

# Influence of the temperature of cure on the mechanical properties of ATBN/epoxy blends

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Mechanical properties (impact strength,  $K_{IC}$ , elastic modulus, hardness and loss factor) of ATBN/DGEBA blends of various compositions, cured at different temperatures, have been investigated. Both homogeneous and heterogeneous materials, with different properties, have been obtained. ATBN has been shown to be ineffective if a massive phase separation does not occur. Maximum toughness, measured by  $K_{IC}$ , is obtained in the 120°–140°C cure temperature range. SEM micrographs of impacted specimens show that the moving cracks go through the segregated particles and suggest they lack ductility. The improvement in fracture resistance also depends on modifications of matrix properties.

(Keywords: epoxies; reactive rubbers; fracture; impact strength)

## INTRODUCTION

Extensive work has been carried out on the mechanical properties of rubber modified epoxy resins and it has been shown that a substantial increase in fracture resistance can be achieved by using carboxyl terminated acrylonitrile-co-butadiene rubbers (CTBN)<sup>1–16</sup>. It has also been shown that similar results can be obtained with amine terminated rubbers (ATBN)<sup>16,17</sup>. Although the effects of such reactive rubbers have been widely investigated, the levels of achievable toughness so far reported (evaluated either by the critical parameters  $K_{IC}$  and  $G_{IC}$  or by the impact strength) are very different. For instance the increase in fracture energy,  $G_{IC}$ , ranges from ~1 to ~30 times.

A large number of blend formulations have been studied and different testing methods adopted, so that the results cannot be directly compared to one another. Nevertheless discrepancies are still present in materials of similar composition, which suggest that also the processing conditions can influence the final properties.

Unlike toughened thermoplastics that are manufactured with the appropriate morphology of the dispersed phase, the epoxy resins are polymerized by the end user in conditions that, depending on the application, can be very different.

For instance, in the building industry room temperature cure systems are preferentially used, whereas high temperature cure systems are preferred for composites.

Therefore, the knowledge of the dependence of mechanical properties on temperature of cure is important in deciding whether or not the use of a rubber modified epoxy resin is convenient for a certain application.

In this study the effects of temperature and cure on ATBN/epoxy blends have been specifically investigated and compared with those brought about by variations in rubber content.

## EXPERIMENTAL

### Materials

The amine terminated rubber was the type Hycar 1300X21 (BF Goodrich) with an acrylonitrile content of 10% and an amine equivalent weight of about 1100. The non-reactive rubber was the type Hycar 1312 (BF Goodrich). The epoxy prepolymer was a low molecular weight liquid diglycidyl ether of bisphenol-A, Epon 828 (Shell Co.). The curing agent was reagent grade piperidine.

Resin and rubber were first mixed together and heated for a short time at 60°C, piperidine was then added and the mixture thoroughly stirred. The resulting blends were poured into PTFE-coated trays and cured at constant temperature, in the range 10°–160°C. All the blends were given a postcure of 15 h at 120°C.

It has been found in this study that curing at low temperature promotes the formation of optically clear materials, therefore all the blends obtained at 10°C are transparent. Increasing the temperature of cure causes a gradual loss in transparency and the temperature at which this occurs depends on the ATBN content. For instance, the 5% ATBN blend is already opaque if cured at 25°C whereas the 50% ATBN blend is still transparent if cured at 60°C. Postcuring at 120°C does not have any effect on the morphology of the cured materials. The blend formulations, along with temperatures and times of cure, are summarized in *Table 1*. The postcured slabs were saw cut and machined to form the test pieces for the various measurements.

### Testing

Impact tests were performed with a Charpy pendulum following the procedure DIN 53453. Stress intensity factors,  $K_{IC}$ , were measured with an instrumented Charpy pendulum (CEAST MK2) at the impact speed of 1 m s<sup>-1</sup>.  $K_{IC}$  was evaluated by the equation:  $K_{IC} = Y\sigma_c\sqrt{a}$  in which

$Y$  is a geometrical factor,  $\sigma_c$  is the stress at failure and  $a$  is the crack length. The starter cracks were introduced by pressing a razor blade into the test pieces previously heated at 120°C. A typical plot of  $\sigma_c$  vs.  $1/(Y\sqrt{a})$  is shown in Figure 1. Shear moduli were measured with a torsion tester following the ASTM D 1043-61T method. Mechanical damping measurements were carried out on samples in the form of circular plates (2 mm thickness, 36 mm diameter) vibrating in their lowest flexural symmetrical modes. Specific heats were determined with a DSC-2 Perkin Elmer calorimeter at a scanning rate of 10 deg min<sup>-1</sup>. Rockwell hardness was measured according to the ASTM D785 method, at 21°C. SEM examinations were made on samples impacted at room temperature.

