Relaxations in thermosets: 22. Curing kinetics and the dielectric properties of an elastomer containing epoxide cured with Ancamide

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The dielectric properties of homogeneous solutions of amine-terminated butadiene acrylonitrile (ATBN) elastomer, diglycidyl ether of bisphenol-A and Ancamide have been measured during their curing at 298 and 373 K. As curing progresses, the dielectric permittivity, ε', decreases and dielectric loss, ε'', first decreases and then peaks. The ε' and ε'' of the cured thermosets increase with increase in the elastomer content but decrease with increase in the temperature of cure for the same elastomer content. The rate of curing becomes slower with increased amount of ATBN in the thermoset. A prominent relaxation process in the cured thermoset is the α-relaxation of the elastomer, whose height increases with increase in the elastomer content. The broad shoulder to the low-temperature side of the peak is due to the γ- and β-relaxation processes of the epoxy. The dielectric properties of the thermosets are relatively insensitive to the inversion of the thermoset matrix to an elastomer matrix as the elastomer concentration is increased.

(Keywords: thermosets; curing kinetics; dielectric properties; elastomer)

INTRODUCTION

Rubber-toughened epoxies or thermosets are produced when an elastomer dissolved in the liquid components of a thermosetting solution phase separates as curing occurs[1–5]. The phase separation, which usually takes on the morphology of dispersed spheres of the elastomer in a rigid matrix, becomes a co-continuous or continuous morphology when the volume fraction of the dispersed elastomer exceeds a certain value. At a much higher volume fraction of the elastomer, the morphology is expected to become inverted, i.e. the elastomer becomes the matrix and the epoxy the dispersed phase.

As part of our investigations on the electrical properties of rubber-toughened thermosets[6–8], we have studied the curing kinetics and the dielectric and mechanical behaviour of amine-terminated butadiene acrylonitrile (ATBN) elastomer dispersed in an epoxide thermoset. The dielectric study showed that at the onset of phase separation during curing, the permittivity increased before decreasing monotonically to a value characteristic of an amorphous polymer. This was considered as a dielectric indication of phase separation of the elastomer when Maxwell–Wagner polarization dominated in a phase-separated mixture[6]. This paper is an extension of these investigations. It was prompted by a report by Yamanaka *et al.*[9] that curing of diglycidyl ether of bisphenol-A (DGEBA), containing the elastomer ATBN, by Versamid 125 (or equivalently Ancamide 260 A)* under certain conditions of composition, temperature and time produces varying morphologies of the dispersed elastomer phase. That is, curing at or above 373 K yielded a spherical domain structure with fairly uniform domain size and curing at 298 K yielded a co-continuous two-phase structure. We report here the dielectric monitoring of the cure of pure and ATBN-containing DGEBA–Ancamide 260 A thermosets, the properties of their cured states and an analysis of these results in terms of phase separation and its effect on the ultimate properties of the thermoset.

EXPERIMENTAL

Samples of thermosets were prepared by mixing at room temperature 1 mol of Ancamide 260 A with 2 mol of DGEBA and the required amount of amine-terminated butadiene in a 10 mm diameter glass vial. Commercial samples of DGEBA, under the name of EPON 828, and the curing agent Ancamide 260 A were donated by Shell Petroleum. EPON 828 has been carefully characterized by LeMay *et al.*[10] and its properties have been listed by Choy and Plazek[11]: $M_n = 380\ \mathrm{g\ mol^{-1}}$; glass transition temperature, $T_g = 259$ K; $f = 2.0$; and the soluble fraction of the resin when completely cured with diaminodiphenylsulphone is 0.3 w/w%. Here, M_n is the number average molecular weight and f is functionality. Ancamide

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* Ancamide 260 A is the US trade name for Versamid 125; both are available from Shell Petroleum

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Table 1 Dielectric properties of pure DGEBA–Ancamide thermoset and the ATBN-containing thermosets during their curing at two temperatures

ATBN in DGEBA (wt%)	ε' $(t\to 0)$	ε' $(t\to\infty)$	ε'' $(t\to 0)$	ε'' $(t\to\infty)$
	Curing at 298 K			
0	9.98	3.84	3.10	0.17
20	10.30	4.70	6.75	0.25
40	8.75	4.46	4.93	0.24
60	10.6	4.90	7.78	0.78
80	10.8	5.76	7.63	0.38
	Curing at 373 K			
0	5.43	3.62	–	–
20	7.61	4.52	–	–
40	7.32	5.05	–	–
60	7.45	4.98	–	–
80	9.15	6.59	–	–

260 A is an imide containing four NH groups. Its density and viscosity at 293 K are 0.96 g cm^{-3} and 40 Pa s, respectively. ATBN was purchased from B.F. Goodrich Chemical Company under the trade name Hycar ATBN 16. Its NH equivalent weight is 850 g, and it contains a residual amount (~3 wt%) of *N*-(2-aminoethyl)piperazine from its synthesis. Its T_g is ~223 K.

Each mixture (compositions given in *Table 1*) was mechanically mixed at room temperature when very fluid. A capacitor consisting of 10 rigid parallel plates was carefully immersed in the mixture contained in a glass vial (5 mm diameter) and the absence of any air bubble in the sample and between the plates was ensured. The glass vial was electrically shielded and transferred to a cavity drilled in a massive aluminium block whose temperature could be maintained within ± 0.1 K. A copper constantan thermocouple was immersed in the mixture with its junction held in contact with the ceramic spacer of the capacitor.

The capacitance of the parallel plate assembly was measured while in air prior to its immersion in the liquid mixture. It was nominally 16.5 pF and changed by <0.2% over the temperature range 77–400 K and its loss factor ($\tan\delta$) was $<10^{-5}$. The dielectric cell, which now consisted of a parallel-plate capacitor rigidly held inside the solution contained in a glass vial, was isothermally held inside a thermostatically controlled aluminium block at a predetermined temperature. Its capacitance, conductance and loss factor were measured during the cure at different time intervals up to a total period of $\sim 10^5$ s or until the capacitance and loss factor remained almost constant during the intervals of our measurements.

All measurements were made using a GenRad 1689 Digibridge, which was interfaced with a personal computer for automatic data acquisition. All connections between the parallel-plate capacitor and the Digibridge were made by coaxial cables. The measured capacitance is accurate to within 0.1%, conductance to 0.5% and temperature to 0.2 K.

RESULTS

The dielectric permittivity, ε', and dielectric loss, ε'', of pure and 20–80 wt% ATBN-containing thermoset measured for a fixed frequency of 1 kHz are plotted against the time during their curing at 298 and 373 K in *Figures 1* and *2*, respectively. During the cure at 298 K, ε' first slowly increases to a near plateau value, which is clearly evident for ATBN concentrations <60%, and thereafter decreases in one step to a value in the range of 3.8–5.8 for the pure thermoset and ATBN composites. The time when the decrease begins during the curing process remains virtually constant as the amount of ATBN in the thermoset is increased but the period of step decrease becomes longer. In *Figure 1*, ε' of the uncured thermoset or $\varepsilon'(t\to 0)$ slightly increases with increase in the amount of ATBN to 10.8 at the highest concentration. The 40 wt% ATBN-containing thermoset is anomalous in its behaviour. Within the experimental uncertainties of the measurement, the time period, from the instant the thermoset was mixed and thermally equilibrated to the time at which a point of inflection in the stepwise decrease in ε' appears, remains virtually unchanged with the change in the composition.

