Mechanical and Morphological Properties of Polycarbonate and Montmorillonite Filled Epoxy Hybrid Composites

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ABSTRACT: This work investigates the effect of polycarbonate (PC) and montmorillonite (MMT) content on the properties and morphology of epoxy resin (EP). Izod impact strength (IS), flexural strength and critical stress intensity factor (K_C) were estimated as function of modifiers content. The values of IS and K_C parameters increased by respectively 150% and 90% with the addition of 5 wt % PC. Hybrid compositions containing 1 wt % MMT and 5 wt % PC exhibited the best mechanical properties. Indeed, the addition of 1 wt % MMT to EP modified with 5 wt % PC caused enhancement of IS values by 100% in comparison with neat EP. SEM micrographs revealed that the enhancement mechanism of mechanical properties might be due to extensive yielding of EP associated with the formation of stratified elongated structures. Moreover, differential scanning calorimetry analysis revealed that the addition of nanoclay to EP resulted in a decrease of the glass transition temperature. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 752–759, 2011

Key words: epoxy resin; polycarbonate; montmorillonite; impact strength; fracture toughness

INTRODUCTION

Epoxy resins (EPs) are widely used as matrices for high performance composite materials, surface coatings, and adhesive joints. However due to their highly cross-linked density, these materials exhibit low impact strength (IS), poor resistance to crack propagation and small elongation at break, i.e., they are inherently brittle.

In the last few decades, a great deal of effort has been devoted to the improvement of both the fracture toughness and elasticity of EP. Approaches to improve EP toughness included the incorporation of solid particles[1–3] and addition of thermoplastics such as polysulphone,[4] polyethersulphone,[5,6] polyetherimide,[7] polyamide,[8] polyimide,[9] and polycarbonate (PC).[10–13] These systems were studied with the view to overcome the deterioration of EP properties at high temperatures experienced with the incorporation of reactive liquid rubbers, such as butadiene-acrylonitrile copolymers terminated with carboxyl, amine, hydroxyl or epoxy groups.

In their study, Rong and Zeng were able to modify diglycidyl ether of bisphenol-A (DGEBA) by physical and chemical means using PC.[10] The resin was crosslinked with various hardeners under different curing conditions. The phase separation in the blend PC/EP was controlled by varying the modifier content and using curing agents with different reactivity. However, no fracture tests were carried out to determine the effects of the resulting morphology.

In a recent work, PC has been used as a modifier for DGEBA.[12] It was found that the result of PC addition was the improvement of elastic modulus and twofold increase of the IS.

In fact, in the last decade an extensive amount of work was devoted to the modification of EPs with montmorillonite (MMT) to produce nanocomposite materials. Different types of nanofillers and operating conditions were investigated in the production of epoxy nanocomposites.[14–21]

For instance, Yilmazer and coworkers studied the effect of type and content of nanoclays (natural Cloisite Na^+ and organically modified Cloisite 30B) on the properties of DGEBA.[14] Their results showed an increase of the glass transition temperature by more than 10°C with the addition of 9 wt % of modified Cloisite 30B. The addition of 0.5 and 1 wt % of Cloisite 30B resulted in improvement of IS of the EP, toping to about 140%. The tensile strength also exhibited a maximum value at 1 wt % modified clay loading. Moreover, it was confirmed that the addition of Cloisite Na^+ resulted in lower mechanical properties improvement in comparison with modified clay.

The effect of MMT treatment on the reactivity, the cure behavior, the morphology and the mechanical properties of the epoxy networks were investigated by Sautereau and coworkers.[15] They noticed that the clay

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exfoliation was responsible for the increase of the elastic modulus. They also stipulated that intercalated structures could result in a good balance of properties, such as stiffness and toughness, without inducing any reduction in the glass transition temperature.

Messersmith and Giannelis prepared exfoliated layered silicate epoxy nanocomposite based on DGEBA cured with nadic methyl anhydride in the presence of benzyldimethylamine.[16] They obtained a significant increase in stiffness of the epoxy nanocomposite using 4 wt % of organoclay modified with bis(2-hydroxyethyl)methyl tallow-alkyl ammonium chloride.

