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Effect of microwave post-curing upon the micromechanics of model Kevlar/epoxy composites

Abstract
Microwave processing of materials has the potential to deliver several major advantages over conventional thermal processing. One of these is an decrease in the time necessary for manufacture since the microwave energy is absorbed throughout the body of the material rather than relying on thermal conduction and convection. Another potential advantage is that the power is directed to the sample, this together with the decrease in processing time leads to lower energy being consumed. One question which needs to be addressed in the case of polymer composites is whether microwave processed materials are of as good quality as the thermally processed ones. In this work the interfacial properties of model Kevlar fibre reinforced epoxy composites post-cured by both conventional and microwave heating have been examined. Raman spectroscopy was employed to measure the fibre strain distributions along embedded fibres and from this information the interfacial shear stress distribution was calculated. The results show that the interfacial shear strengths and critical lengths of the microwave post-cured composites are comparable to those for thermally post-cured ones. This is potentially of interest in the commercial manufacture of composites since the process could be considerably shortened by the use of microwave post-curing leading to lower cycle times and costs without any deterioration in the interfacial properties of the composites.

Keywords
Composite material, Epoxy resin, Fibre reinforced material, Synthetic fibre, Aramid fibre, Manufacturing, Curing(plastics), Microwave heating, Property processing, relationship Temperature effect Interface properties, Matrix fibre interface, Shear strength, Stress strain, relation Measurement method, Raman spectrometry, Experimental study, Mechanical properties

Disciplines
Applied Mechanics | Engineering | Mechanics of Materials | Organic Chemistry | Polymer and Organic Materials | Polymer Chemistry | Structural Materials

Comments
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Effect of microwave post-curing upon the micromechanics of model Kevlar/epoxy composites

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Abstract

Microwave processing of materials has the potential to deliver several major advantages over conventional thermal processing. One of these is an decrease in the time necessary for manufacture since the microwave energy is absorbed throughout the body of the material rather than relying on thermal conduction and convection. Another potential advantage is that the power is directed to the sample, this together with the decrease in processing time leads to lower energy being consumed. One question which needs to be addressed in the case of polymer composites is whether microwave processed materials are of as good quality as the thermally processed ones. In this work the interfacial properties of model Kevlar fibre reinforced epoxy composites post-cured by both conventional and microwave heating have been examined. Raman spectroscopy was employed to measure the fibre strain distributions along embedded fibres and from this information the interfacial shear stress distribution was calculated. The results show that the interfacial shear strengths and critical lengths of the microwave post-cured composites are comparable to those for thermally post-cured ones. This is potentially of interest in the commercial manufacture of composites since the process could be considerably shortened by the use of microwave post-curing

leading to lower cycle times and costs without any deterioration in the interfacial properties of the composites.

Introduction

Whilst the use of microwave curing is well known both in industry and academia for ceramics, microwave heating is not used widely for polymer composites. The benefits of microwave processing have already been seen in the home where microwave ovens are commonly used to cook food faster than conventional convection ovens. Clearly if this approach could be used for processing composites then there are potential benefits in terms of speed of cure and hence reduced cycle times. It would also require less energy to cure and this, together with the reduced cycle time would lead to lower costs. This would be a significant advance in composites processing.

There has been considerable interest in the use of microwave curing for epoxy based composites (see for example references 1 to 9). One advantage is that the sample is penetrated by the microwaves so that volumetric heating occurs rather than conduction and convection which have associated with them time delays [7-10]. In general during microwave heating the applied power rather than the temperature is kept constant. There are three approaches which can be used for microwave processing. These are waveguides [9], tuneable single mode resonant cavities [2,10] and multimode ovens. The later are the most readily available source but have the disadvantage that they do not, in general, have a uniform field [11] and there are, therefore, hot spots. In the work described here a conventional, multimode oven was used in order to post cure the epoxy matrix because of their ready availability and low cost. Such ovens are thus the most accessible form of microwave heating.

Account was taken of the presence of areas of high field within the oven so that the samples were exposed to a relatively uniform power.

The curing of epoxy resins involves a number of reactions which depend upon the curing system used [12]. These can be complex and so it is difficult to predict the kinetics. There is some dispute in the literature on the effect of microwave heating upon the reaction rate for epoxies [5,6,11]. The effect of microwave heating on these reactions has been reviewed by Jacob, Chia and Boey [13]. The rate of cross-linking in an amine cured epoxy system has been observed by Mijovic and Wijaya to be higher in microwave cured samples than in conventionally thermal cured ones [14]. There are two possible explanations for this outcome. One is that since the reacting groups are polar they are selectively activated by the microwaves. In contrast during thermal curing the whole molecule must be heated before the reaction takes place. Another explanation might lie in the reactivities of the amino groups. Normally the primary amino group would be more reactive than the secondary because of steric hindrance. Under microwave heating the reactivities of both are similar and so this would lead to a higher rate of cross-linking[5].

It has been observed (for example by Bai and Djafari [15]) that microwave cured composites can have larger void contents than conventionally prepared materials. This is probably due to the fact that most workers using microwave heating do not apply pressure to consolidate the composite during the curing process. It might be envisaged that the presence of water may affect the curing and final properties of the resin. Experiments to follow the glass transition temperature, T_g, during curing of an epoxy containing 1 and 2% moisture have been performed by Boey[3]. The higher water content decreased the rate of increase of the Tg. This contrasts with the expectation that the high dielectric constant of the water would

lead to a faster increase in the temperature of the resin and thus speed up the cure. The presence of a higher level of moisture in the resin led to a greater void content in the composites[3]. Work has been performed to determine the mechanical properties of microwave compared to thermally cured materials [7,8,15,16]. These have shown that the microwave cured composites have mechanical properties which are as good as or better than the same composite conventionally cured.

Some investigations have been undertaken on the interfacial properties of microwave cured composites. Bai and Djafari[15] have used transverse tension and four-point bending tests to examine the interfacial properties of high volume fraction (c. 75%) glass/epoxy composites. They found that the interfacial properties of their microwave and conventionally cured materials were almost identical. Yue [7,16] used pull-out tests and compression of double lap shear specimens to obtain interfacial shear strengths. The microwave-cured samples had much higher shear strengths as measured by the lap shear tests and slightly lower shear strengths, as measured by the single-fibre pull-out test, than conventionally cured materials. The results quoted for the latter were, however, literature values.