The plots of ε'' measured for a fixed frequency of 1 kHz against the curing time are shown in *Figure 2*. These initially show a decrease in ε'', which after reaching a local minimum increases to a peak value and thereafter decreases to an ultimate value ranging between 0.17 and 0.38 for the various compositions on curing at 298 K. With an increase in the amount of ATBN in the thermoset, the peak becomes obscured; its height decreases while its position remains unchanged.

The corresponding plots for curing at 373 K show none of the above features. Here the peak in ε'' is masked by a relatively large contribution from the d.c. conductivity as the amount of ATBN in the thermoset increases. A summary of the various dielectric features observed during the curing of the various thermosets is given in *Table 1*.

For an analysis in terms of an alternative representation of dielectric susceptibility, the ε^* data were converted to an electric modulus, M^*, formalism by:

$$M^*(t)=[\varepsilon^*(t)]^{-1}=M'(t)+M''(t)$$

where

$$M'(t)=\frac{\varepsilon'(t)}{\varepsilon'(t)^2+\varepsilon''(t)^2} \quad \text{and} \quad M''(t)=\frac{\varepsilon''(t)}{\varepsilon'(t)^2+\varepsilon''(t)^2}$$

and M' and M'' are the real and imaginary components of M^*. *Figures 3* and *4* show the plots of M' and M'', respectively, against time during the curing of the thermosets. The shape of the M' plot in *Figure 3* has a remarkable resemblance to that of the relative rigidity plots of rubber-modified epoxy measured with torsion-braid analyser by Chan *et al.*[5] (Figures 5 and 12 in ref. 5), and the M'' plot in *Figure 4* with that of the log decrement plot they obtained for a mechanical oscillation frequency in the range of 0.05–5 Hz.

After the measurements of the curing kinetics were complete, each thermoset sample was immediately cooled to 77 K and its ε' and ε'' in the cured state were measured for a fixed frequency of 1 kHz as a function of temperature from 77 to 350 K. In *Figure 5*, where ε' is plotted against temperature, ε' in this temperature range increases from ~2.6 to ~3.7 for pure thermoset. This increase in ε' for a given thermoset over the temperature range 77–300 K increases progressively as the amount of ATBN is increased. For the highest concentration of 80% ATBN, this increase is from ~2.6 to ~6.8.

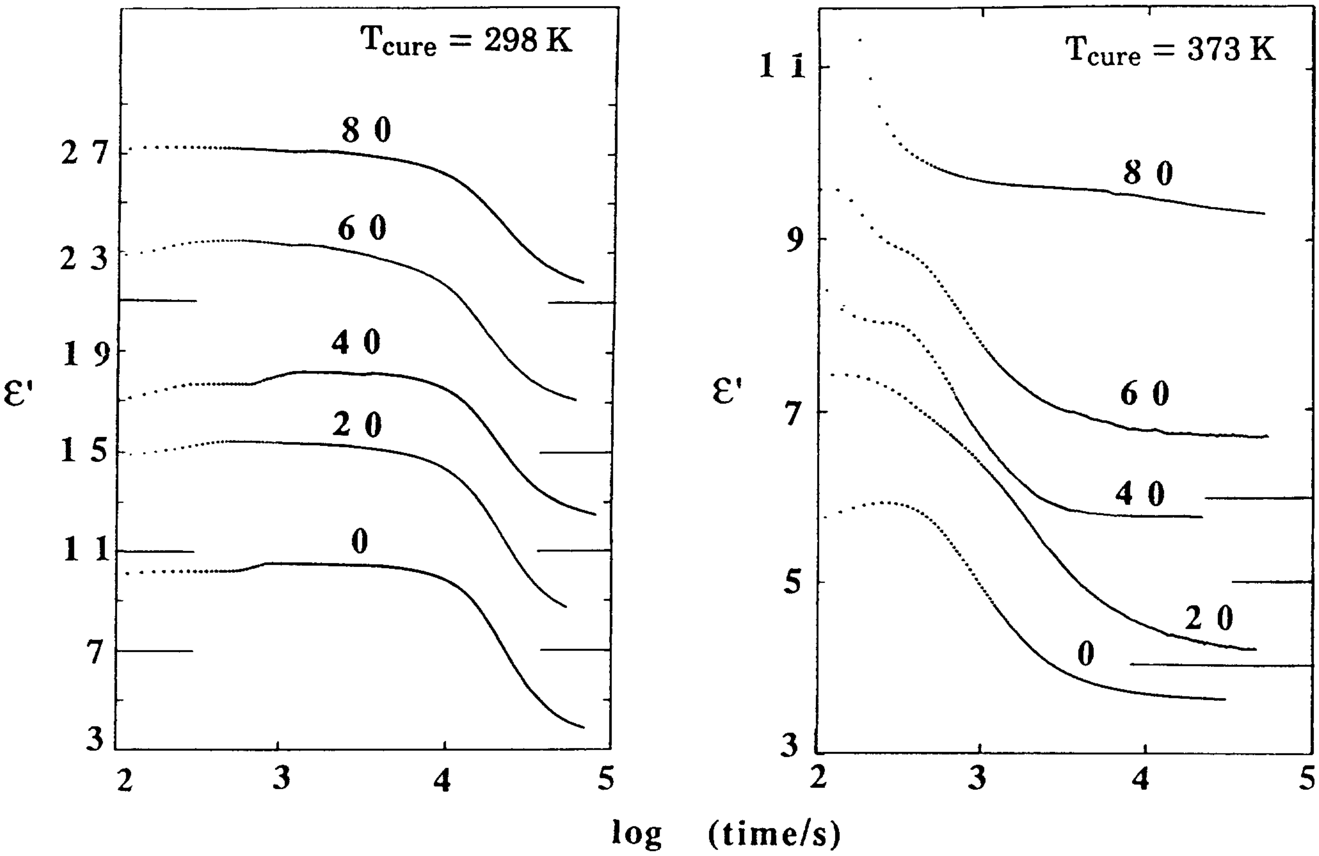

Figure 1 Dielectric permittivity, ε', of ATBN-containing DGEBA–Ancamide thermosets measured for a fixed frequency of 1 kHz plotted against the curing time. The numbers next to the curves refer to the amount (in g) of ATBN in 100 g of the thermoset, as given in *Table 1*. The curing temperature is also indicated. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 4, 8, 12 and 16 units, respectively, as shown by the horizontal lines

The ε'' of the pure and ATBN-containing cured thermosets measured for 1 kHz is plotted against temperature in *Figure 6*. The plots show an ε'' peak at ~125 K for the pure thermoset and over the temperature range 230–250 K for ATBN-containing thermosets, with a shoulder to the peak at <200 K. The positions of both the peak and the shoulder are shifted towards lower temperatures as the amount of ATBN in the thermoset is increased. A rapid increase in the d.c. conductivity increases the measured ε'' as the temperature is increased above 300 K. The features of ε' and ε'' seen in *Figures 5* and *6* are summarized in *Table 2*.

