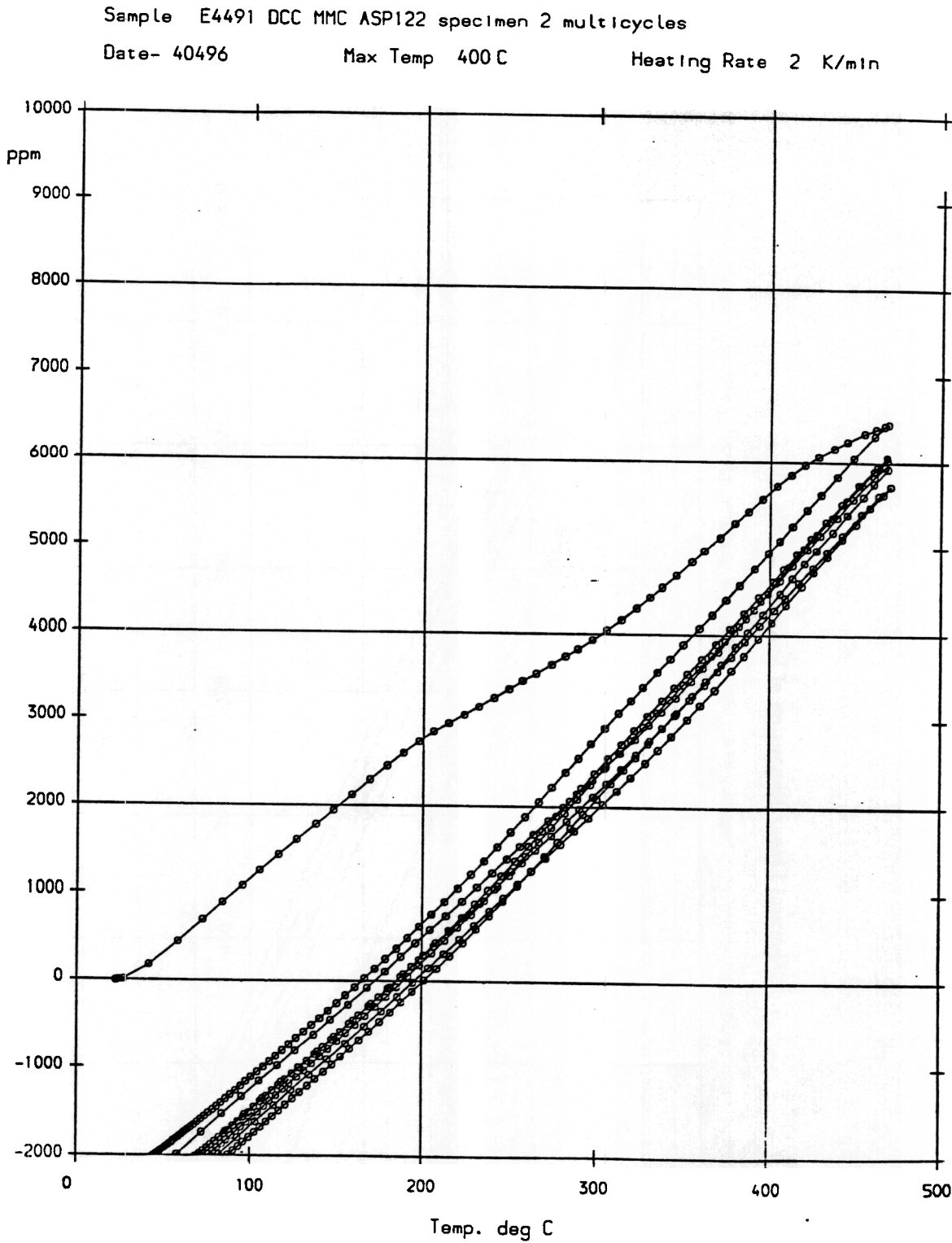


Figure 10 Thermal expansion behaviour of AMC225 (ASP122) in multiple cycles (NPL results).