## RESULTS AND DISCUSSION

The amine terminated rubbers are reactive towards the epoxy resins and different products can be formed depending on the ratio of the equivalents ( $ER$ ) of the two components. If  $ER > 1$  a thermoplastic will be obtained, whereas for  $ER < 1$  a thermoset will result (with the ATBN 1300X21 and the epoxy resin Epon 828,  $ER$  equals 1 when the weight ratio rubber/epoxy is about 5.5). Because of the capability of ATBNs of giving rise to flexible products they are currently used in the field of castable elastomers or for B-stage processes.

At low concentration (say  $ER < 0.05$ ) ATBNs are suitable for toughening epoxy resins. Such rubbers are soluble in the epoxy prepolymer whose capability of keeping them in solution decreases with increasing the molecular weight during the polymerization. Thus, at the early stages of cure, a massive segregation of rubber, in the form of spherical droplets, can take place. The importance of phase separation lies in the capability of the dispersed phase of triggering the dissipative processes necessary to improve the fracture resistance. The morphology of such blends depends on the amount of rubber, temperature of cure and on whether ATBN and resin are prereacted before the addition of the hardener.

The effects of both temperature and cure and rubber content on the impact strength are shown in Figure 2. The lower curve refers to optically clear blends cured at low

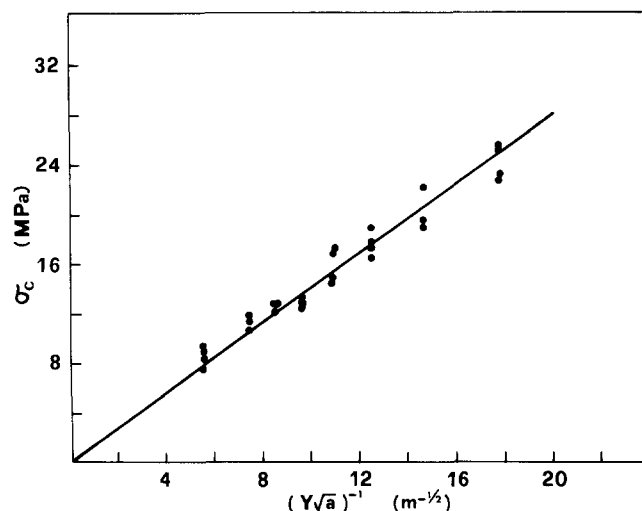


Figure 1 Critical stress data for 10% ATBN blends cured at 100°C.  $K_{IC} = 1.42 \text{ MNm}^{-3/2}$

temperature (10°C) and the upper one refers to optically opaque blends cured at high temperature (120°C). The graph emphasizes the importance of the temperature of cure which, to some extent, is more important than the amount of rubber itself.

It is worth noting that also the fracture resistance of the unmodified resin varies with the cure temperature. The effect is barely noticeable in the impact strength, but is clearly revealed by  $G_{IC}$  that from a value of  $120 \text{ J m}^{-2}$  goes up to  $350 \text{ J m}^{-2}$  (at  $1 \text{ m s}^{-1}$ ). The poorer performance of the low temperature cured resin can have several sources. Morgan and O'Neal<sup>18</sup> have shown that the DGEBA epoxy monomer can crystallize during the setting of epoxies at low temperature. This results in a vitreous matrix in which small crystals of monomer are dispersed. On postcuring at high temperature the crystals melt and the liquid migrates, thus leaving a network of microvoids with a weakening effect on the glass. On the other hand, different network structure can result both from different reaction paths brought about by different temperatures (an effect also known in other thermosetting systems) and from a possible volatilization of the hardener. The last two phenomena account for the lower  $T_g$  of the high temperature cured resin.

Although the low temperature cured blends are optically clear, their thermal behaviour suggests a very low degree of segmental mixing. Specific heat measurements, Figure 3, did not show any shift of the transition temperatures of the two components, the blends almost behaving like mechanical mixtures. The only relevant effect is a broadening of the transition of the epoxy resin. Internal friction data, Figure 4, confirm a two phase structure, although the dimensions of the rubbery domains must be below the wavelength of light. The impact strength only increases when the volume fraction of the added rubber is greater than 0.5, possibly as a result of phase inversion.