DISCUSSION

Dielectric monitoring of curing

As the components of a thermoset liquid react and the terminal epoxide group opens to form one OH group and one covalent bond with the amine[4,12–18], its dielectric properties change. The change is partly caused by a concurrent decrease in the net dipole moment per monomer unit, which is related to the equilibrium permittivity, a static property, and partly by an increase in the viscosity or molecular relaxation time of the monomer unit, which is related to the dynamic properties[19–25]. The static properties discussed here are associated with the number and magnitude of the dipole moments of the various segments and the dynamic properties with the ionic transport, and molecular chain or network segment diffusion. Both are expected to depend upon the manner by which temperature, curing time, extent of crosslinking and densification affect the effective dipole moment, their number density and the rate of dipolar reorientation as well as ion transport by creating steric hindrance for mass diffusion. These effects have been discussed in earlier papers where it was shown that the increase in the molecular relaxation time predominantly controls the properties of a thermoset during its curing[19–26].

In addition to the above aspects, which are common to all thermosets, in the elastomer-containing thermoset mixtures, the elastomer (ATBN) phase separates prior to the gelation of the thermoset, when the increase in the molecular weight of the polymer lowers the compatibility of the elastomer with the epoxy network. This phase separation ceases as the viscosity increases rapidly near the gel point of the mixture. Thus, both the curing temperature, on which the time for gelation depends, and the amount of ATBN have a profound effect on the morphology of the cured thermoset. The effect of this morphology on the dynamic mechanical and fracture behaviour has been extensively studied[1,2,3,9,27–30], but the effects of this morphology on the dielectric behaviour seem to have been studied only by Daly and Pethrick[31]. In addition to the effects of this phase separation, it is anticipated that if the gelation time of the thermoset were to remain unaffected by the presence of ATBN, the morphology of the dispersed phase alone would also affect the rate of reactions between the amine and epoxide and thereby the density of the network ultimately formed. Thus, the dielectric behaviour observed during the curing of the thermoset would be influenced by both the amount and the morphology of the dispersed phase.

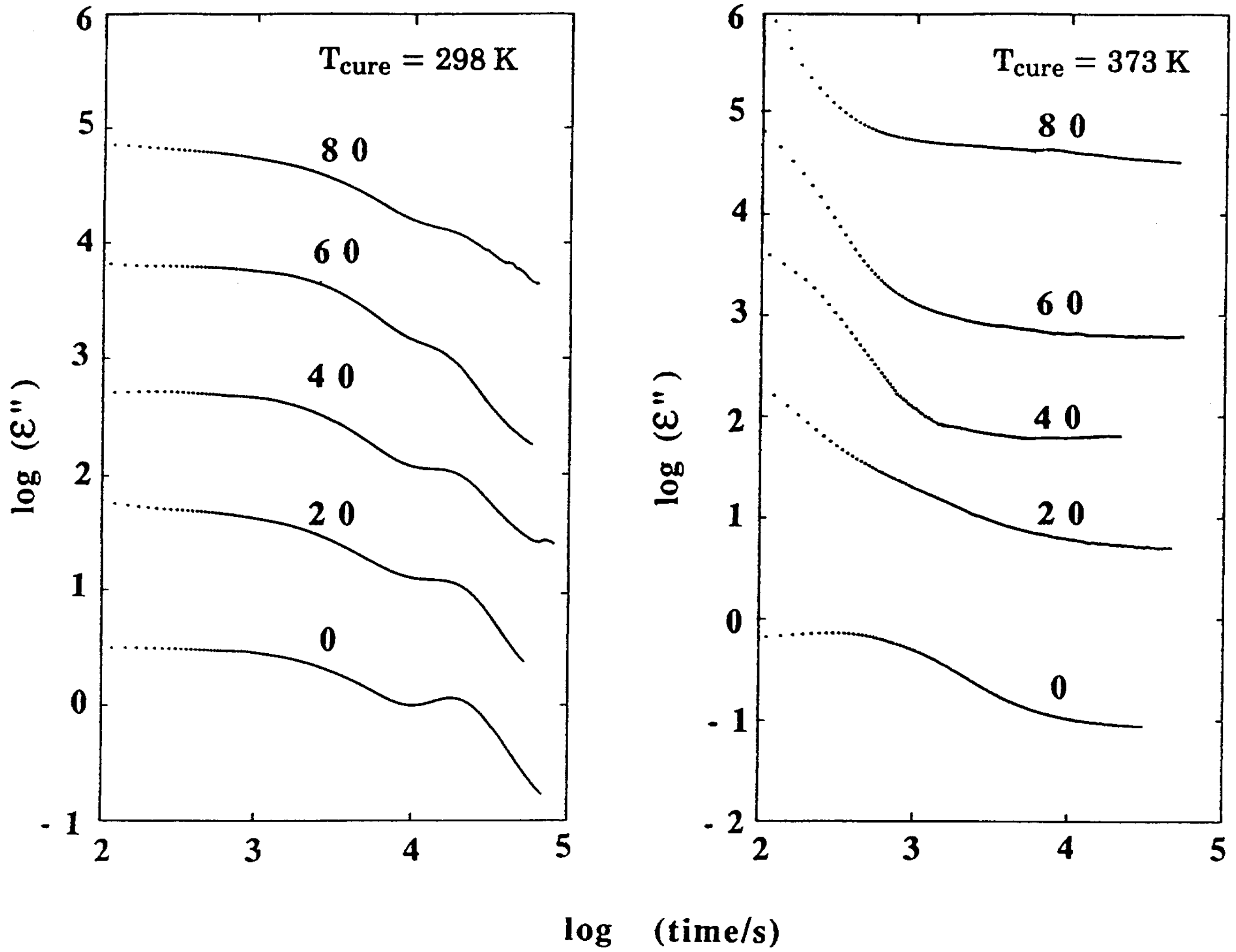

Figure 2 Dielectric loss, ε'', of ATBN-containing DGEBA–Ancamide thermosets measured for a fixed frequency of 1 kHz plotted against the curing time. The notations are the same as in *Figure 1*. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 1, 2, 3 and 4 logarithmic cycles, respectively

The ε' of the pure and ATBN-containing thermosets changes in a qualitatively similar manner at 373 and 298 K cure, as seen in *Figure 1*, but the sigmoidal decrease in ε' shifts to shorter times of $\sim 10^3$ s in comparison with $\sim 2 \times 10^4$ s observed at 298 K. Despite the care taken in keeping the time interval between mixing and thermostatically controlling the thermoset at 373 K, when curing occurs rapidly there may be significant errors in the ε' of the unreacted mixture, i.e. corresponding to $t \rightarrow 0$. Nevertheless, the values listed in *Table 1* do show an increase in ε' at $t \rightarrow 0$ with increase in the amount of ATBN for the thermoset at 373 K.