The mechanism of the exfoliation process was also analyzed by Lan et al., who have prepared nanocomposites based on DGEBA cured with *m*-phenylenediamine.[17] They demonstrated that the exfoliation of the clay was not only dependent on the reactivity of epoxy system but also on the accessibility of the matrix and the curing agent to the clay galleries. Moreover, the exfoliation was facilitated by the acidic exchange of cations.

The effect of aliphatic and cycloaliphatic diamines on the structure of nanocomposites based on a diglycidyl ether of bisphenol A was studied by Kornmann et al.[18] The results of their investigations showed that the extent of exfoliation of the clay depended on the type of curing agent as well as the curing conditions. They suggested that the aliphatic diamine allowed a larger amount of curing agent to diffuse into the galleries, which led to a more effective exfoliation.

The purpose of the present work was a mere investigation into the effect of the addition of a combination of PC modifier and MMT nanofiller on the extent of improvement in the fracture toughness of EP.

EXPERIMENTAL

Materials

The following ingredients were used in this work: diglycidyl ether of bisphenol A: Epidian 5 with $M_w = 381$ g/mol and epoxy number = 0.49–0.52 mol/100 g produced by Organika-Sarzyna, Poland; curing agent: triethylenetetramine (trade name Z1), from Organika-Sarzyna, Poland; MMT: nanoclay modified with methyl tallow bis(2-hydroxyethyl) quaternary ammonium chloride (Cloisite 30B from Southern Clay Products) was selected for the present study; PC: Lexan produced by Sabic IP.

Samples preparation

The samples were prepared with EP and different amounts of modifier.

In the case of PC based formulations, polymer pellets were dissolved in dichloroethane to obtain a 15% solution. Then the solution was mixed with EP for 5 min using a mechanical stirrer to yield compositions containing different PC contents (2.5, 5, and 7.5 wt %). Next, the solvent was evaporated under low pressure using rotation Büchi apparatus.

For MMT based formulations, the modifier was first dispersed in acetone to form 10% solutions. Next, the dispersion was added to EP to obtain different concentrations (1, 2, and 3%). The solvent was evaporated from the compositions by heating at 40° C.

Two sets of hybrid compositions were prepared: hybrid compositions based on EP containing 1 wt % MMT and different amounts of PC as well as compositions with 2 wt % MMT and containing 2.5–7.5 wt % of polymeric modifier. The preparation was carried out as follows: a specific amount of EP was mixed with MMT, then PC was added to the solution and the obtained composition was heated under vacuum to remove the solvent. Finally, the hardener was added followed by mixing for 5 min. The obtained mixture was then poured into teflon coated plates with adequate sample geometries (80 mm × 10 mm × 4 mm) before the testing of solid state mechanical properties.

Curing was carried out at 80°C for 1 hour and at 120°C for 3 h. After removal from the mold, the samples were kept in a desiccator for 3 days before testing.

Properties evaluation

- The IS was estimated using Zwick 5012 apparatus and the Izod method in accordance with the Polish Norm PN-87/C-89050 on samples having 8 cm in length, 1 cm in width, 4 mm in thickness, and 1 mm of crack length.
- The critical stress intensity factor (K_C) of the obtained formulations was evaluated under three point bending mode on single edge notched specimen (80mm x 10mm x 4 mm) and the following equation[19]

$$K_C = \frac{3 \cdot P \cdot L \cdot a^{1/2}}{2 \cdot t \cdot w^2} \cdot Y \tag{1}$$

where: P – critical load for crack propagation [N], L – distance between the spans [m], a – the precrack length [m], w – specimen width [m], t – specimen thickness [m], Y – geometrical factor expressed by the following formula:

$$Y = 1.93 - 3.07 \cdot \left(\frac{\mathrm{a}}{\mathrm{w}}\right) + 14.53 \cdot \left(\frac{\mathrm{a}}{\mathrm{w}}\right)^2 - 25.11 \cdot \left(\frac{\mathrm{a}}{\mathrm{w}}\right)^3 + 25.80 \cdot \left(\frac{\mathrm{a}}{\mathrm{w}}\right)^4 \tag{2}$$

