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Impedance spectroscopy of highly cross-linked epoxy resin/bisphenol A-based polycarbonate

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Abstract

This study deals with the effect of temperature and frequency on the electrical properties of highly cross-linked epoxy resin/bisphenol A-based polycarbonate. Impedance measurements were carried out on neat resin and on blend containing 10 and 15 wt% of polycarbonate in the frequency range $10–10^6$ Hz at 25°C and 100°C. It was found that the impedance, dielectric constant are stable and nearly constant in the measured frequency and temperature range. The plot of the imaginary component of the impedence Z'' vs frequency at 100°C shows a relaxation peak in the blend which was interpreted as a local mode motion of the hydroxyl group formed during the high temperature dissolution process.

Keywords: Impedance; Blend; Dielectric constant

1. Introduction

Chemical alloying and physical mixing of different components into blends and composites are widely used throughout the electronic industry as insulators fixed in printed circuit boards [1–4]. Whence the electrical insulation is obviously an important property in choosing the appropriate polymeric blend or composite for a specific electronic application. The insulating and mechanical properties of blends and composite materials can be modified by careful control of structure and composition. To achieve this objective a deeper understanding of the dielectric properties usually governed by several processes including AC and DC current condition, dipolar relaxation, interfacial polarization and side group motion. All these factors influence the whole dielectric response of an epoxy blend [5–9].

Recently, high cross-linked epoxy resin was successfully toughened in the C.N.R. laboratories in Naples (Italy) by reactive blending with bisphenol A-based polycarbonate [10,11]. The researchers found that the fracture toughness of this blend system increases markedly with increasing the polycarbonate content in the blend. Further investigation of this reactive system by FTIR spectroscopy demonstrated the occurrence of physical and chemical interaction among the blend components. The authors found that the molecular structure of the two components epoxy/polycarbonate in the form that polycarbonate chains are incorporated with the epoxy network during reaction, forming a single phase homogenous system. Thus, a desire was born to report the dielectric characterization for this novel blend system.

This communication which is one of a series of published papers [12–14] dealing with the electrical characteristics of polymeric materials report the results of impedance measurements in the temperature range 25°C–100°C and frequency range $10–10^6$ Hz for three compositions of cross-linked epoxy/bisphenol A-based polycarbonate.

2. Experimental

2.1. Materials preparation

The materials used in this study were prepared at C.N.R. laboratories in Naples, Italy as follows: 20 g of polycarbonate ($M_n = 12 \times 10^3$ and $M_w = 31 \times 10^3$) was dissolved in 39.6 of epoxy resin, Novolac DEN 438 for 5 h at 220°C. The temperature of the mixture was allowed to decrease to 80°C and 40.4 g of Nadic Methyl Anhidride (MNA) and 0.4 g of the accelerator

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Table 1
Composition wt% of the epoxy/polycarbonate (PC) blends

PC	Epoxy	MNA	Epoxy + MNA
0	49.5	50.5	100
10	44.5	45.5	90
15	41.5	43.5	80

Benzyl Dimethylamine were added. The curing process was carried out at 120°C for 20 h. After these processes, a visually transparent sheet was recovered. The measurements were performed on three compositions as shown in Table 1.

2.2. AC impedance measurements

The AC impedance measurements were carried out in the temperature range 25°C–100°C and in the frequency range 10 Hz–10^6 Hz, using a Solarton (Schlumberger Instrument) apparatus. The detailed description of the measurements were reported in a previous paper [15].

3. Results and discussion

The general feature of the variation of the real component of AC impedence (Z') for the three concentrations of PC as shown in Fig. 1(a,b,c) is the same with a slight increase in the value of Z' with increasing the PC content. At frequencies less than 10^4 Hz a rapid decrease with increasing frequency was observed for all specimens. The decrease rate is more pronounced in the sample containing 15% of PC. The high impedance value at low frequencies may be because of space charge polarization in the bulk material, structural defects and electrode effects [7]. However, the AC impedance of the neat resin and the blend with 10 wt% and 15 wt% of PC is nearly constant in the temperature range 25°C–100°C. It can be seen by comparing Fig. 1(a,b,c) and Fig. 2(a,b,c) that the values of Z' in the measured frequency range are nearly the same. This behaviour indicates that the electrical insulation for such a blend is relatively stable in the measured temperature range.