Of greater importance is the ε' value of the cured thermosets at $t \rightarrow \infty$. This increases with the amount of ATBN, but shows no consistent change on raising the curing temperature, as noted in *Table 1*. For the pure and 20 wt% ATBN-containing thermoset $\varepsilon'(t \rightarrow \infty)$ slightly decreases when the curing temperature is raised from 298 to 373 K, while for higher weight per cent ATBN-containing thermosets it increases.

The manner in which ε' of a thermoset mixture decreases during its cure and the appearance of a peak in ε'', seen in *Figures 1* and *2*, respectively, remarkably resembles the manner in which ε' shows a dispersion and ε'' a peak when the properties of a dipolar substance are measured as a function of frequency in an isothermal experiment and when no chemical or physical changes occur during the period of measurements. This resemblance, as discussed in detail in our earlier papers[22–25], is a reflection of the fact that chemically and physically irreversible changes in the structure that occur during the curing of a thermoset are dielectrically analogous to the increase in the measurement frequency in an isothermal experiment of a chemically and physically stable dipolar substance. It is due to the fact that relaxation functions are invariant with respect to the choice of the electrical frequency of measurement, ω, or the relaxation time, τ, as an experimental variable.

As mentioned earlier, an increase in the molecular weight of the polymer decreases the solubility of a substance dissolved in it. Phase separation of ATBN in the thermoset begins at a certain molecular weight, or equivalently after a certain time of isothermal cure when the particular molecular weight is reached. At this time, the ε' and ε'' would show a discontinuity only if (1) the contributions to ε^* and τ from the pure ATBN, or from its phase-separated state, are greater than those from its dissolved state in the homogeneous solution of the resin and the curing agent, and (2) the reactions between ATBN and DGEBA are too slow and too insignificant to alter the onset of phase separation. In an earlier study[6,7] of ATBN-containing DGEBA cured with diaminodiphenylmethane, we found that, for all concentrations of ATBN, phase separation was clearly observ-

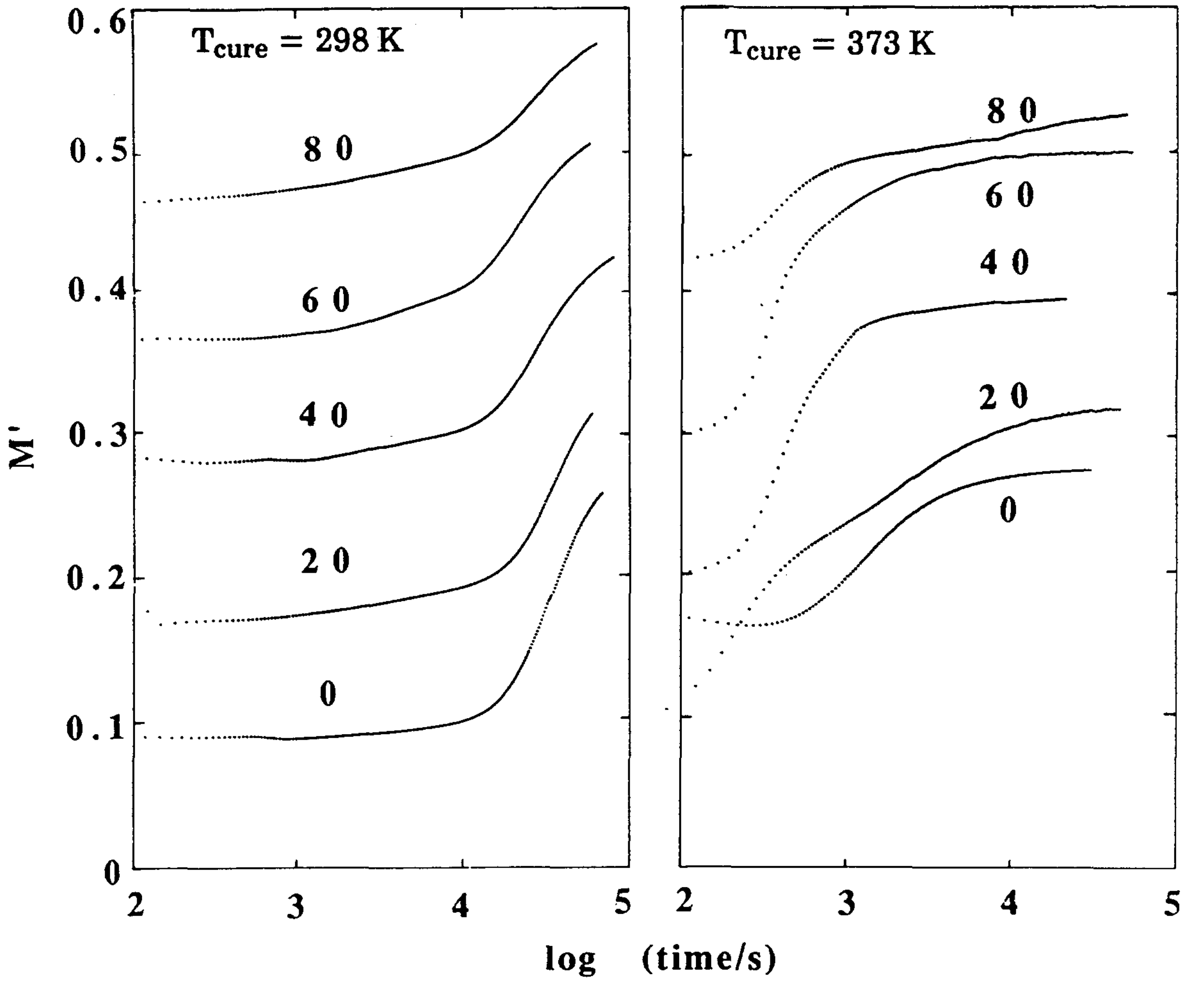

Figure 3 Real component of electrical modulus, M', of ATBN-containing Ancamide thermosets measured for a fixed frequency of 1 kHz plotted against the curing time. The notations are the same as in *Figure 1*. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 0.1, 0.2, 0.3 and 0.4 units, respectively

able in the plots of ε' and ε'' against curing time. But here for ATBN-containing DGEBA cured with Ancamide, none of the curves in *Figures 1* and *2* show any indication of phase separation. This is anticipated in view of our observation in *Table 2* that ε' (which is approximately the same as the static or limiting low frequency dielectric permittivity, ε_0) of ATBN at 300 K is 8.72, which is not much different from that of its homogeneous solution in the thermoset at that stage of cure where phase separation is anticipated as discerned from *Figures 1* and *2*. Although ε_0 of ATBN at 373 K is not known, the absence of any dielectric feature of phase separation during curing at 373 K implies that the differences between ε_0 of ATBN and its homogeneous thermoset solution at 373 K are also nearly the same.