TABLE I
Properties of Epoxy Based Compositions Containing Different Amount of Polycarbonate

Polycarbonate content (wt %)	Impact strength (kJ/m^2)	K_C (MPa.m$^{1/2}$)	Stress at break (MPa)	Strain at break ($\times 10^2$)	Energy at break (kJ/m^2)
0	2.35 ± 0.05	1.7 ± 0.1	52 ± 2	3.1 ± 0.4	7.7 ± 0.7
2.5	5.1 ± 0.1	2.5 ± 0.2	110 ± 10	3.30 ± 0.35	16.0 ± 1.5
5	6.0 ± 0.2	3.20 ± 0.15	61 ± 6	3.6 ± 0.4	13.3 ± 1.2
7.5	7.70 ± 0.15	2.2 ± 0.1	42 ± 5	3.1 ± 0.2	5.2 ± 0.6

The tests were performed at room temperature using an Instron 5566 tensile machine with a strain rate of 5 mm/min and 6 cm of distance between the spans.

- The flexural test was carried out at room temperature on unnotched samples with the same dimensions and under similar testing conditions as for K_C parameter. The flexural strength, strain and energy were calculated for each obtained composition. Five samples were tested for each data point.

Morphological and thermal characterization

Scanning electron microscopy was used to examine the fracture surfaces for further insight into the toughening mechanism. The specimens obtained from the impact tests were prepared and examined using JSM-6490LV scanning electron microscope.

Various samples were also subjected to thermal analysis using TA instrument DSC with heating scan of 10°C/min and over the temperature range between –50 and 250°C, in a stream of nitrogen.

RESULTS AND DISCUSSION

Tables I and II present the mechanical properties coefficients of compositions containing PC and MMT respectively. It can be noted that as the amount of PC increases, both fracture toughness parameters (Izod IS and K_C) increase almost linearly up to 5 wt % PC.

The K_C reaches a maximum value with 5 wt % PC, which represents 90% increase in relation with K_C value of neat resin (EP). Moreover, a maximum of about 150% increase of IS was obtained with 5 wt % of PC. However, we noted that IS of composition containing 7.5 wt % PC was higher by ~ 230% as compared with IS of nonmodified EP.

Furthermore, with only 1 wt % MMT, we observed maximum increase of K_C value by about 90%, flexural strength by 30% but almost no increase of IS. However, the addition of 2 wt % MMT led to maximum IS enhancement (100% in comparison with IS of nonmodified EP).

The improvement of the resistance to crack propagation (as expressed by K_C or IS values) of EP modified with PC might be attributed to the fairly high toughness of this latter at room temperature. It has to be mentioned that the polymer exhibits a quite wide transition region at room temperature specific to its high capacity of energy absorption during the fracture process (i.e., its high fracture toughness).

However, considering Table I which represents the mechanical parameters values as function of PC content, one can note a very small increase of the flexural stress at break (~ 15%) opposed to a quite significant increase of flexural energy at break (about 70%) with 5 wt % PC compared to neat EP. The energy at break was evaluated from the area under the load-displacement curve up to the point of fracture. Moreover, the composition containing 2.5 wt % PC reached maximum values of the stress at break and energy at break corresponding to 100% in comparison with that of neat resin. The enhancement of the energy at break might be directly associated with the stress at break improvement of the tested composition.