In recent years Raman spectroscopy has been shown to be a powerful technique for examining the interfaces in a range of model and high volume fraction composites with transparent matrices [17-21]. This technique does not rely upon assumptions being made as regards the state of stress in the interfacial region. Day and Samoladas[22] have used this technique to explore the micromechanics of model carbon epoxy composites which have been post-cured either conventionally or by the use of microwaves. The results of this work show that there is little difference in the interfacial properties of the model carbon/epoxy composites which have been thermally and those which have been microwave post-cured. The

duration of the post-cure was decreased, however, from several hours to minutes. This is potentially a considerable benefit in the industrial manufacture of composites.

In order to investigate the interface in a Kevlar/epoxy system, model composites containing short single fibres have been fabricated in this present study using the same epoxy resin (Ciba LY5052/HY5052) as the previous study using this approach[22]. These were post-cured using microwaves or a conventional convection oven. Raman spectroscopy was used to measure the strain along the fibre in these composites and from this data the critical lengths could be found and the interfacial shear stress distributions calculated.

Experimental

Sample preparation

Single short fibre model composites suitable for examination by Raman spectroscopy were prepared. These consisted of a single fibre of Kevlar 49, approximately 3mm long, in an epoxy matrix. The fibres had a poly (vinyl alcohol) surface treatment. A Ciba Geigy epoxy resin system was used. This consisted of a resin (Araldite LY 5052) with a hardener (HY 5052). These were mixed in the recommended ratio of 100:38 parts by weight. The mixture was degassed in a vacuum oven for ten minutes in order to remove the air bubbles formed during the mixing process. The mixture was then removed, stirred gently to break the bubbles formed at the surface, and replaced in the vacuum oven for a further twenty minutes. The mixture was then poured into a mould which was half filled. This was allowed to gel and a single Kevlar fibre, approximately 3mm long, placed on it. The mould was then completely filled and then allowed to gel at room temperature. Samples of the resin without fibres were prepared by a similar technique. The latter samples were

then cut into strips of approximate size 10 X 1 cm and either thermally or microwave post-cured. These samples were used in order to determine the required cure time for the microwave and thermal curing. Thermal post-curing was carried out at 100°C for three hours or at 200°C for ten minutes using a conventional convection oven. Some samples were also allowed to cure at room temperature for one week.

Microwave post-curing was effected using a conventional multi-mode microwave oven (Moulinex Quickchef 1000QE). Hot spots were identified using a large sheet of gelled resin which was placed inside the oven. It could be readily seen that there was greater darkening of the resin at the centre of the oven and in rings out from the centre. Curing of the samples was achieved by placing four samples at circumferencial positions where the field had been found to be even. The 1kW microwave oven was more powerful than required and rapid degradation of the samples took place if used at full power. A beaker of chilled water (300ml at 1-2°C) was thus placed in the centre of the microwave to absorb some of the energy. There is the potential that this could raise the humidity of the environment in which the specimens were being cured, but this is unlikely to be an important factor since for most of the curing cycle the water would be too cold to evaporate rapidly. The samples were post-cured for 220 seconds in the microwave oven.

Some samples were prepared by dipping the Kevlar fibres in a solution of the mixed resin (5% by weight in ethanol) and then curing the resin coated fibre in the microwave oven. The coating on the fibres did not have a constant thickness and there were droplets of resin periodically along the fibre. The coated fibres were then also used to prepare model composites which were cured at room temperature for one week.

Extent of cure

The extent of cure for the thermally and microwave cured samples was measured using differential scanning calorimetry (DSC). From this information suitable cure times were determined. A Du Pont thermal analyst 2000 system was used. Samples with weights in the range 10 to 14 mg were cut into small pieces and placed in aluminium pans. The samples were heated at 20°C/min under a nitrogen atmosphere from 0 to 250°C.

Interfacial measurements

Measurements of the interfacial properties of the model composites were made using Raman spectroscopy [20]. The Raman spectra were taken using a modified Olympus optical microscope connected to a Spex 1000M single monochromator. A laser line rejection filter was used between the microscope and spectrometer. The spectra were excited using a 10 mW HeNe laser and were recorded using a Wright instruments charge coupled device (CCD) detector. The single fibre samples were routed into a dog-bone shape and strained using a minature tensile testing machine (Polymer Laboratories Minimat). The latter was fastened to the stage of the optical microscope allowing the specimens to be loaded whilst the Raman spectra were taken. The overall strain in the composite was measured using an electrical resistance strain gauge. Since comparatively large strains were being measured the resistance of the strain gauge was measured using a digital multimeter and the strain derived from the resistance. The model composites were loaded and Raman spectra were taken from positions along the fibre. The spectra were analysed by fitting a quadratic background and a Lorenzian function to each peak using the Levenberg-Marquardt approach [23]. The peak

frequency was related to the strain in the fibre using a calibration curve obtained for single fibres loaded in a small straining rig.

Results and discussion

Differential Scanning Calorimetry

The DSC trace for the gelled, but not post-cured, resin is shown in figure 1. It shows a glass transition at about 50 ^{o}C and a broad curing exotherm from about 80 to 225 ^{o}C. After some experimentation it was found that the curing exotherm could be removed by curing in a conventional oven for three hours at 100 ^{o}C, for ten minutes at 200 ^{o}C or in the microwave oven with the sample arrangement described earlier for 220 seconds. Figures 2 and 3 show the DSC traces for the resins post-cured under these conditions respectively. It is clear from figures that the Tg is much broader in the case of the microwave cured resin, but the maximum is at the same temperature for both, i.e. 145 ^{o}C. This agrees with the observations of other workers using different epoxy systems [14,24,25]. There have been some attempts to understand the cause of this broadening, but at the moment the origin is not clear. Mijovic and Wijaya [14] point out that the broader Tg range is indicative of a broader molecular weight distribution or morphological heterogeneity. The temperature variation across the volume of sample required for DSC during curing cannot be large and so this does not seem a likely ultimate reason for the broadening. The onset of the glass transition because of the broadening is at a lower temperature for the microwave post-cured samples. This would in practice give a lower limit to the temperature microwave cured epoxy resins could be used compared to thermally cured ones.

Interfacial measurements- calibration of change of Raman frequency with strain

There are six well-defined bands in the Raman spectrum of Kevlar fibres in the region 1100 to 1700 cm^{-1}. Most investigations centre on the strong band at 1610cm^{-1} and the decrease in the frequency of this band with increased strain is well documented [17-21,26-28]. A calibration curve of Raman frequency for the 1610cm^{-1} band against strain is shown in figure 4. Fibres with a variety of gauge lengths were measured but the rate of Raman band change with strain did not vary systematically with gauge length, although only samples with fairly long gauge lengths were used. An average value of Raman band shift with strain for the 1610cm^{-1} band of 4.1cm^{-1} was found from a linear regression fit to the data. This was then used to convert the Raman frequency as a function of position along the fibre data from the Raman spectroscopy measurements into strain distributions during testing of the model composites.