The high temperature cured blends have a much improved impact resistance, Figure 2, attributable to changes in the morphology of the segregated phase, its apparent volume fraction and properties of the rigid matrix. For ATBN contents up to 20% the increase in impact strength parallels the loss in optical clarity caused by the development of larger rubber particles. The step

Table 1 Composition, temperature and time of cure of the examined blends

Epoxy (828)	Amine terminated rubber (1300 × 21)	Non-reactive rubber (1312)	Hardener*	Temp. (°C)	Time (h)
100	—	—	6.4	10	72
100	—	—	6.4	60/160	15
95	5	—	5.6	10	72
90	10	—	5.6	10	72
90	10	—	5.6	60/160	15
80	20	—	4.9	10	72
80	20	—	4.9	120	15
70	30	—	4.2	120	15
60	40	—	3.4	10	72
50	50	—	2.7	10	72
90	—	10	5.8	120	15
80	—	20	5.1	120	15

\* The amount of piperidine was changed so as to have a constant amine to epoxy groups ratio.  
Compositions in parts by weight.  
All the blends postcured at 120°C for 15 h.

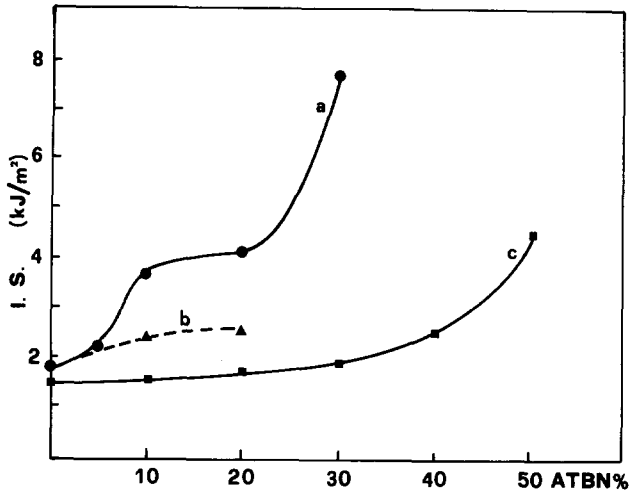


Figure 2 Impact strength (DIN 53453) vs. ATBN content (wt%) for (a) heterogeneous blends ( $T_c=120^\circ\text{C}$ ), (b) heterogeneous blends ( $T_c=120^\circ\text{C}$ , Hycar 1312), (c) homogeneous blends ( $T_c=10^\circ\text{C}$ )

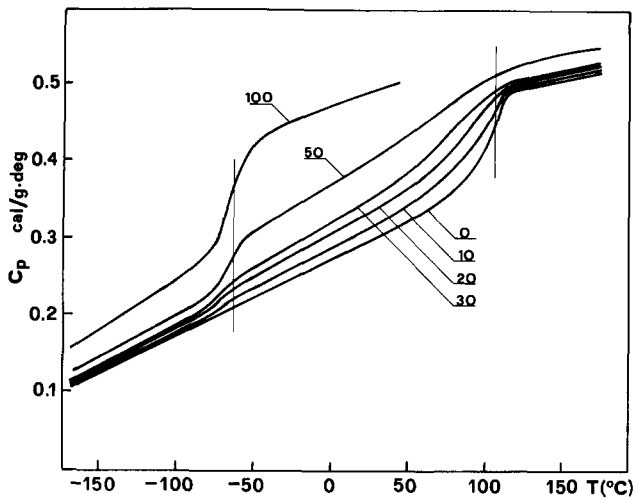


Figure 3 Specific heat vs. temperature for homogeneous blends of different ATBN content (wt%)

increase above 20% can be attributed to phase inversion which seems to occur for a rubber concentration lower than in the case of optically clear blends. ATBN has been shown to behave similarly to CTBN<sup>16</sup> and it has been reported that in CTBN/epoxy blends cured at high temperature phase inversion has already occurred for a CTBN concentration of 30%<sup>19-21</sup>. Also the hardness depends on both the amount of rubber and temperature of cure, Figure 5. The small variation in the hardness of the pure resin can be attributed to the lower yield stress (in compression) of the high temperature cured resin. In the range 0-20% of ATBN the effects of temperature of cure and rubber content are almost the same.

The room temperature modulus decreases with increasing the rubber content, Figure 6, and so does the softening temperature, particularly above 20% of ATBN. Low values of modulus, hardness and distortion temperature prevent the blends with more than 20% of rubber from being used, at least in structural applications. Since an amount of rubber of 10% already produces a twofold increase in impact strength and keeps the loss of other