TABLE II
Properties of Epoxy Based Compositions Containing Different Amount of Montmorillonite

Montmorillonite content (wt %)	Impact strength (kJ/m^2)	K_C (MPa.m$^{1/2}$)	Stress at break (MPa)	Strain at break (x10^2)	Energy at break (kJ/m^2)
0	2.35 ± 0.05	1.7 ± 0.1	52 ± 2	3.1 ± 0.4	7.7 ± 0.7
1	2.45 ± 0.05	3.25 ± 0.15	67 ± 7	2.3 ± 0.3	7.2 ± 0.5
2	4.7 ± 0.2	2.20 ± 0.15	61 ± 5	2.40 ± 0.25	5.4 ± 0.4
3	2.6 ± 0.1	2.3 ± 0.1	54 ± 6	2.8 ± 0.3	5.2 ± 0.3

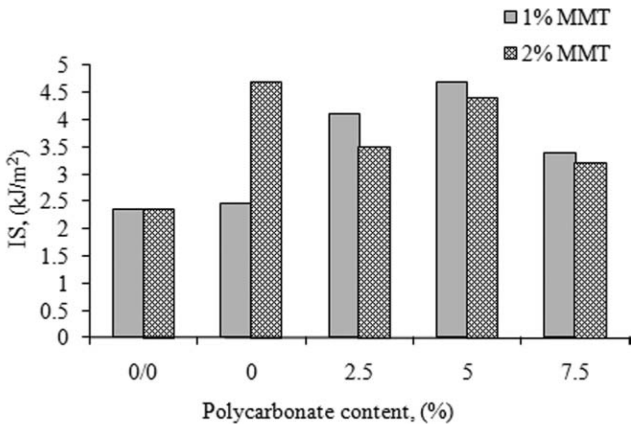

Figure 1 Effect of polycarbonate content on the impact strength (IS) of hybrid compositions containing 1% and 2 wt % montmorillonite (MMT).

The Izod IS, K_C, stress at break and energy at break values as function of MMT content are summarized in Table II. Maximum values are observed with 1 wt % and 2% MMT except for the strain at break and the energy at break. It can be noticed that the addition of 1 wt % MMT to EP gives maximum value of the K_C while adding 2 wt % MMT results in a composition with the best IS. Furthermore, for samples with only 1 wt % MMT, we noted maximum increase of flexural strength by 30% as compared with neat epoxy composition.

Unexpectedly, compositions containing nanoclay exhibited lower flexural strain at break and energy to break than neat EP. The decrease of these parameters induced by nanoclay incorporation might be attributed to the aggregates of MMT particles, which act as stress concentration.

Results of EP compositions modified with 1% and 2 wt % MMT for Izod IS as function of PC content are presented in Figure 1. The composition with 0/0% PC designates the unmodified EP. It can be noted that all tested formulations exhibit higher resistance to impact compared to nonmodified EP. Indeed, maximum IS enhancement was obtained with EP based hybrid composition containing 5 wt % PC and 1 wt % MMT as well as epoxy composition containing 2% MMT without PC. The IS of this latter increased by about 100% in relation with neat cured EP. However, no synergism effect was observed with tested hybrid compositions.

Furthermore, it can be noted that hybrid compositions containing 2 wt % MMT and PC are characterized by lower IS than composition with only 2 wt % MMT but without polymer modifier. This might be regarded as a consequence of the poor adhesion between both modifiers and the agglomeration of MMT particles at higher nanoclay loading.

As it is shown in Figure 2, the K_C values of hybrid compositions containing respectively 1 and 2% MMT and different amounts of PC were higher than those of neat EP (0/0), similarly to IS results (Fig. 1). In addition, maximum K_C value is exhibited by the hybrid compositions containing 1 wt % MMT and 5 wt % PC.

The improvement of the IS and resistance to crack propagation can be explained by the good dispersion of the nanofiller within the polymer matrix and the second modifier, but also by the intercalation of EP between silicate layers.

Mulhaupt and coworkers demonstrated that the enhancement of the fracture toughness parameter K_C of EP was mainly attributed to the intercalated nanoclay.[20] They stipulated that nanoparticles subjected to stresses were able to produce voids and initiate shear yielding at the tip of the propagating crack, increasing thus K_C parameter. The improvement was associated with energy-absorbing and shearing of intercalated of the nanoclay layers. Moreover, the silicate layers can act through a crack pinning mechanism.