Phase separation during the curing of a thermoset has an important consequence for further curing reactions. This is because after the phase separation of an elastomer has occurred, the probability of chemical reactions, as a result of molecular diffusion, between the DGEBA and Ancamide would decrease as the intervening regions of ATBN, by occupying randomly distributed macroscopic sites in the incompletely formed polymer network, would prevent the diffusion of reactants necessary for the chemical reaction. This means that the higher the concentration of ATBN in the thermoset the slower would be its curing after phase separation has occurred. In dielectric monitoring of the cure of an elastomer–epoxy mixture, one would thus expect that the (time) duration over which the step decrease in ε' occurs would be longer for a high ATBN concentration than for a low ATBN concentration and that the width of the ε'' peak would be broader. The corresponding plots of M' would show that the time period for reaching the limiting plateau increases and the M'' peak broadens with an increase in the amount of ATBN. *Figures 1–4* show that such changes in the dielectric features of the ATBN-containing thermosets do occur during their cure. We therefore conclude that an increase in the amount of ATBN decreases the rate at which crosslinking reactions between DGEBA and Ancamide occur to form the network structure.

Dielectric properties of cured thermosets

Rubber-toughened thermosets are often used for electric encapsulation either as bulk or as thin films. Therefore, a knowledge of the dielectric properties of their cured states are particularly valuable. These properties of the ATBN-containing DGEBA–Ancamide thermosets cured at two different temperatures are summarized in *Table 2*, and the ε', ε'', M' and M'' measured for a fixed frequency of 1 kHz are plotted against the temperature in *Figures 5–8*. Here, ε' of the cured thermosets at 300 K increases with increase in the amount of ATBN. This is true for both curing temperatures. But, ε' at 100 K, which is approximately equal to the limiting high frequency permittivity, ε_∞, decreases

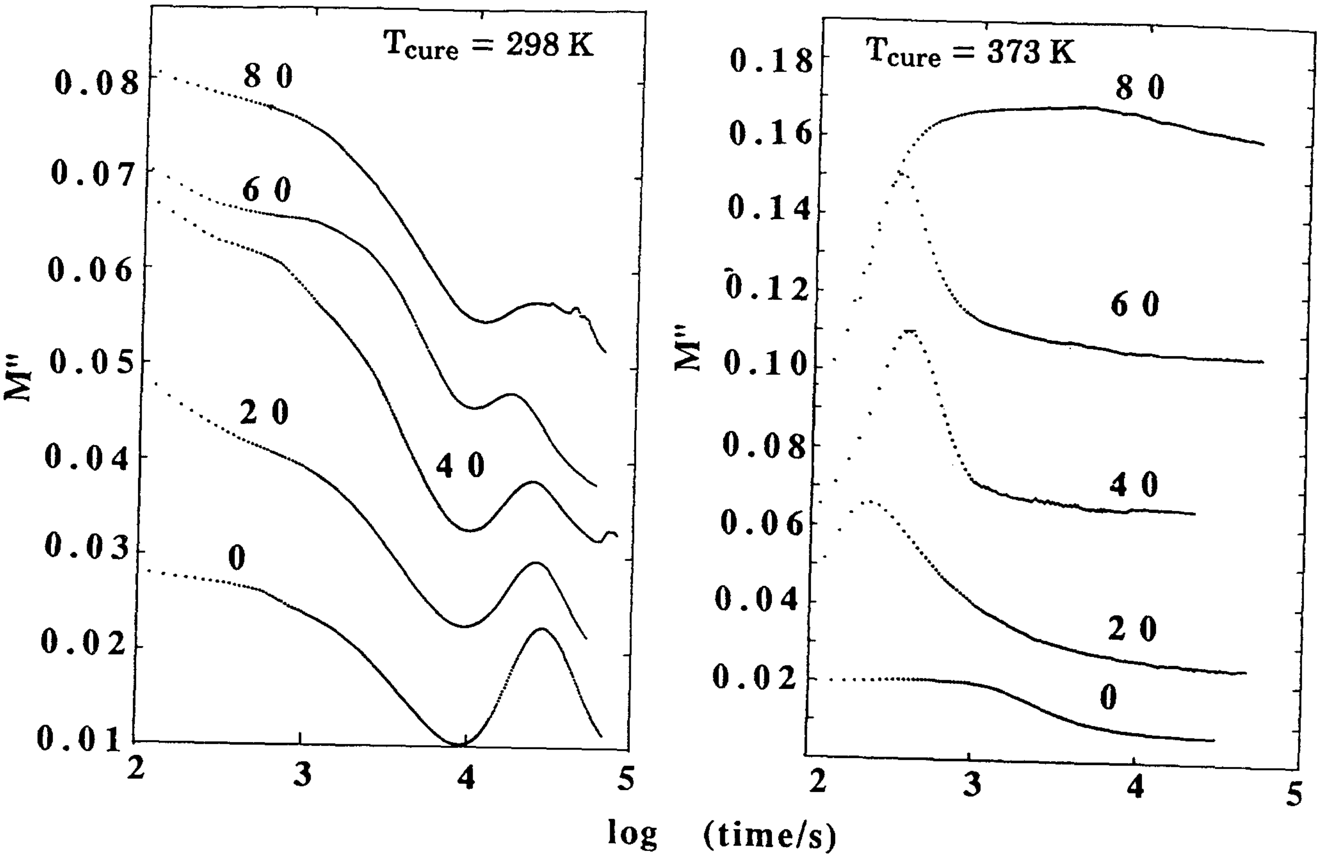

Figure 4 Imaginary component of electrical modulus, M'', of ATBN-containing Ancamide thermosets measured for a fixed frequency of 1 kHz plotted against the curing time. The notations are the same as in *Figure 1*. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 0.02, 0.04, 0.04 and 0.06 units, respectively, for the left-hand side figure and by 0.02, 0.04, 0.06 and 0.08 units, respectively, for the right-hand side figure