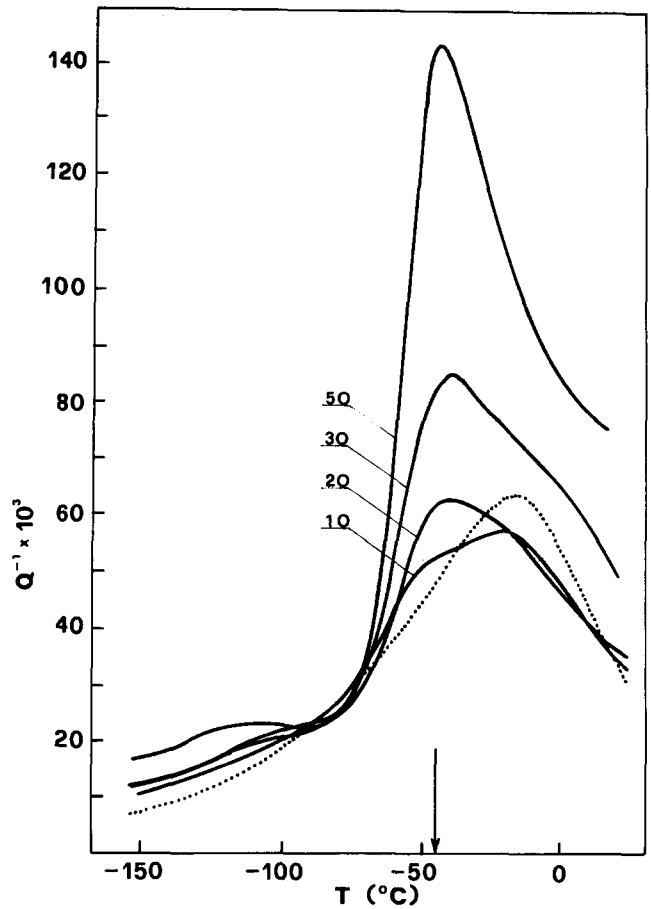


Figure 4 Dissipation factor for homogeneous blends of different rubber content (wt%). The arrow indicates the location of the peak maximum of the pure rubber. The dotted line refers to pure resin

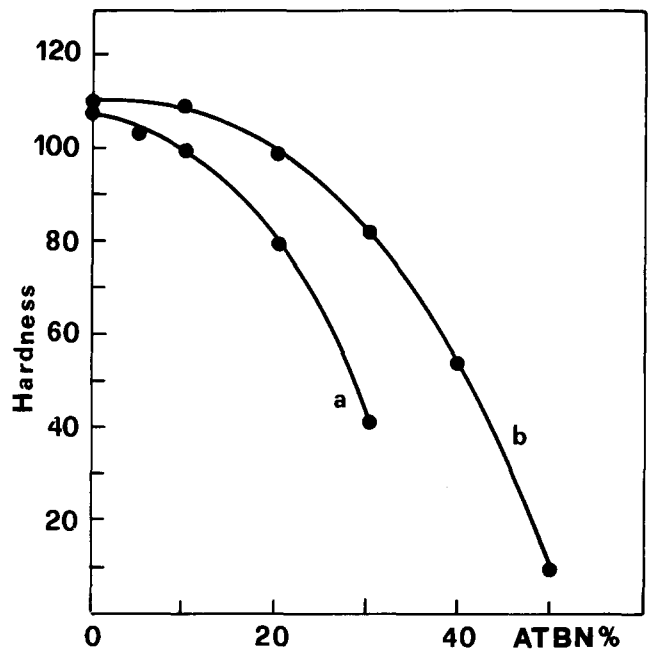


Figure 5 Rockwell hardness (HRL) vs. ATBN content (wt%) for (a) heterogeneous blends, (b) homogeneous blends