Interfacial measurements-strain distribution and critical lengths

The strain distributions in the fibres at a number of matrix strain levels were measured for model composites prepared under a variety of conditions. These are presented in figures 5 to 9. The strain distribution close to one end of the embedded fibre is shown. It can be seen in all cases that the critical length increases at larger matrix strains. This is because the resin yields at higher load levels[29] and the shear compliance will, therefore, increase leading to an increase in the critical length. In general the form of these strain distributions is much as would be expected from the Cox shear lag analysis [30] and is similar to those previously observed for this Kevlar / epoxy system [18,31]. In all of the samples there is a

compressive stress in the fibre with no externally applied load. This is, of course, due to resin shrinkage and to differential thermal contraction from the curing temperature. This is particularly evident in these samples because there is only one fibre in the matrix. The effect would not be so large in higher volume fraction samples. There is a limit to the residual compressive strains measured because Kevlar undergoes kinking at approximately 0.3% compressive strain[32]. The compression in the samples must be overcome before a tensile stress can be applied to the fibres. Due to this residual compression the strain in the plateau region of the fibre does not always correlate with the overall sample strain given by the strain gauge.

It is difficult to make comparisons between the critical lengths in the different model composites because the point at which the fibre strain reaches the plateau value is not clear because of scatter in the measurements. Figure 10 summarises the information regarding the critical length for different matrix strains and curing conditions. Since it is difficult to define the onset of the plateau in fibre strain the distance over which the strain in the fibre builds up to 95% of the plateau value is plotted. This shows some scatter but it is clear that no one curing scheme is significantly better than the others used in terms of the critical lengths found.

Interfacial measurements- shear stress distributions

It is well known that interfacial shear stress distributions can be calculated from the strain distribution data by making use of the relationship

$$\tau = E_f \frac{r}{2} \frac{\mathrm{d}e_f}{\mathrm{d}x}$$

where τ is the interfacial shear stress at a point a distance x from the end of the fibre, E_f is the fibre modulus, r is the fibre radius and $\mathrm{d}e_f/\mathrm{d}x$ is the slope of the

strain distribution curve at that point. The strain distribution data has, therefore, been fitted with a spline function and differentiated to give de_f/dx data. From this the interfacial shear stress data for each of the figures 5 to 9 has been obtained.

Figures 5 and 11 show the fibre strain and interfacial shear stress respectively for a sample which was allowed to post cure for seven days at room temperature prior to measurement. The initial strain distribution is very much as has been predicted [30] and previously observed[31]. The shear stress profiles show that the maximum interfacial shear stress is approximately 32 MPa. Upon loading shear yielding of the resin takes place and this is evident from the interfacial shear stress profile in figure 11 which shows that as the load on the sample increases a plateau of constant high shear stress develops at the end of the fibre. At the highest strain level used the entire region investigated has shear yielded.

Figures 6 and 12 show the fibre strain and interfacial shear stress measurements following post-curing of a sample at 100 ^{O}C. The strain plot (figure 6) shows that there is some residual compression in the fibre following the post curing treatment as might be expected. Figure 12 shows that the interfacial shear strength is higher in this post-cured sample than in the room temperature cured one (figure 11) and a maximum valve of approximately 46 MPa was obtained. This agrees well which previously reported values for this system[31]. The interfacial shear stress plot shows that the interface begins to yield at higher load levels because a plateau develops.

Data were obtained from a composite post-cured at 200 ^{o}C and this is shown in figures 7 and 13. The strain plot shows that there was residual compression in the fibre. The interfacial shear stress again shows signs of shear yielding at the higher load levels. This is evident at the 3% strain level were there is a plateau at

approximately 33 MPa. The maximum interfacial shear stress recorded was 38 MPa which is within the range expected for this matrix/fibre system[31].

Figures 8 and 14 show the strain distribution and shear stress distribution for a sample which was post-cured in a microwave oven as described above for 220 s. The strain plot shows residual compression in the fibre as with the thermally treated samples. The interfacial shear stress data is a little more noisy than for the thermally cured samples. It is still possible to observe that the interfacial shear strength is in the region of 45 MPa, i.e. in the same range as previously obtained for conventionally post-cured samples and that shear yielding is occurring because of the shape and magnitude of the interfacial shear stress profiles observed in figure 14.

Finally figures 9 and 15 show data obtained from a composite where the fibre was dipped into an ethanol solution containing the catalysed resin, the solvent was allowed to evaporate and the fibre heated in the microwave to cure the resin. Model composites were then made from these coated fibres and allowed to cure at room temperature for seven days prior to testing. The Interfacial shear strength data shows that the maximum shear stress recorded is significantly higher than observed previously with a maximum value of approximately 65 MPa being recorded. Shear yielding still appears to be present at higher strain levels

Table 1 gives a summary of the maximum interfacial shear strengths measured for model composites manufactured using different curing schemes. The interfacial shear strengths yielded by microwave curing of the resin are similar to those from conventional thermal curing. The values from the composites made with the coated fibre are higher than the others. The reason for this is not clear. Perhaps as the temperature generated within the curing resin in this case will be very high there will be greater shrinkage and thus higher radial stresses upon the fibre. A

further possibility is that the proportions of resin, hardener and reactive diluent have been altered during the coating process leading to different mechanical properties of the cured resin[31]. The mode of failure, shear yielding, is the same in the conventionally and microwave post-cured materials.

Conclusions

In this work the critical lengths of model composites containing single Kevlar fibres in an epoxy matrix have been measured using Raman spectroscopy. This has allowed the stress transfer characteristics of the interfaces in these composites to be examined. Some of the samples were made using conventional thermal post-curing whilst others were made either by post-curing in a multi-mode microwave oven. Some samples were made by coating the fibre in a solution of epoxy in ethanol, microwave curing the coating and then making single fibre model composites from these coated fibres. The critical lengths and interfacial shear strengths measured for the microwave cured composites are comparable to those from thermally cured model composites. Thus the large decrease in the curing time has not resulted in any deterioration of the interfacial properties of the model composites. There is no change in mechanism of failure. The glass transition, although the maximum is unchanged, is broader in the case of resins which have been microwave post-cured. Hence the onset is at a lower temperature than in conventionally cured resins. This leads to the only disadvantage of the microwave curing process which is that the maximum practical use temperature of the microwave cured resins will be slightly lower than the same resin thermally cured.