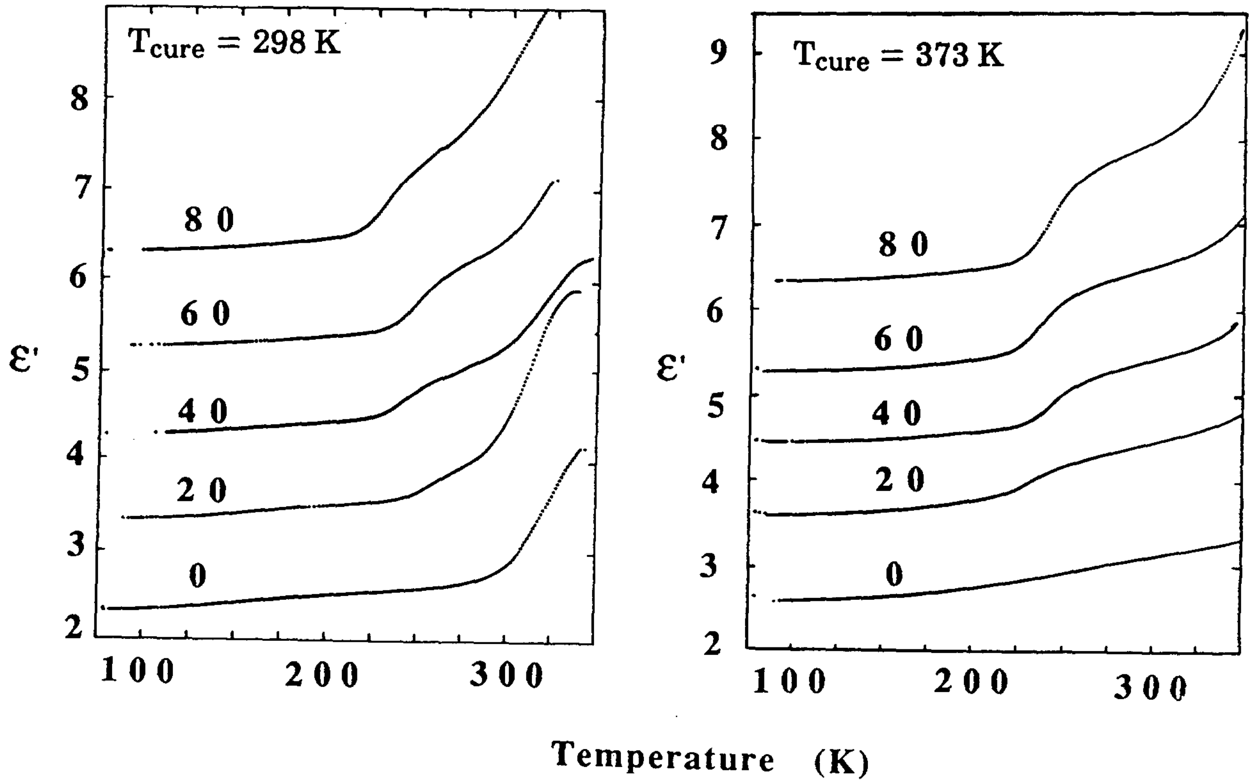

Figure 5 Dielectric permittivity, ε', of ATBN-containing DGEBA–Ancamide thermosets measured for a fixed frequency of 1 kHz after their cure is plotted against temperature. The notations are the same as in *Figure 1*. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 1, 2, 3 and 4 units, respectively

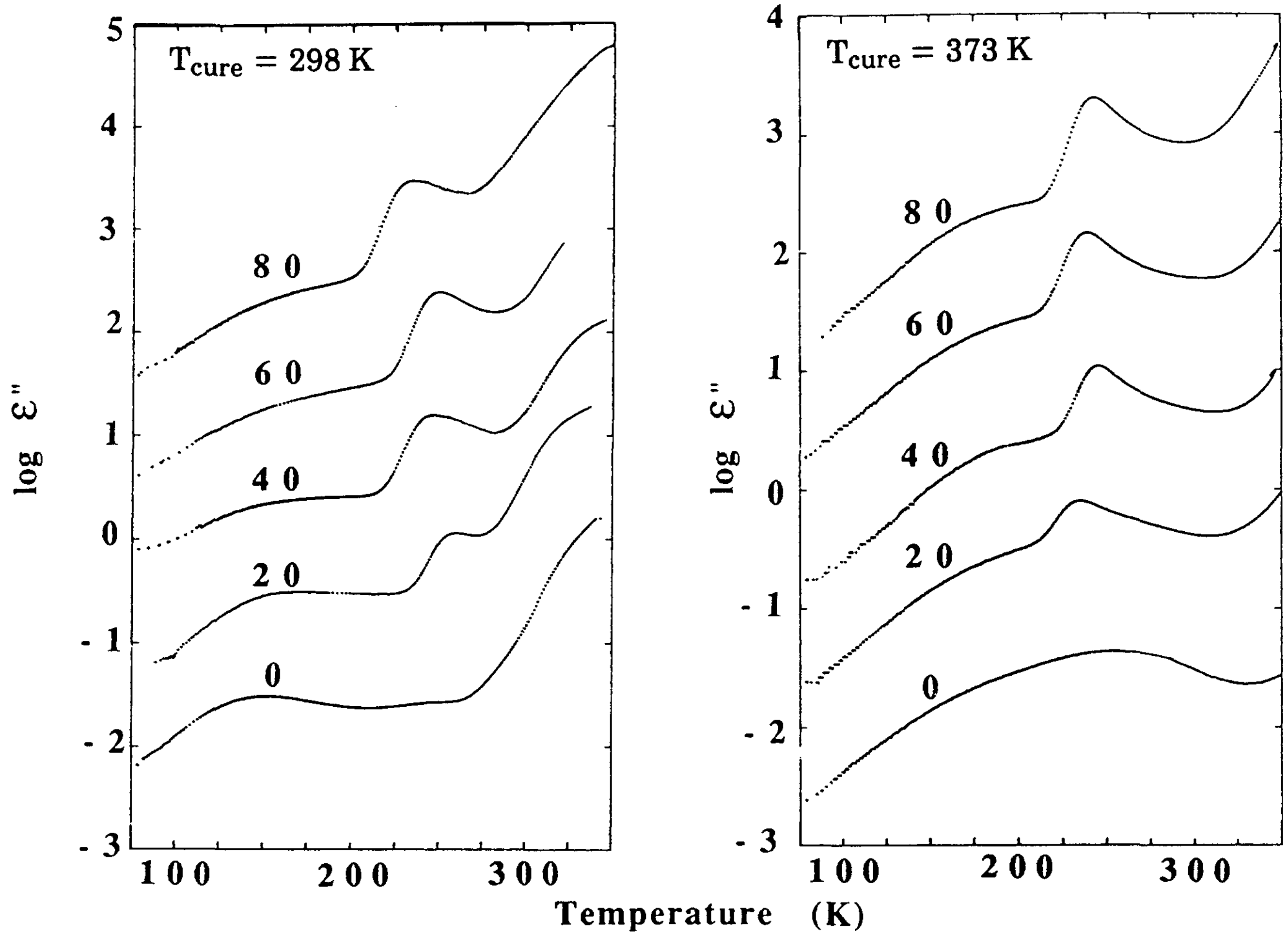

Figure 6 Dielectric loss, ε'', of ATBN-containing DGEBA–Ancamide thermosets measured for a fixed frequency of 1 kHz after their cure is plotted against temperature. The notations are the same as in *Figure 1*. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 1, 2, 3 and 4 logarithmic cycles, respectively

Table 2 Dielectric properties of pure DGEBA–Ancamide thermoset, the ATBN-containing thermosets in their cured states and pure ATBN