Thermal Linear Expansion of Sample ASP122

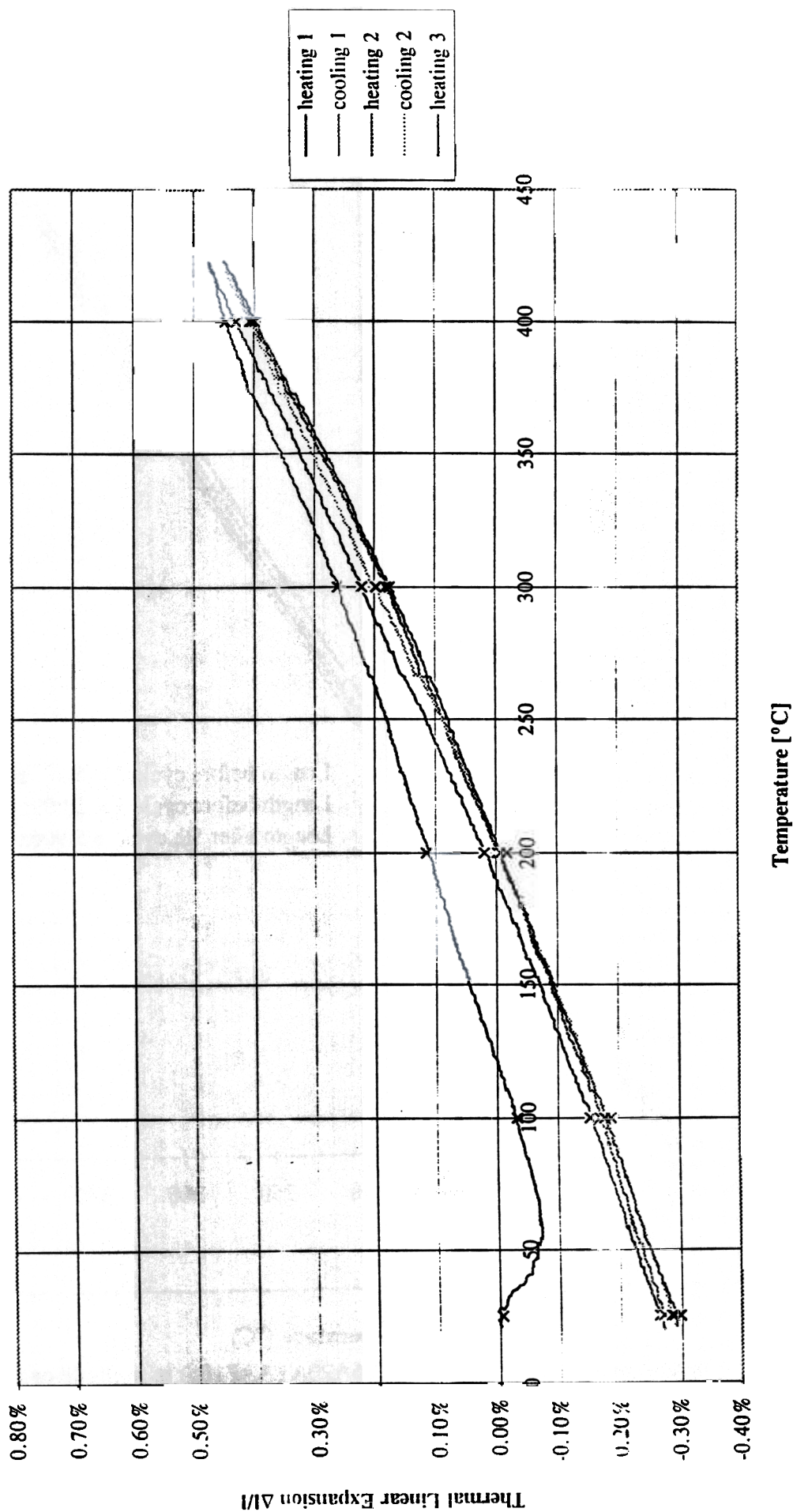


Figure 11 Thermal expansion behaviour of AMC225 (ASP122) in multiple cycles (UMIST results).