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Table 1. Interfacial shear strength of model composite samples

Sample	Interfacial shear strength /MPa
Room temperature cure	32
Three hours post cure at 100 ^{o}C	46
Ten minutes post cure at 200 ^{o}C	38
Microwave cured for 220 seconds	45
Fibre coated with resin, microwave cured then mounted in cold cured resin	65

Figures

Figure 1 - *DSC thermogram of epoxy resin with no post cure.*

Figure 2 - *DSC thermogram of epoxy resin post-cured for three hours at 100°C.*

Figure 3 - *DSC thermogram of epoxy resin post-cured in a microwave oven for 220 seconds.*

Figure 4- *Graph showing variation of Raman frequency for the 1610cm^{-1} band against strain.*

Figure 5- *Variation of fibre strain with distance along Kevlar 49 fibre in single-fibre composite at different levels of matrix strain. The sample was left to cure for seven days at room temperature*

Figure 6- *Variation of fibre strain with distance along Kevlar fibre in single-fibre composite at different levels of matrix strain. The samples was post-cured for three hours at 100 ^{o}C*

Figure 7- *Variation of fibre strain with distance along Kevlar fibre in single-fibre composite at different levels of matrix strain. The sample was post-cured for ten minutes at 200 ^{o}C*

Figure 8- *Variation of fibre strain with distance along Kevlar fibre in single-fibre composite at different levels of matrix strain. The sample was post-cured for 220 seconds in a microwave oven*

Figure 9 - *Variation of fibre strain with distance along surface coated Kevlar fibre in single-fibre composite at different levels of strain. The sample was made by dipping the fibre in a solution of catalysed resin in ethanol, drying, microwaving the coated fibre and then putting the fibre into epoxy resin and allowing the sample to cure for seven days at room temperature.*

Figure 10- *Plot of 0.95 of the distance along the embedded fibre to reach the plateau value of strain against the plateau value of fibre strain following curing by different methods.*

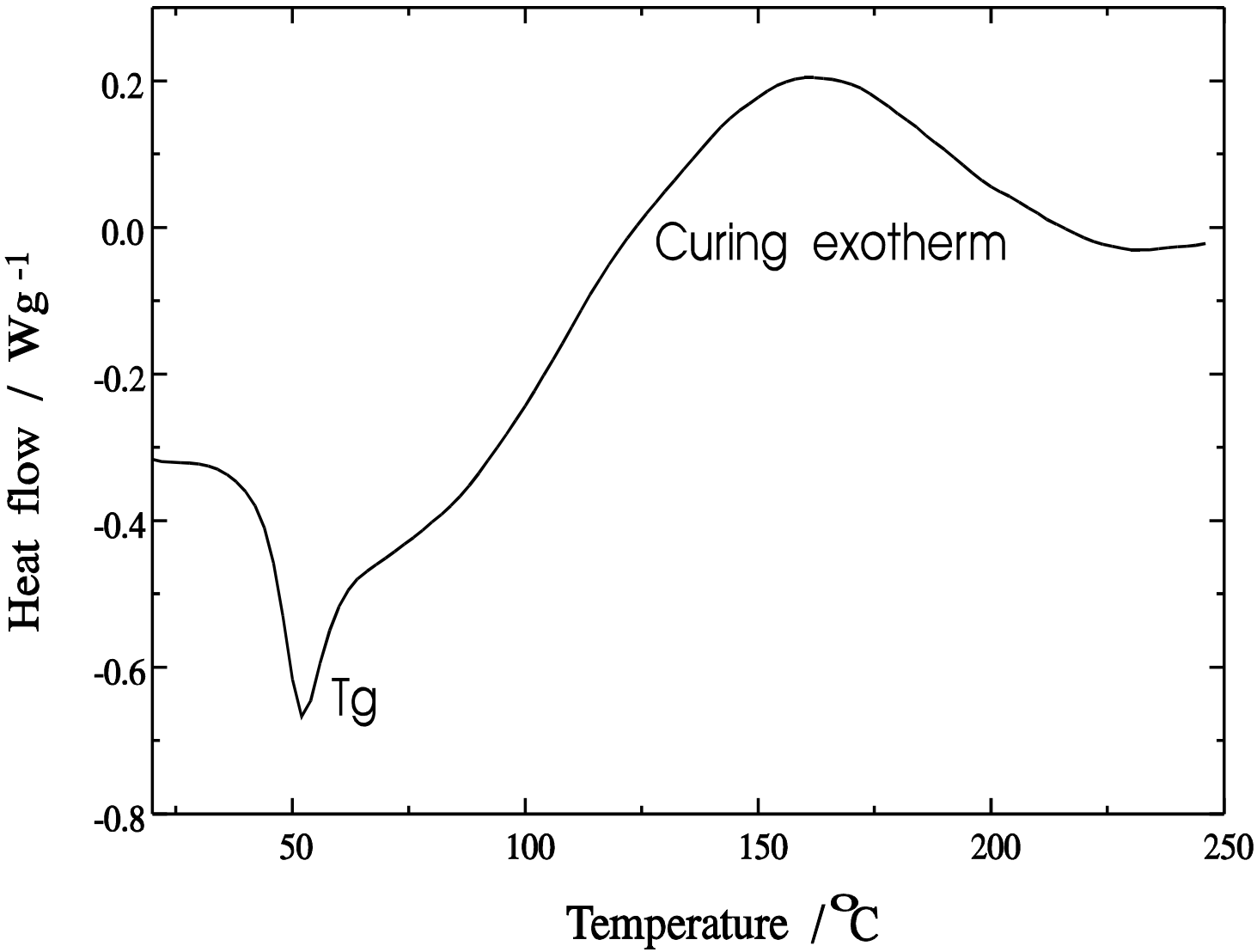
Figure 11- *Variation of interfacial shear stress with distance along Kevlar 49 fibre in single-fibre composite at different levels of matrix strain. The sample was left to cure for seven days at room temperature*

Figure 12- *Variation of interfacial shear stress with distance along Kevlar fibre in single-fibre composite at different levels of matrix strain. The samples was post-cured for three hours at 100 ^{o}C*

Figure 13- *Variation of interfacial shear stress with distance along Kevlar fibre in single-fibre composite at different levels of matrix strain. The sample was post-cured for ten minutes at 200 ^{o}C*

Figure 14- *Variation of interfacial shear stress with distance along Kevlar fibre in single-fibre composite at different levels of matrix strain. The sample was post-cured for 220 seconds in a microwave oven*