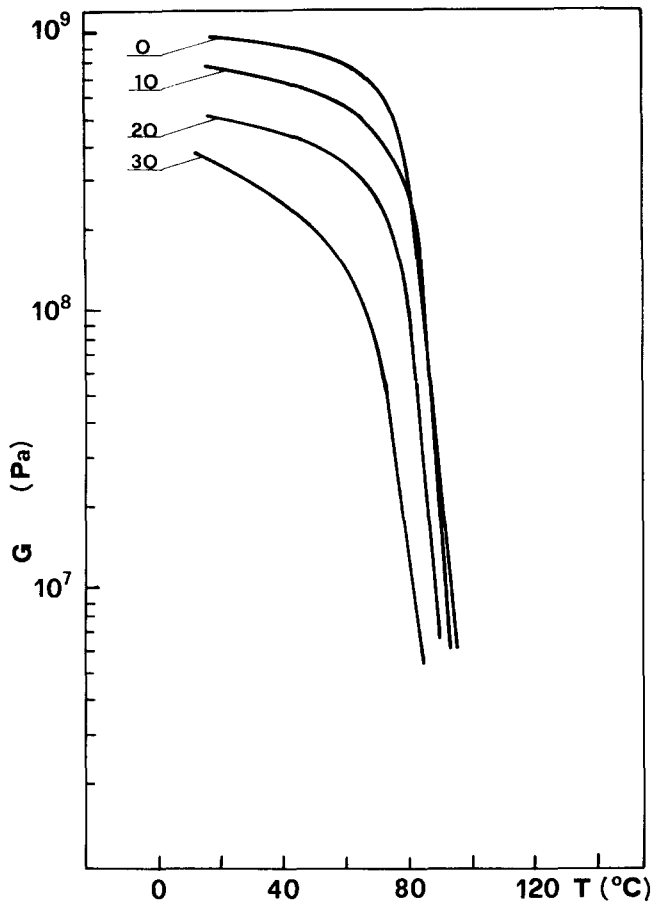


Figure 6 Five second shear modulus as a function of temperature for blends of different ATBN content (wt%,  $T_{\text{cure}} 120^{\circ}\text{C}$ )

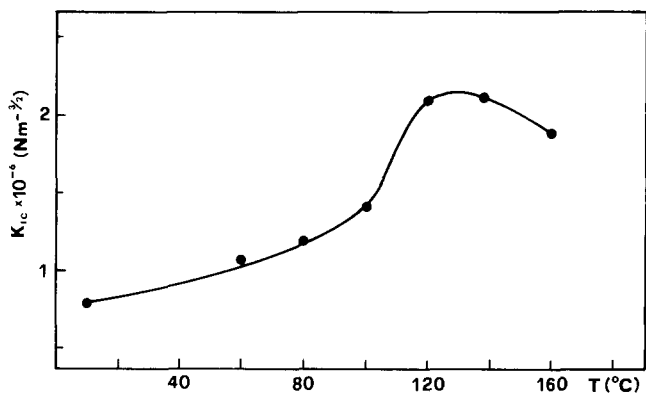


Figure 7 Critical stress intensity factor as a function of temperature of cure for 10% ATBN blends