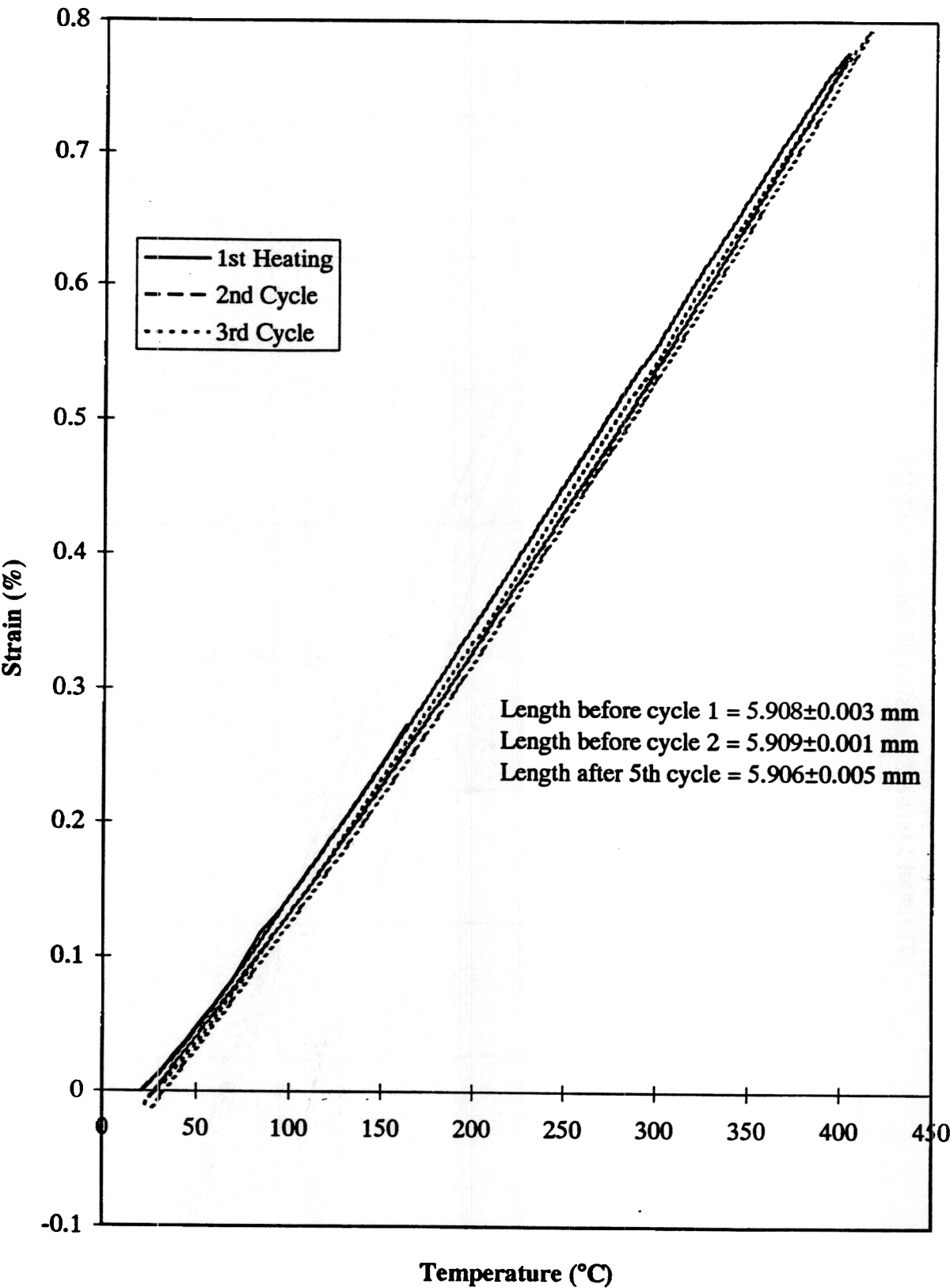


Figure 12 Thermal expansion behaviour of W6A20A (AAP105) in multiple cycles using the interferometric method (AEAT results).