Figure 15- *Variation of interfacial shear stress with distance along surface coated Kevlar fibre in single-fibre composite at different levels of strain. The sample was made by dipping the fibre in a solution of catalysed resin in ethanol, drying, microwaving the coated fibre and then putting the fibre into epoxy resin and allowing the sample to cure for seven days at room temperature.*


0.2
0.0
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Heat flow / Wg-1
Curing exotherm
Tg
50
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250
Temperature /°C

Figure 1

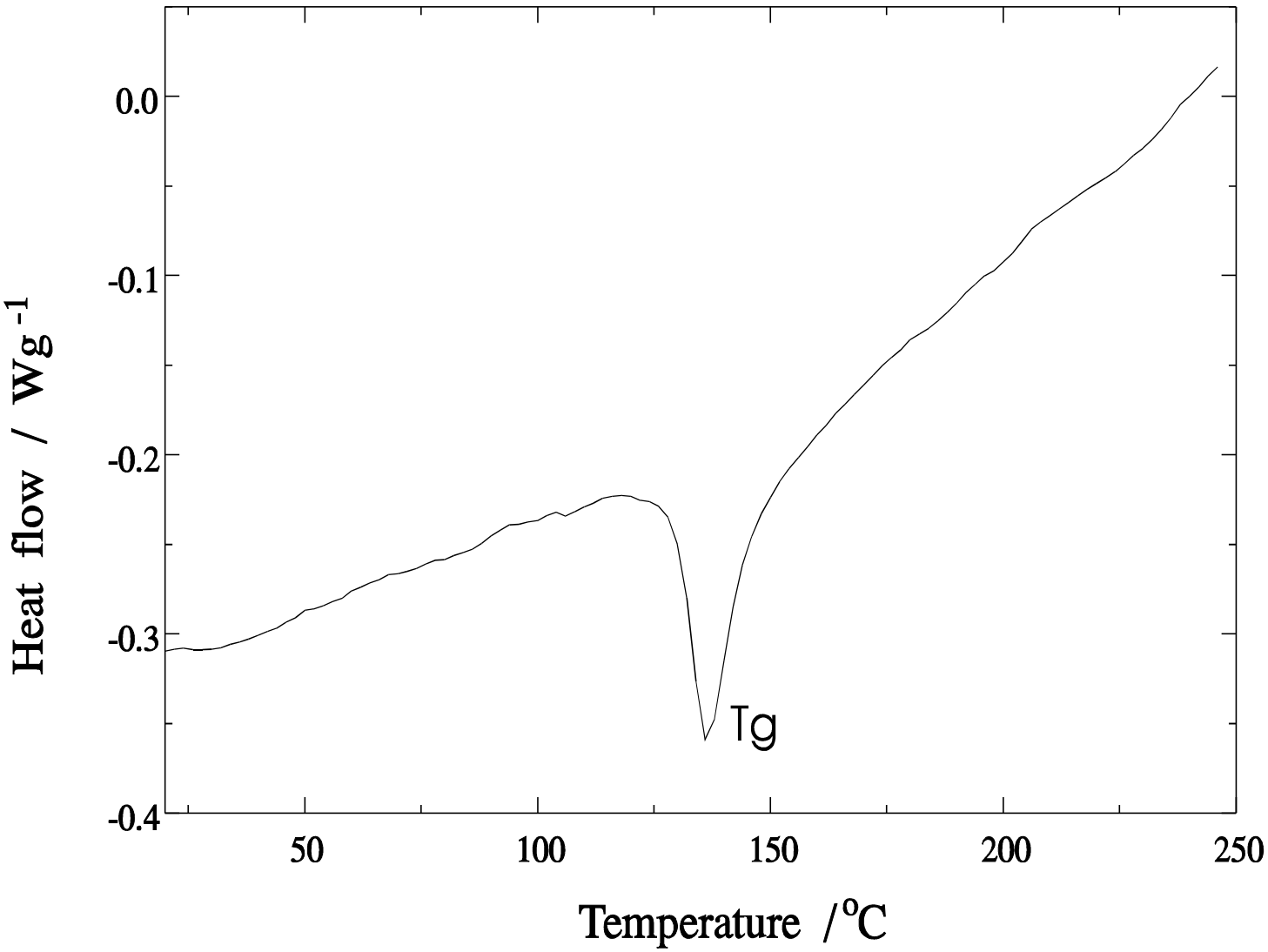
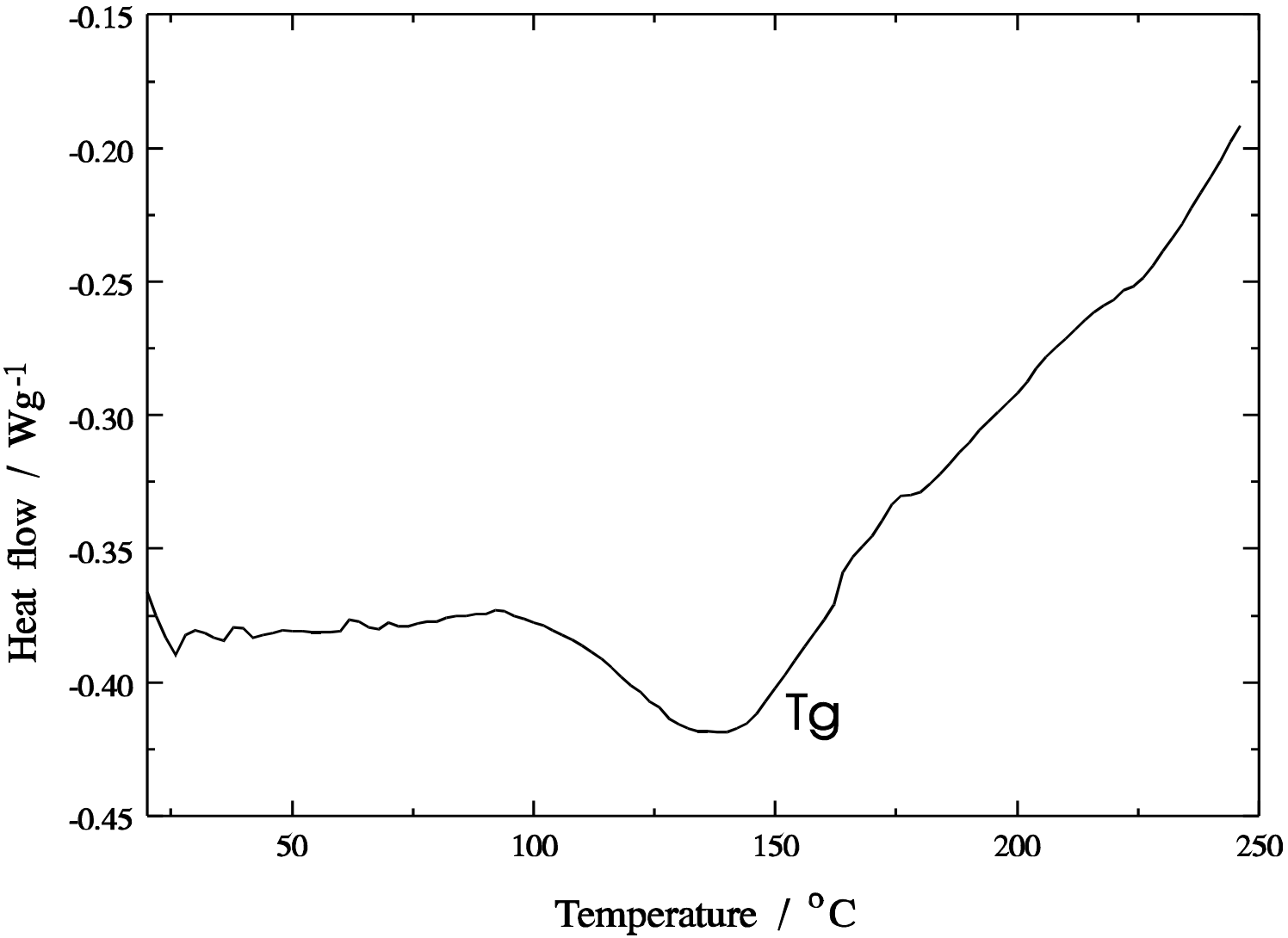