The plot of Z'' vs frequency for neat resin and blends as shown in Fig. 3(a,b,c), show a small relaxation peak in the sample containing 10 wt% of PC, the relaxation peak becomes a well defined peak for the sample containing 15 wt% of PC. However, the absence of the relaxation peak in neat resin samples suggests that the mechanism of the relaxation process may be related to side chain groups formed during the curing process. On the other hand, the study of the dynamic mechanical behaviour for both neat and blend [10] showed that the temperature relaxation peaks for the neat resin and blend coincide with each other at about

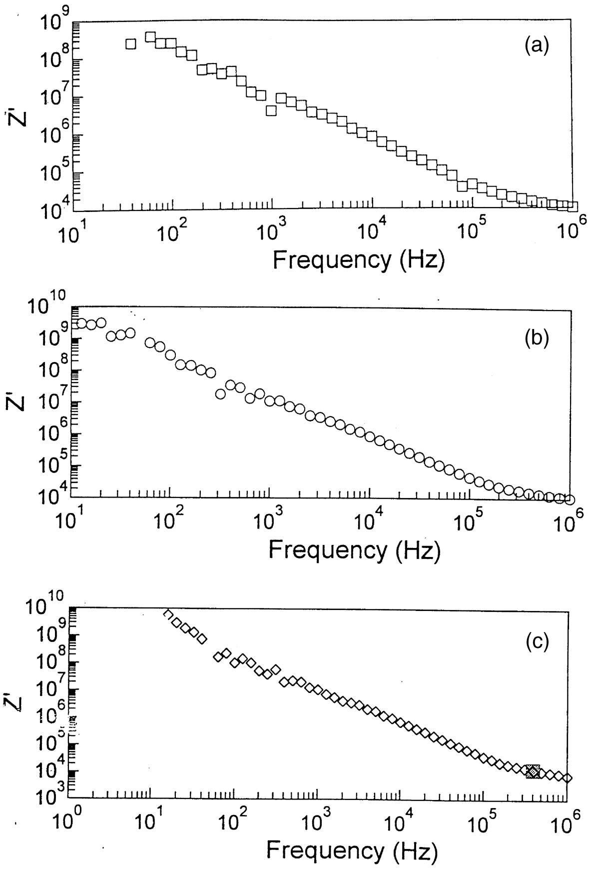

Fig. 1. The real components of AC impedance/unit length (ohm/m) vs frequency for: (a) neat resin; (b) blend containing 10 wt% of polycarbonate; and (c) blend containing 15 wt% of polycarbonate at 100°C.