Thermal Linear Expansion of Sample CFS003

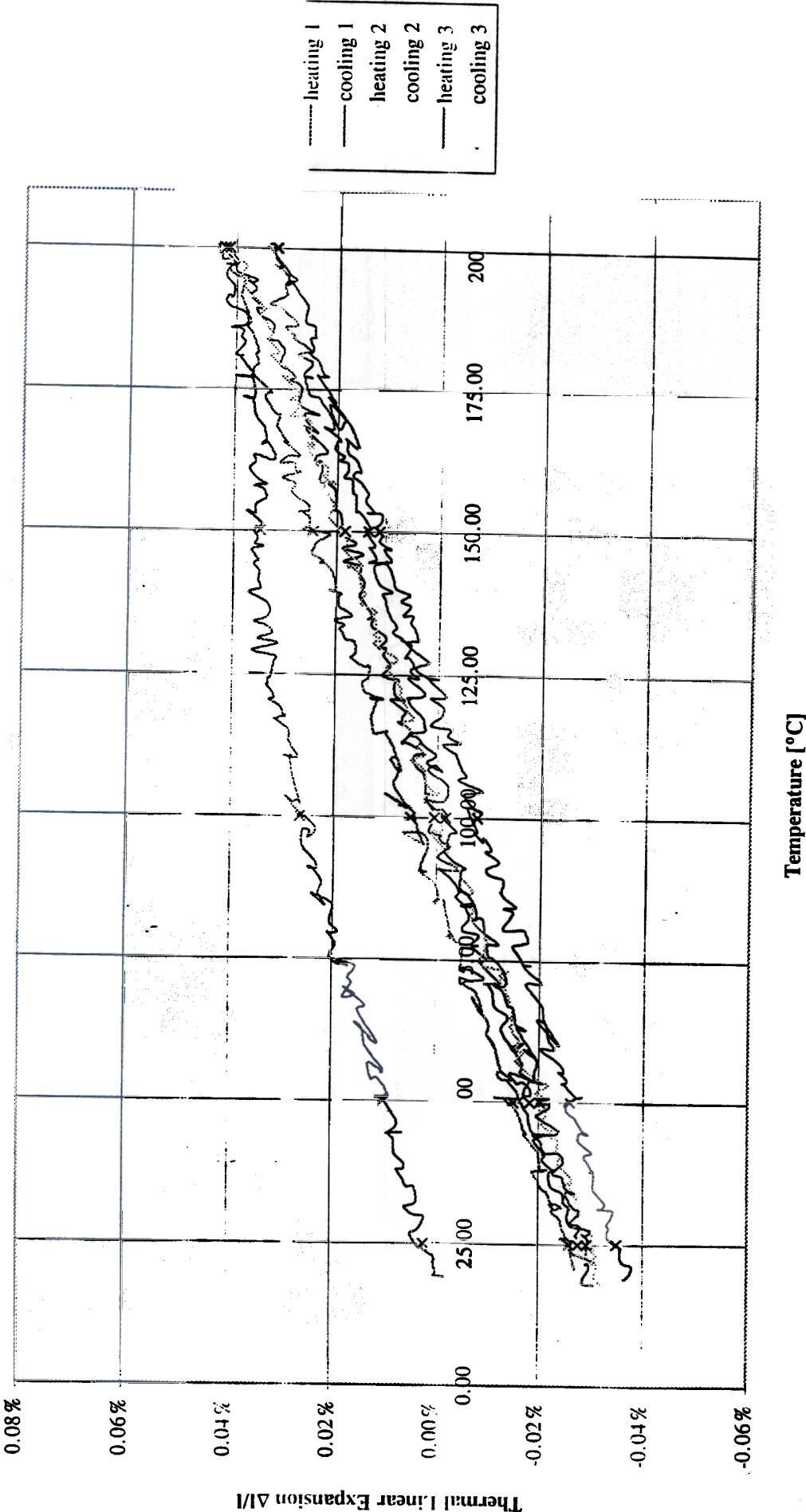


Figure 13 In-plane thermal expansion behaviour of LTM10/carbon in multiple cycles (UMIST results).