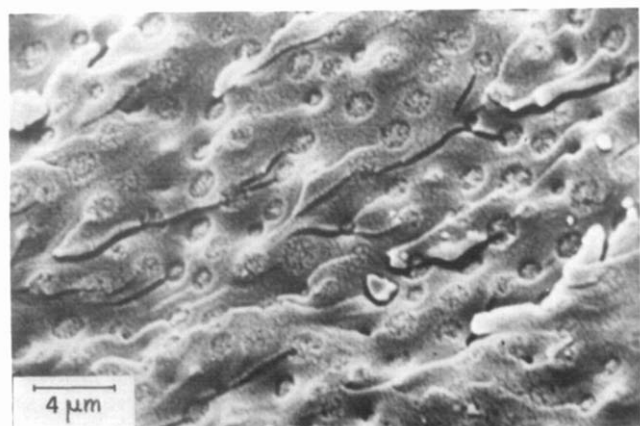


Figure 8 SEM micrograph of a 10% ATBN blend cured at  $60^{\circ}\text{C}$

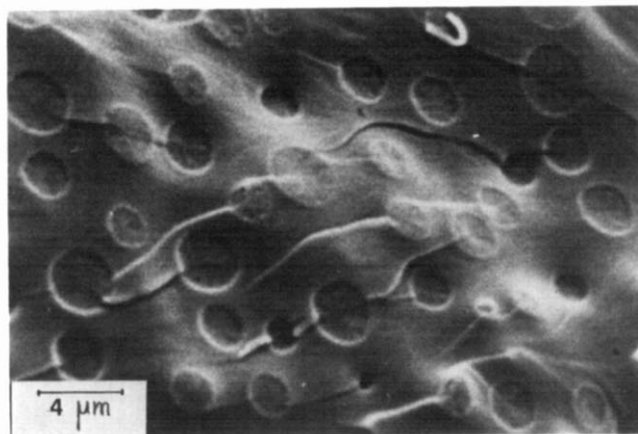


Figure 9 SEM micrograph of a 10% ATBN blend cured at  $100^{\circ}\text{C}$

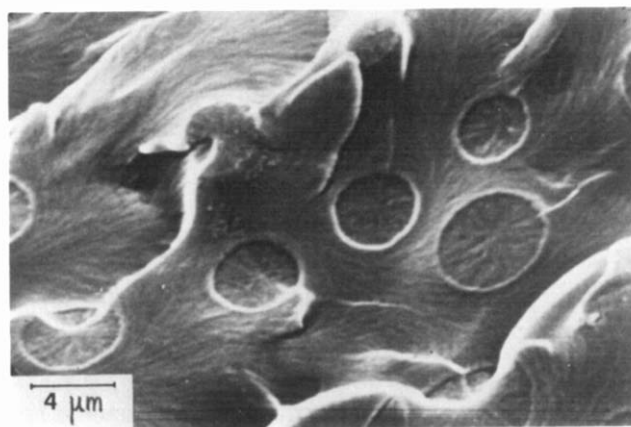


Figure 10 SEM micrograph of a 10% ATBN blend cured at  $140^{\circ}\text{C}$

mechanical properties to a minimum, the behaviour of blends of this composition has been investigated over a wide range of cure temperatures. Figure 7 shows the dependence of the critical stress intensity factor,  $K_{IC}$ , on  $T_{\text{cure}}$  and also shows that the addition of ATBN does not necessarily produce an increase in fracture resistance. The  $K_{IC}$  value of the pure resin cured at  $120^{\circ}\text{C}$  ( $1.12 \times 10^6 \text{ Nm}^{-3/2}$ ) is in fact higher than the values of the 10% blends cured below  $80^{\circ}\text{C}$ .

A large number of factors have been shown to influence the mechanical properties of toughened epoxy resins and it is always difficult to establish which one predominates in a particular system. The difficulty arises from the fact that such factors are dependent on one another. Thus, a change in cure temperature brings about changes in viscosity, reactivity (both rubber and hardener react with the resin), surface tension (the rubber separation is expected to follow the rules of homogeneous nucleation), solubility, thermal stresses around the rubber particles when the blend is cooled down and so on. On the other hand, factors that have been shown to be relevant in some cases seem not to be so in others.

For instance, it is recognized that the functionality of the rubber is important for it allows a strong rubber-matrix interface to form. Riew *et al.*<sup>11</sup> and Siebert *et al.*<sup>21</sup> did not observe toughness improvements using non-reactive rubbers and the present data agree with their results (cf. dashed line in Figure 2). However, Ting *et al.*<sup>14</sup> and Bascom *et al.*<sup>15</sup> obtained good increases in fracture resistance using non-reactive liquid and solid rubbers.

SEM micrographs of fracture surfaces of blends cured at 60°, 100° and 140°C are presented in Figures 8, 9 and 10. The specimens were impacted at room temperature and there was no evidence of stress whitening. The micrographs show an increase in the dimensions of the rubber particles with increasing the cure temperature. At 60°C the phase separation is not clear cut and very small rubber particles, with dimensions of ~1 μm, have been observed.

On the contrary, at 100°C the phase separation is complete, with particle diameters up to 3 μm; the fracture toughness shows a definite increase above this temperature. A further increase in cure temperature brings about an enlargement of the particles whose dimensions reach 5 μm at 140°C. The increase in both fracture resistance, Figure 7, and particle dimensions does not necessarily imply that the former is to be attributed only to the latter, although it agrees with data published by Sultan and McGarry<sup>7</sup>. Also the modifications of the properties of the matrix must be taken into account. For instance, the unmodified resin cured at 60°C does not show any yielding in tension and breaks at ~75 MPa, whereas it yields at about 65 MPa, showing a certain degree of strain softening, if cured at 120°C. Matrix modifications could also be promoted by the rubber itself and, in order to check this point, shear moduli of both unmodified resin and 10% ATBN blends, cured in different conditions, have been measured as function of temperature. The  $T_g$  values derived from the thermomechanical curves indicate that the presence of ATBN causes the formation of a looser network structure only at high temperature, Figure 11. However, this is a minor effect, most of the decrease in  $T_g$  being attributable to the temperature of cure.

The influence of several factors such as rate of nucleation, domain growth and quenching due to gelation have been studied by Manzione *et al.* in detail<sup>19</sup>. They have pointed out that the kinetics and thermodynamic factors involved in the phase separation are similar to those controlling the crystallization of linear polymers. Thus, a plot of the volume fraction of precipitated rubber vs. temperature of gelation exhibits a maximum whose location on the temperature axis depends on blend formulation. Several authors<sup>13,16,23</sup> have shown that the precipitated particles are composite in nature, and this leads to increasing the volume fraction of the dispersed phase and to magnifying its efficiency. Bucknall and Yoshii<sup>13</sup> were able to establish that a linear relation exists between  $G_{IC}$  and rubber phase volume. The dependence of