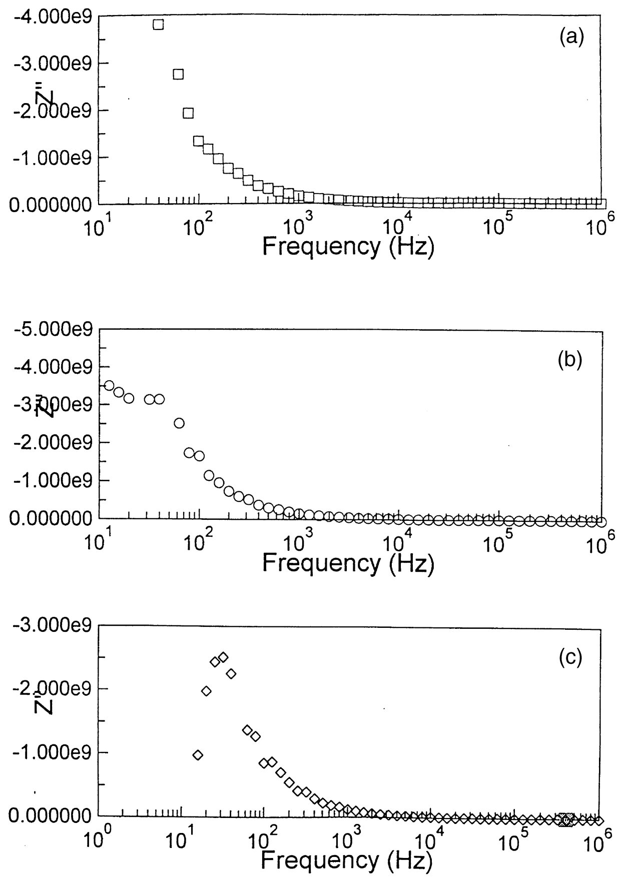

Fig. 2. The real components of AC impedance/unit length (ohm/m) vs frequency for: (a) neat resin; (b) blend containing 10 wt% of polycarbonate; and (c) blend containing 15 wt% of polycarbonate at 25°C.

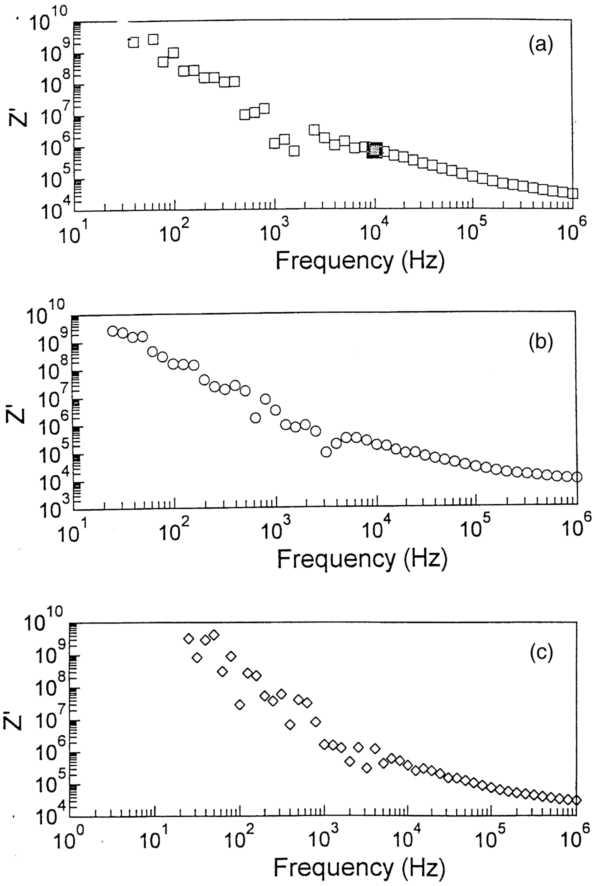

Fig. 3. The imaginary components of AC impedance/unit length (ohm/m) vs frequency for: (a) neat resin; (b) blend containing 10 wt% of polycarbonate; and (c) blend containing 15 wt% of polycarbonate at 100°C.

170°C which corresponds to the glass transition temperature. Therefore, we can conclude that the relaxation peak which was observed in the blend at about 100°C is associated with local mode motion of the hydroxyl group on the polycarbonate chain which was formed during the high temperature dissolution process [10].

The dielectric constant for neat resin is nearly independant of temperature and frequency in the measured range of frequency and temperature with the value of 9. However, the increase in dielectric constant for epoxy resin/bisphenol A-based polycarbonate increases slightly with increasing the PC content as given in Table 2, indicating high electrical stability for such a material.

4. Conclusion

The present study of the AC electrical properties of neat resin and highly crossed epoxy resin/bisphenol A-based polycarbonate as a function of frequency and temperature indicate that the AC electrical properties of neat resin and blends is stable in the temperature range 25°C–100°C.

Table 2
Dielectric constant for epoxy resin/bisphenol A-based polycarbonates at 25°C and 100°C at different frequencies

PC	10^6 Hz	10^3 Hz	10^2 Hz
25°C			
0	8.4	8.9	8.9
10	8.3	8.6	9.1
15	9.2	9.6	9.8
100°C			
0	8.6	9.2	9.6
10	9.2	9.8	10
15	9.8	10.0	105

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