Thermal Linear Expansion of Sample KNC

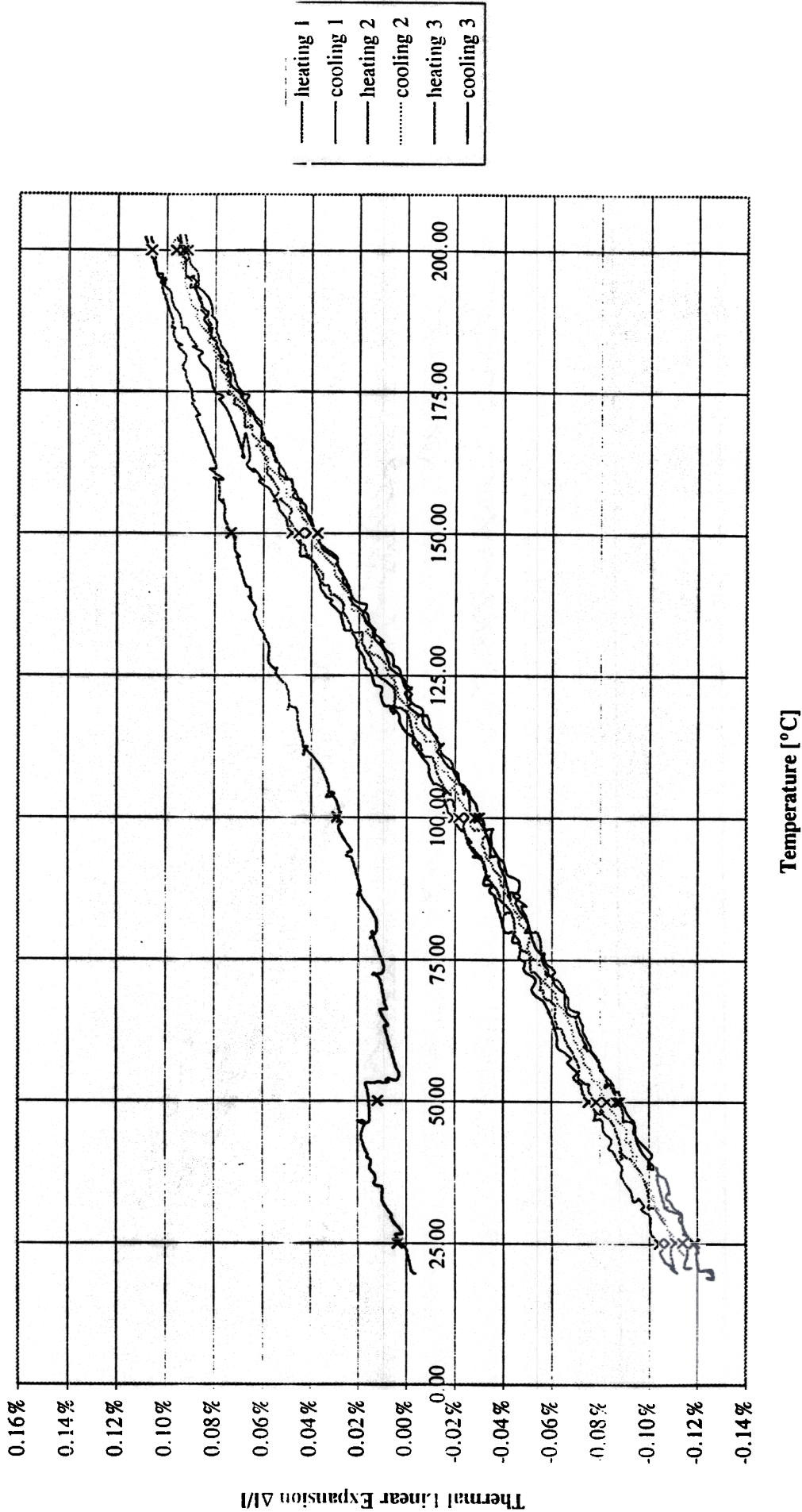


Figure 14 In-plane thermal expansion behaviour of LTM10/glass in multiple cycles (UMIST results).