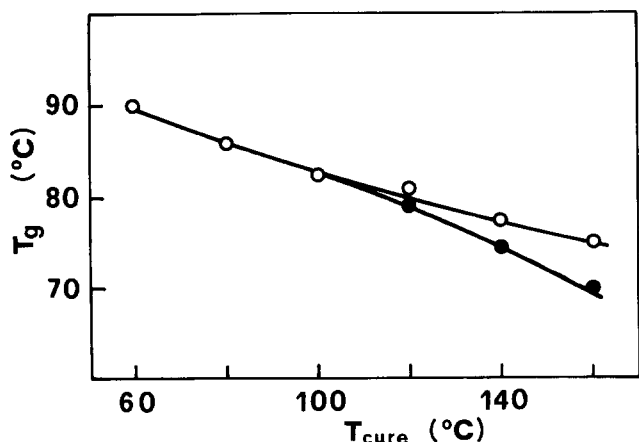


Figure 11 Transition temperature vs. cure temperature for (●) 10% ATBN blend, (○) pure resin

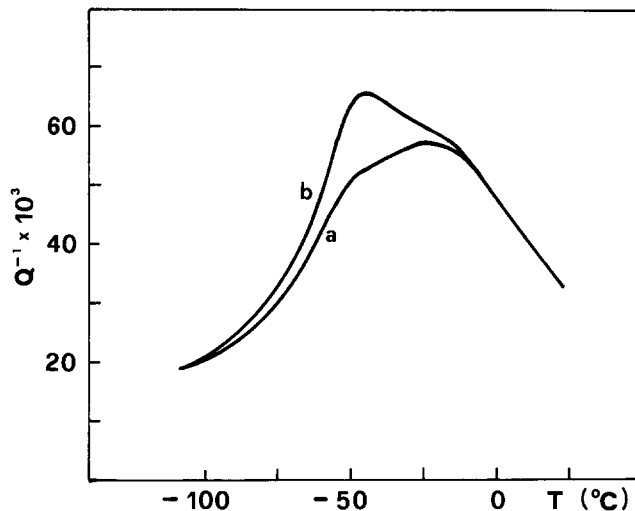


Figure 12 Dissipation factor vs. temperature for 10% ATBN blends; (a) homogeneous blend, (b) heterogeneous blend ( $T_{cure}$  120°C)

fracture energy on the precipitated phase volume and the dependence of the latter on the temperature of gelation account for the trend shown in Figure 7. Manzione *et al.*<sup>19</sup> have studied the phenomenon of phase separation in the system CTBN 1300X15/Epon 828/Piperidine (this reactive rubber has an acrylonitrile content of 10%, the same as the ATBN used in this work. This parameter is known to influence the phase separation<sup>3</sup>). They found that the maximum of rubber phase volume occurs at 120°C, a value that coincides with the temperature at which maximum toughness was observed in this work, Figure 7.

The toughening mechanisms first proposed to account for the increased fracture resistance of epoxy blends were the same known to be active in thermoplastic systems, i.e. shear banding and crazing. The difficulty of accepting a large scale crazing in matrices that are not craze prone can be overcome admitting the occurrence of voiding processes due to matrix stretching and cavitation around the rubber particles<sup>7,23</sup>.

Kunz-Dougllass *et al.*<sup>24</sup> have rejected such mechanisms and have proposed a model based on the absorption and dissipation of energy by stretching and tearing the rubber particles as the crack opening increases. This model allows a quantitative analysis to be done but does not explain the intense stress whitening which develops when the blends are tested in tension at low rates. Stress whitening is recognized to be a dissipative process and some experimental facts suggest it is caused by dilatational effects. For instance, the Rockwell hardness of a stress whitened piece is lower than that of unstrained material (a Rockwell test consists of pressing a spherical indenter onto the surface of the test piece). Moreover, the material beneath the indenter loses its whiteness and goes back to the initial colour. The simplest way to account for these phenomena is to admit the formation of microvoids in the matrix or at the interface which close up when the material is subjected to pressure. The occurrence of dilatational phenomena during the deformation of CTBN/epoxy blends has been demonstrated by Bucknall and Yoshii<sup>13</sup> by means of volumetric creep measurements.

The model proposed in ref. 24 assumes the dispersed rubber capable of high elongation so that its molecular weight needs to be high. Chain extension reactions can