ATBN in DGEBA (wt%)	$T_{\varepsilon'_{\max}}$	$\varepsilon''_{\max}$	ε'' (T=300 K)	ε' (T=100 K)	ε' (T=300 K)	ε' ($T_{\varepsilon''_{\max}}$)
			Curing at 298 K			
0	153.6	0.03	0.15	2.65	3.73	2.83
20	259.7	0.10	0.36	2.63	5.03	3.52
40	245.1	0.15	0.18	2.59	4.63	3.46
60	249.8	0.21	0.19	2.48	4.98	3.54
80	234.3	0.28	–	2.67	6.80	3.91
			Curing at 373 K			
0	273.1	0.05	0.03	2.56	3.12	3.02
20	235.1	0.08	0.04	2.57	3.45	3.02
40	244.2	0.11	0.04	2.42	3.42	2.91
60	239.3	0.14	0.06	2.26	3.50	2.88
80	242.4	0.19	0.08	2.32	3.95	3.08
Pure ATBN[a]	245.4	1.16	2.07	2.12	8.72	5.77

[a] Data from ref. 6 measured for 300 K

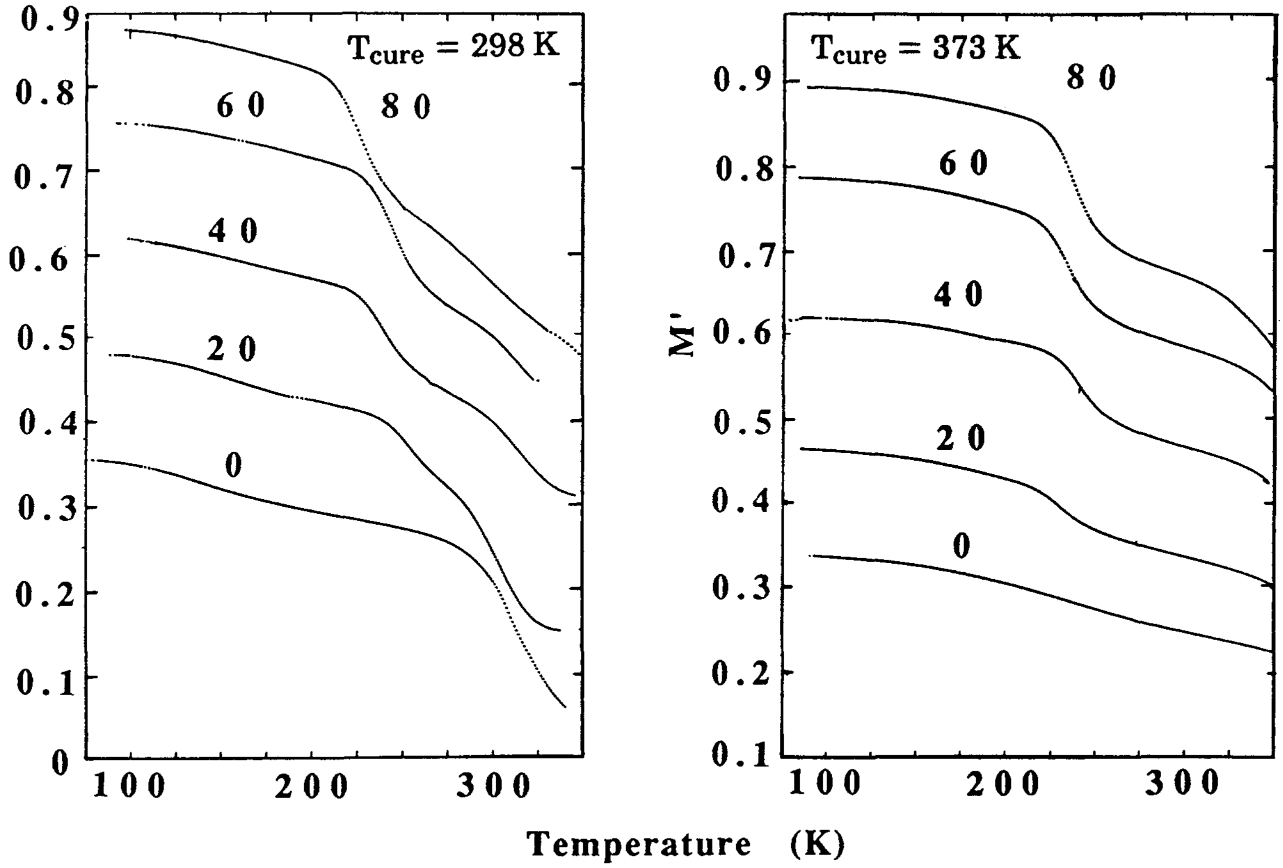

Figure 7 Real component of the electrical modulus, M', of ATBN-containing DGEBA–Ancamide thermosets measured for a fixed frequency of 1 kHz plotted against temperature. The notations are the same as in *Figure 1*. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 0.1, 0.2, 0.3 and 0.4 units, respectively

with increase in the amount of ATBN in the thermoset. The $\varepsilon''_{\max}$ consistently increases with the amount of ATBN, but the temperature at which the ε'' peak appears decreases for the thermoset cured at 298 K and remains constant or slightly increases for that cured at 373 K. The ε'' peak for pure ATBN appears at 245 K with a peak height of 1.16 as noted in *Table 2*. Even for 80% ATBN-containing thermoset where the weight ratio of (DGEBA + Ancamide) to ATBN is 1.87:1, the ε'' peak height is <0.24 of that of pure ATBN. This means that either part of the ATBN is consumed by its reaction with DGEBA and/or that a substantial amount of ATBN remains molecularly dissolved in the epoxy network and does not phase separate. In this respect, it is revealing that curing at 373 K causes the height of the ε'' peak to further decrease to less than one-sixth of that observed for pure ATBN. If interpreted in terms of the difference between the morphologies of the ATBN-containing thermoset cured at different temperatures, as suggested by Yamanaka *et al.*[9], it implies that a spherical domain structure with fairly uniform domain size on curing at 373 K produces a thermoset with an ε'' value lower than that in which a co-continuous two-phase morphology is achieved on curing at 298 K. Clearly, high-temperature curing produces a plastic more suitable for electrical applications.

The free energy of an elastomer-containing thermoset system decreases as the polymerization proceeds because the configurational entropy of the system decreases more than its enthalpy. By using the Flory–Huggins[32] approximation for free energy per unit volume, Williams *et al.*[33] have developed a thermodynamic model to show that phase separation increases the thermodynamic stability of the system. In this model, phase separation would produce a dispersed phase rich in epoxy at high volume fraction of the elastomer, i.e. the epoxy becomes a reinforced elastomer with dispersed thermoset particles in an elastomer matrix, rather than a reinforced thermoset with dispersed elastomer in a thermoset matrix. In our studies this inversion is likely to occur for the 80 wt% ATBN-containing thermoset (it occurs at 0.28 volume fraction in the model of Williams *et al.*[33]). The dielectric properties seem, however, relatively insensitive to the occurrence of this phase inversion, which confirms our earlier and similar observations on ATBN-containing DGEBA cured with diaminodiphenylmethane[6,7].