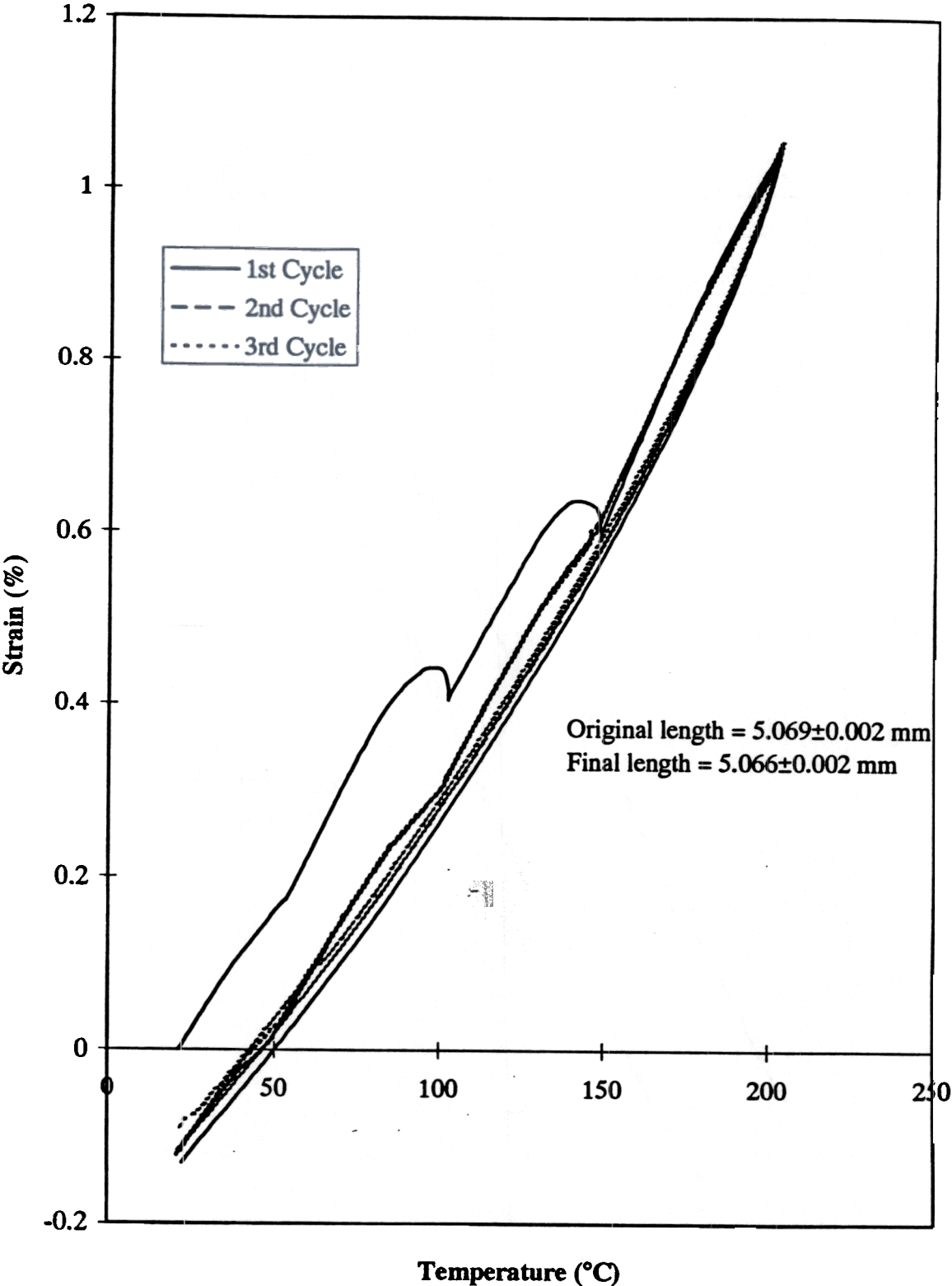


Figure 15 Through-thickness thermal expansion behaviour of LTM10/carbon in multiple cycles (AEAT results).