Therefore, it seems evident that the addition of 5 wt % of polymeric modifier and 1 wt % of MMT allows to prepare hybrid composition possessing higher resistance to high speed, as well as low speed crack propagation (as estimated respectively by impact tests and expressed by K_C parameter).

The stress at break measured under three point bending of epoxy compositions modified with 1 wt % and 2 wt % MMT as function of PC content is presented in Figure 3. It can be observed that the highest flexural strength is obtained with the composition containing 5 wt % PC and 1 wt % MMT. All other hybrid compositions with 1 wt % MMT have, within experiment error, comparable values as the pristine epoxy samples.

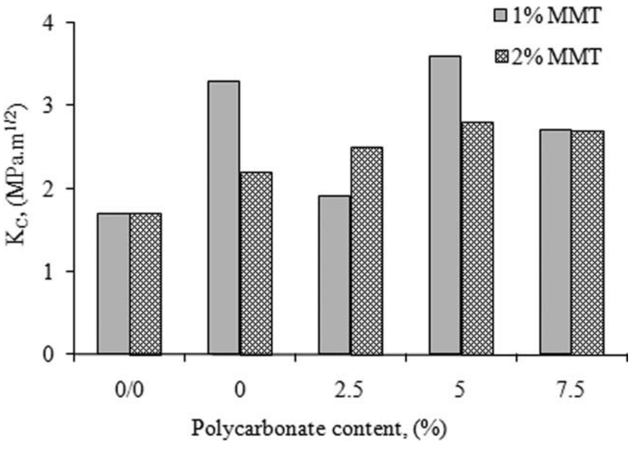

Figure 2 Critical stress intensity factor (K_C) of hybrid compositions containing 1 and 2 wt % montmorillonite (MMT) and different amount of polycarbonate.

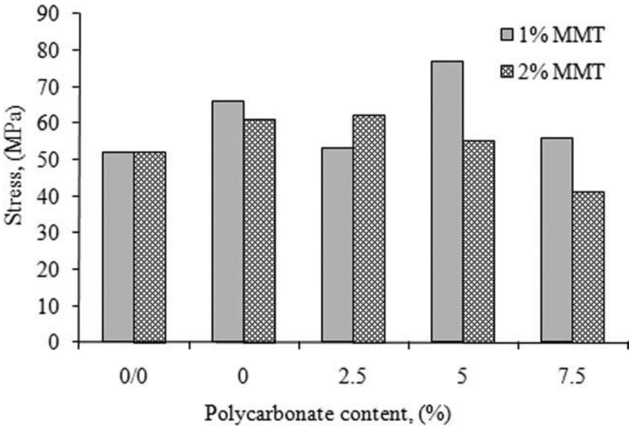

Figure 3 Flexural strength of hybrid compositions containing 1% and 2 wt % montmorillonite (MMT) and different amount of polycarbonate.

The incorporation of PC to EP modified with 2 wt % of MMT resulted in a decrease of the flexural strength of the hybrid compositions except for 2.5 wt % PC. Such behavior can be attributed to a poor adhesion between the polymer modifier and MMT at higher loadings of this latter as well as higher stress concentration effect brought out by the nanoclay agglomerates. In fact, the addition of 7.5 wt % PC to epoxy composition modified with 2 wt % MMT leads to ~ 20% decrease in flexural strength in comparison with neat EP.

It is reasonable to summarize that these amounts of nanoparticle (1 wt % MMT) and polymeric modifier (5 wt % PC) are found to be optimal to prepare hybrid composition with maximum mechanical properties improvement.