The plots in *Figure 6* and *8* show that the sub-T_g relaxation peak in the ATBN-containing thermosets is a combination of broad γ- and β-relaxation peaks of the pure DGEBA–Ancamide thermoset and the α-relaxation peak of the phase-separated, pure ATBN. Furthermore the plots show that the shoulder to the low-temperature side of the peak, which also constitutes the background over which the peak from the α-relaxation of the phase-separated ATBN is superposed, is a result of contributions from both the sub-T_g relaxation in the ATBN-dispersed phase reported earlier[6] and in the DGEBA–Ancamide matrix. The height of this shoulder in the ε'' plots in *Figure 6* decreases with an increase in the amount of ATBN and a decrease in the amounts of DGEBA and Ancamide. This confirms that the predominant contributions to ε'' at low temperatures are from the γ- and β-relaxations of the DGEBA–Ancamide network in the matrix of the thermoset.

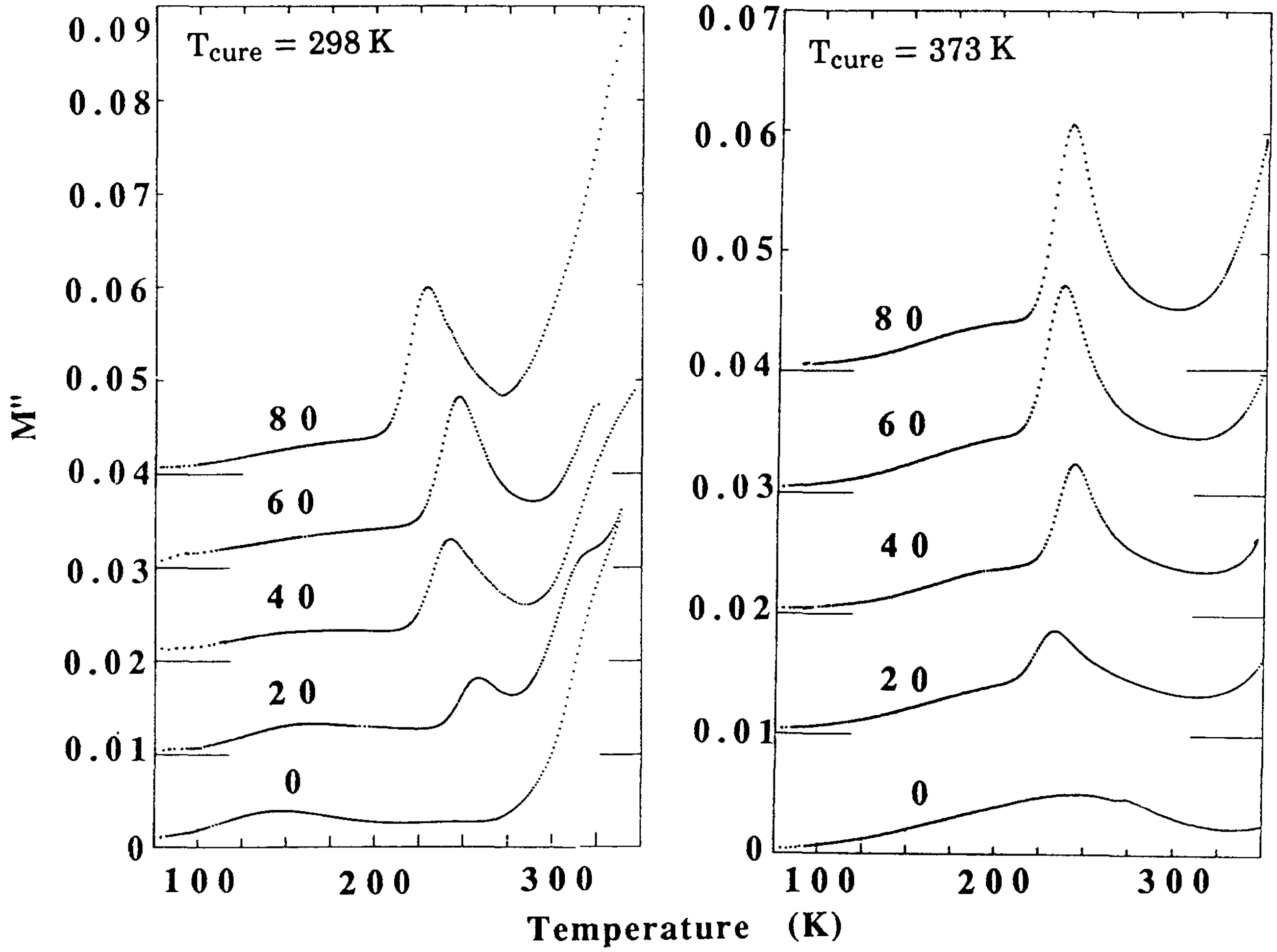

Figure 8 Imaginary component of the electrical modulus, M', of ATBN-containing DGEBA–Ancamide thermosets measured for a fixed frequency of 1 kHz plotted against temperature. The notations are the same as in *Figure 1*. Plots labelled 0 are drawn to scale. Those labelled 20, 40, 60 and 80 have been shifted upwards by 0.01, 0.02, 0.03 and 0.04 units, respectively, as shown by the horizontal lines

As seen in *Figures 5* and *6* and *Table 2*, the ε' and ε'' of the elastomer-toughened DGEBA–Ancamide thermosets at 300 K are almost entirely determined by the properties of the dispersed phase of ATBN in the matrix of the thermoset. At first sight it may seem that further cure or post cure of the ATBN-containing thermoset would not significantly decrease its ε' and ε'' from the values given in *Table 2*. But, since curing at 373 K significantly decreases both ε' and ε'' at 300 K, this suggests that post-curing at a higher temperature of an elastomer-toughened thermoset, which was cured at a relatively low temperature, may further reduce its ε' and ε''. These effects, which seem useful in the processing of a thermoset, are yet to be studied in detail.

CONCLUSIONS

Studies of curing kinetics and the dielectric properties of ATBN-containing DGEBA cured with Ancamide show several features as follows:

1. The rate of curing becomes slower with increased amounts of ATBN in the thermoset, mainly because the probability of diffusion of reacting groups towards each other is decreased when these groups are obstructed by the dispersed elastomer after its phase separation.
2. The ε' and ε'' ultimately reached on curing increases with increase in the amount of ATBN but decreases with increase in the temperature of cure for the same ATBN concentration.
3. The prominent relaxation peak in the cured ATBN-containing thermosets is the α-relaxation peak of pure ATBN, which is superposed on the γ- and β-relaxation peaks of the pure thermoset.
4. The change in the dielectric properties of the cured thermosets with increasing amount of ATBN indicates that the expected inversion in the constituents of the matrix from a predominantly cured epoxide to a predominantly elastomer ATBN is dielectrically undetectable.

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