Figure 2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Heat flow / Wg-1
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Temperature / °C
Tg

Figure 3

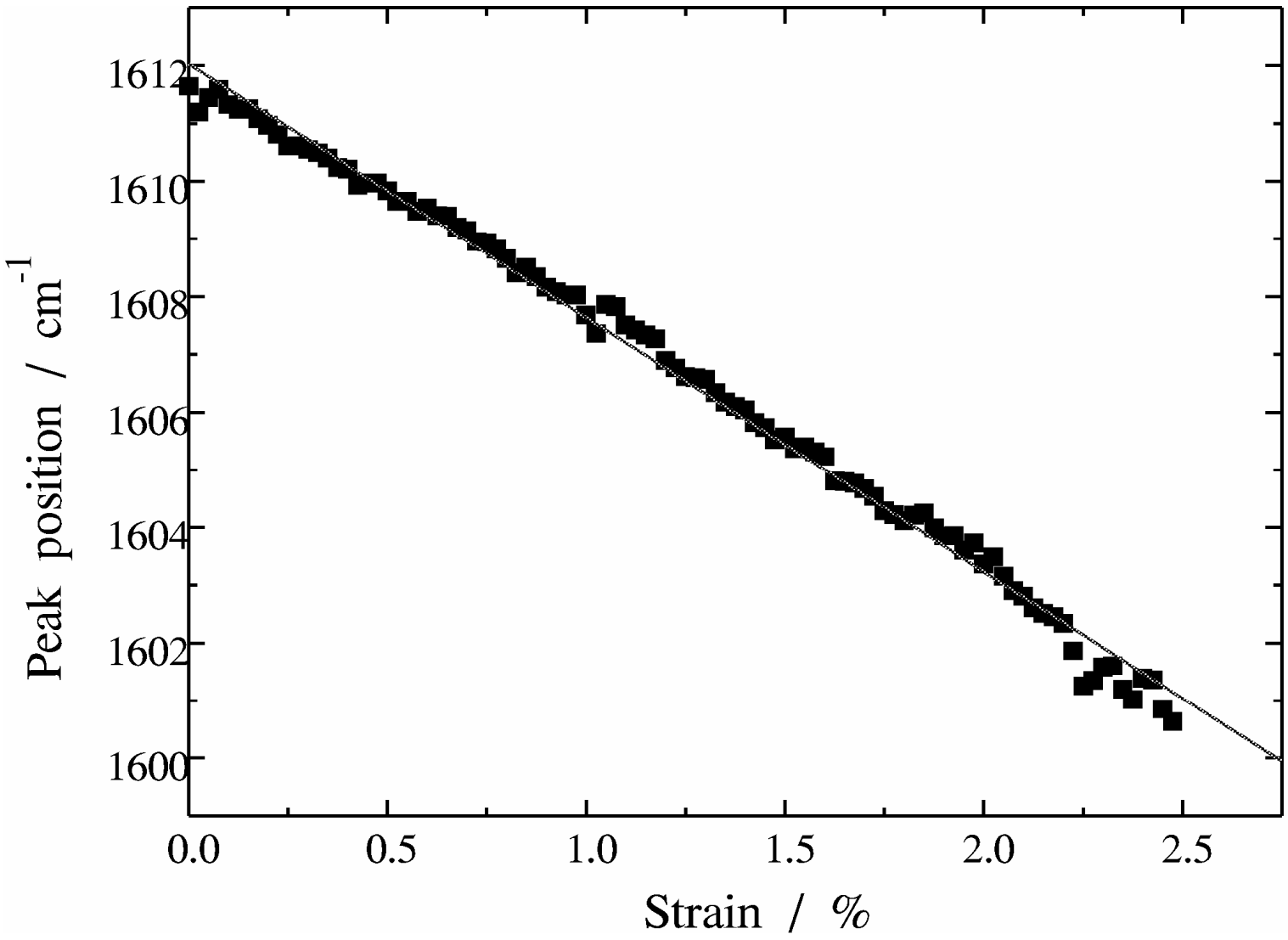
1612
1610
1608
1606
1604
1602
1600
Peak position / cm-1
0.0
0.5
1.0
1.5
2.0
2.5
Strain / %