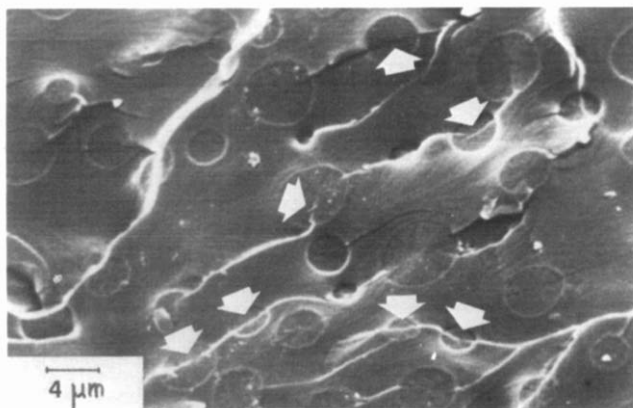


Figure 13 SEM micrograph of a 10% ATBN blend cured at 140°C

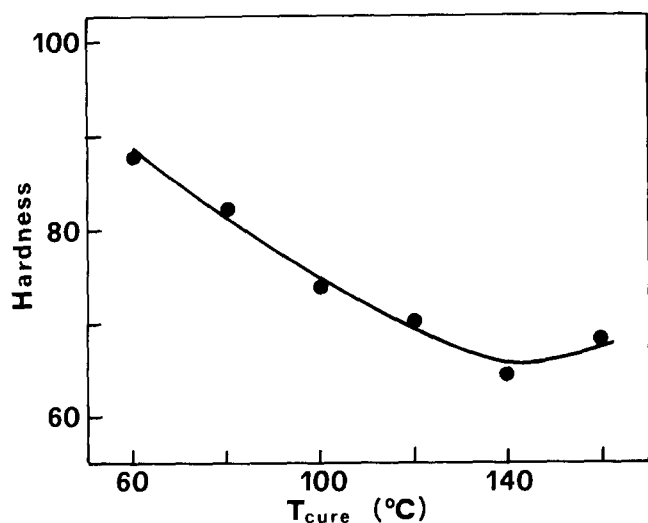


Figure 14 Rockwell hardness (HRM) as a function of cure temperature for 10% ATBN blends

occur between ATBN and epoxy resin, which results in a certain amount of resin trapped within the particles. The consequent increase in the apparent volume fraction of the rubber is accompanied by an increase in the magnitude of the loss peak at the glass transition of the rubber phase. The phenomenon is well known in thermoplastic systems<sup>25</sup> and also in rubber toughened epoxy resins<sup>13</sup>. The properties of ATBN/epoxy elastomers strongly depend on the relative amounts of the two components. For instance, in the system ATBN/Epon 828/DMP-30 the elongation at break is high ( $\epsilon_b = 770$ ) when  $ER = 0.85$ , but becomes much smaller ( $\epsilon_b = 180$ ) when  $ER = 0.60$ <sup>26</sup>. A rough estimate of the amount of resin present within the particles of 10% ATBN/epoxy blends cured at 120°C can be made by comparing the increase in peak value of  $Q^{-1}$ , Figure 12, with data reported in ref. 13. A value of about 20% for the volume fraction of the precipitated phase could be appraised, corresponding to an amount of epoxy resin of about 1/3 on the volume of the particles. The ratio of equivalents would then be about 0.35 and the extensibility of a copolymer of this composition is expected to be limited. Against the hypothesis of a two step process ((1) the matrix breaks and the distance between the fracture surfaces increases; (2) the rubber particles are stretched and broken) there is some micrographic evidence. It can be seen in Figure 13 that in impact tests the particles have neither stopped the cracks nor induced evident plastic damage around them. The arrows point to fracture lines

that have entered the particles, suggesting that the passing cracks went straight through them and that the rupture of the two phases occurred simultaneously. The stress field around the rubber particles is very complex, being influenced not only by their geometry or by the difference in elastic moduli, but also by interactions among the particles and volume changes brought about by the difference in thermal expansion coefficients. All such factors have been recently analysed by Kinloch *et al.*<sup>27,28</sup> who have proposed a novel toughening mechanism based on void formation within the particles (cavitation) at the particle/matrix interface is however admitted) and by blunting of the crack tips caused by an extensive shear yielding which would be produced as a consequence of the particle cavitation.

Figure 14 shows the dependence of the Rockwell hardness on cure temperature and stresses the influence of cure condition on the overall mechanical behaviour of rubber/epoxy blends. Interestingly, the hardness has a minimum at the same temperature at which  $K_{IC}$  has a maximum.

## CONCLUSION

The mechanical properties of ATBN/epoxy blends of various rubber contents, cured in a wide range of temperatures, have been studied. The morphology of these blends is strongly influenced by the cure temperature and either optically clear or opaque materials can be obtained, displaying very different mechanical behaviour. However, thermal and dynamic properties suggest a two phase structure, irrespective of the optical appearance. The fracture toughness changes by a factor of about 2.7 by varying the cure conditions and it has been shown that the addition of the rubber (ATBN) does not necessarily cause an increase in fracture resistance. Thus, to a certain extent, the cure conditions are more important than the amount of rubber. Also hardness measurements confirm this conclusion. The increase in  $T$  cure results in an increase in both particle size and matrix plasticity. Finally, it has been shown that the rubber particles lack ductility at impact rates.

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