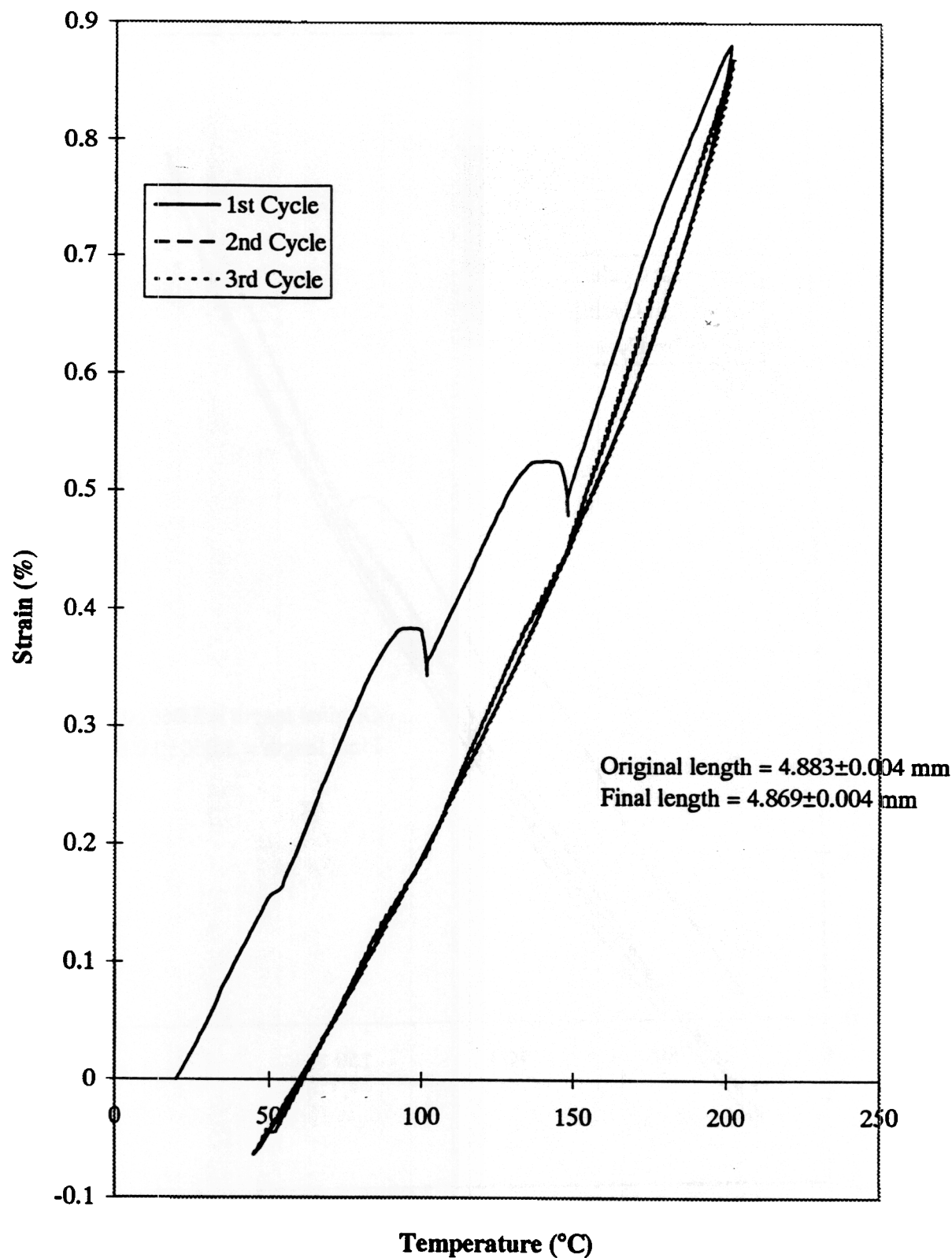


Figure 16 Through-thickness thermal expansion behaviour of LTM10/glass in multiple cycles (AEAT results).