Figure 4

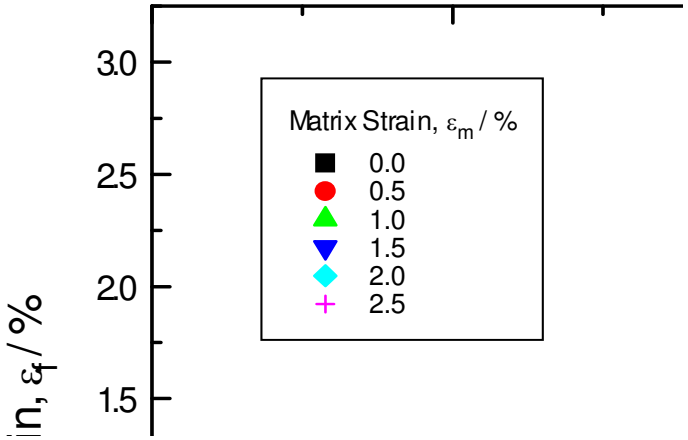

Figure 5

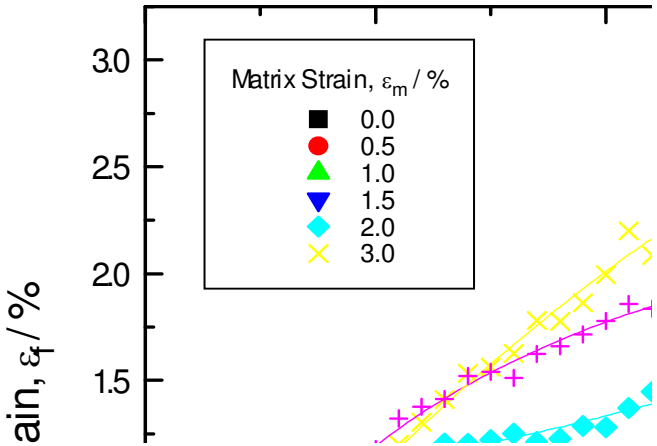
Matrix Strain, ε_m / %
0.0
0.5
1.0
1.5
2.0
3.0
3.0
2.5
2.0
1.5
ain, ε_f / %

Figure 6

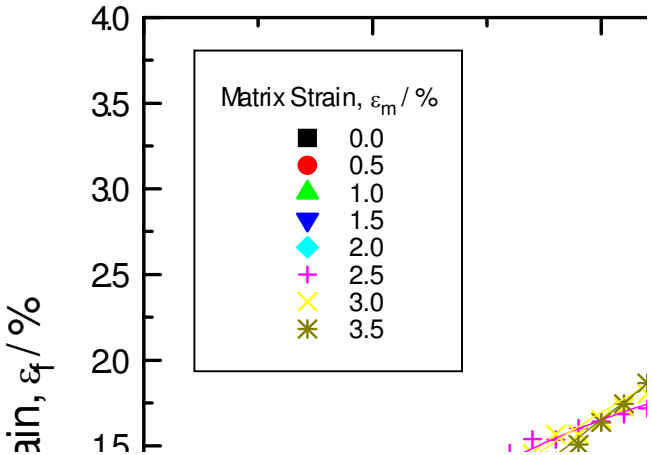

Figure 7

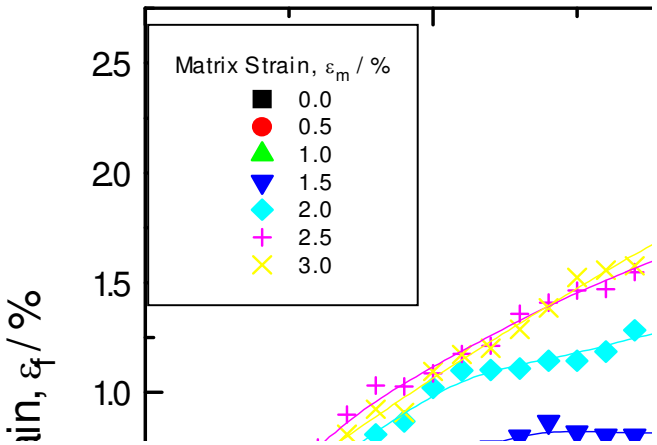
Matrix Strain, ε_m / %
0.0
0.5
1.0
1.5
2.0
2.5
3.0
2.5
2.0
1.5
1.0
in, ε_f / %

Figure 8

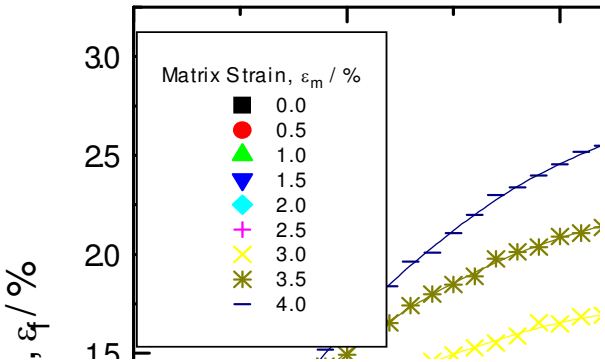
Matrix Strain, ε_m / %
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
3.0
2.5
2.0
1.5
ε_f / %

Figure 9

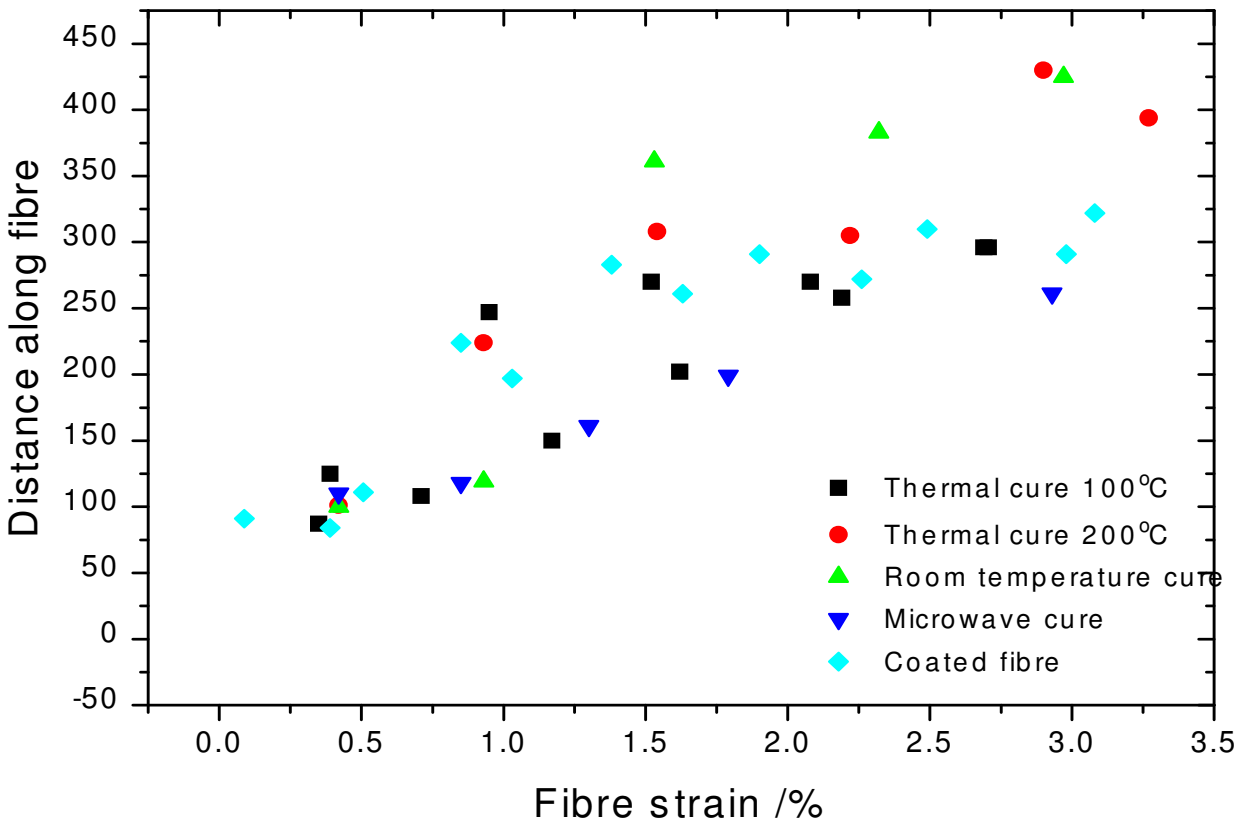
Distance along fibre
Fibre strain /%
450
400
350
300
250
200
150
100
50
0
-50
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
Thermal cure 100°C
Thermal cure 200°C
Room temperature cure
Microwave cure
Coated fibre

Figure 10

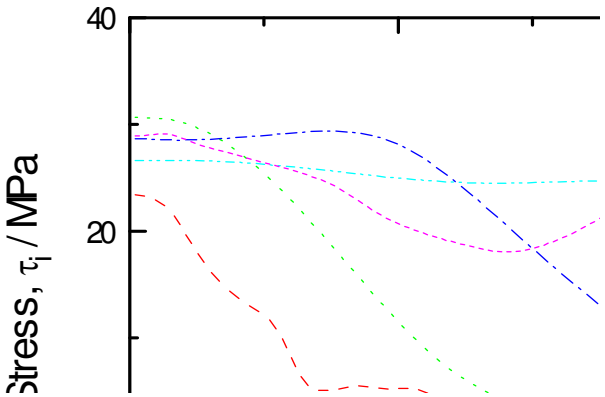

Figure 11

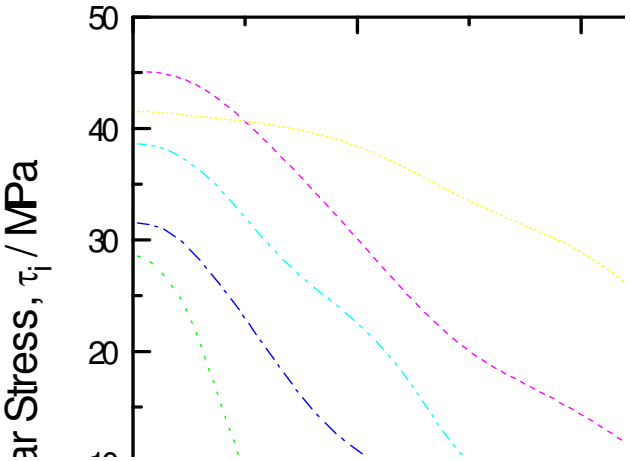

Figure 12

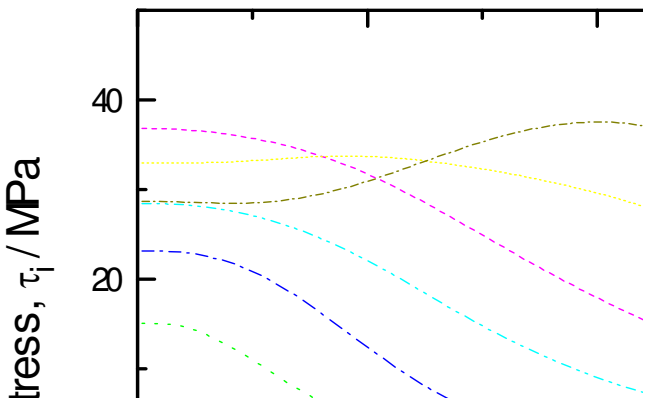

Figure 13

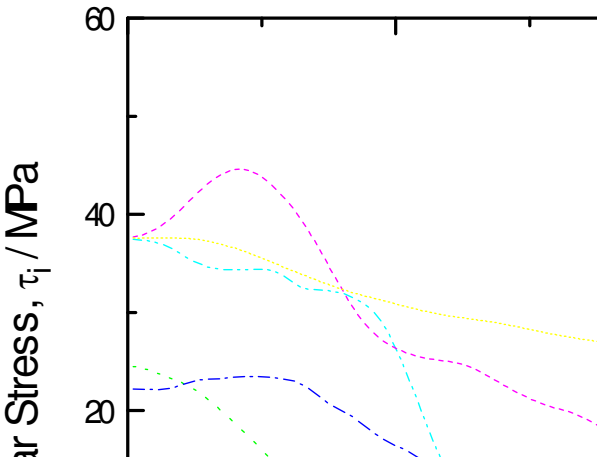

Figure 14

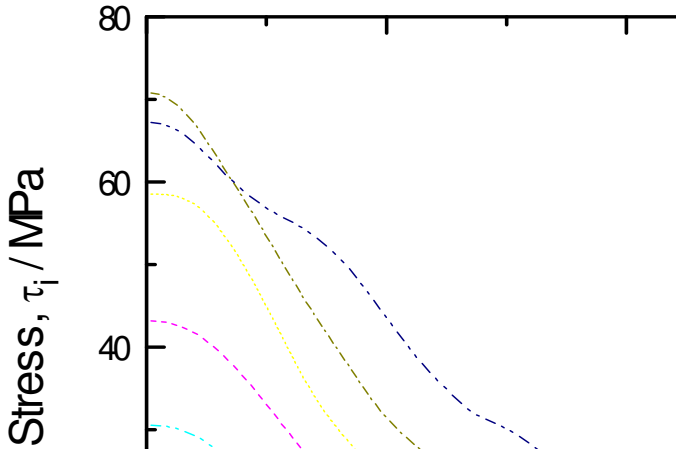

Figure 15