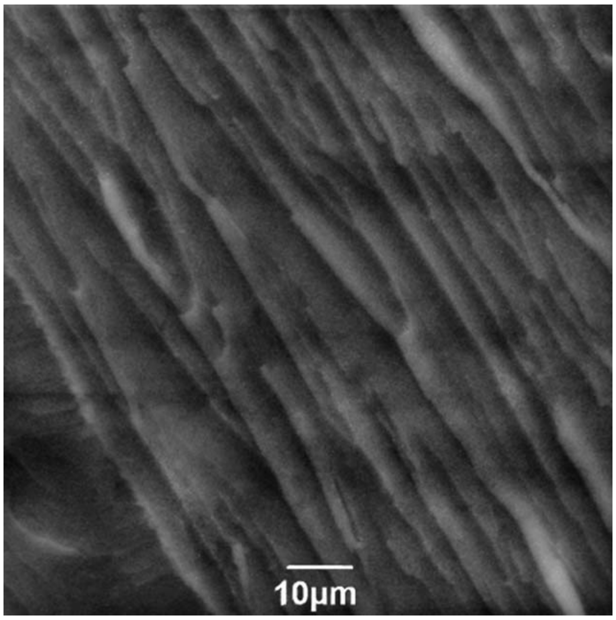

Figure 4 SEM micrographs of unmodified epoxy resin.

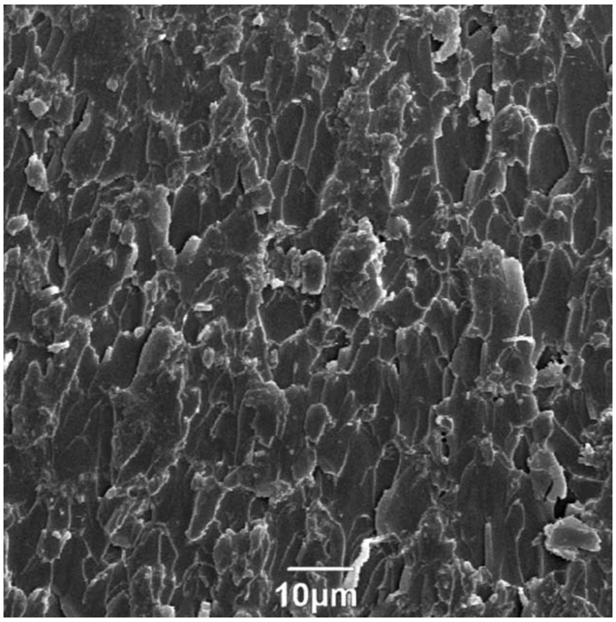

Figure 5 SEM micrographs of epoxy composition containing 1 wt % MMT (magnification ×1000).

Fracture surface morphological studies

SEM micrographs obtained from fracture surfaces of pristine and nanocomposite samples allow elucidating the toughening mechanism induced by nanoclay incorporation. The micrograph of unmodified epoxy composition fracture surface (shown in Fig. 4), indicates a regular uninterrupted crack propagation path. Generally, optimally cured EP samples without modifier exhibits a glassy structure with no plastic

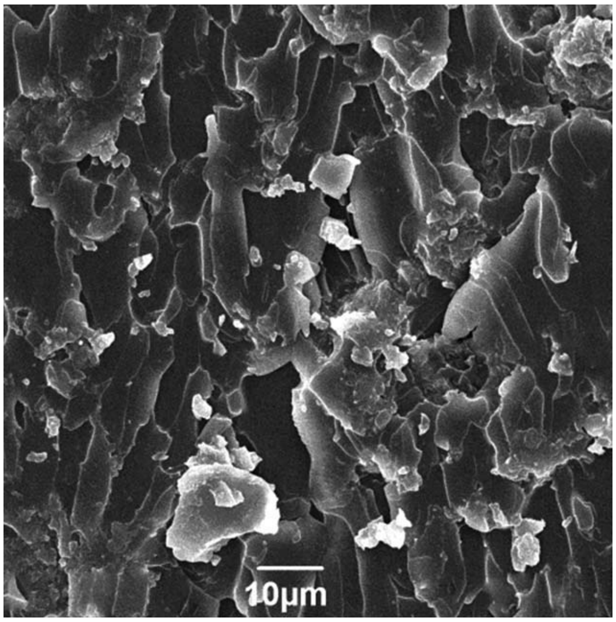

Figure 6 SEM micrographs of epoxy based composition with 2 wt % MMT.

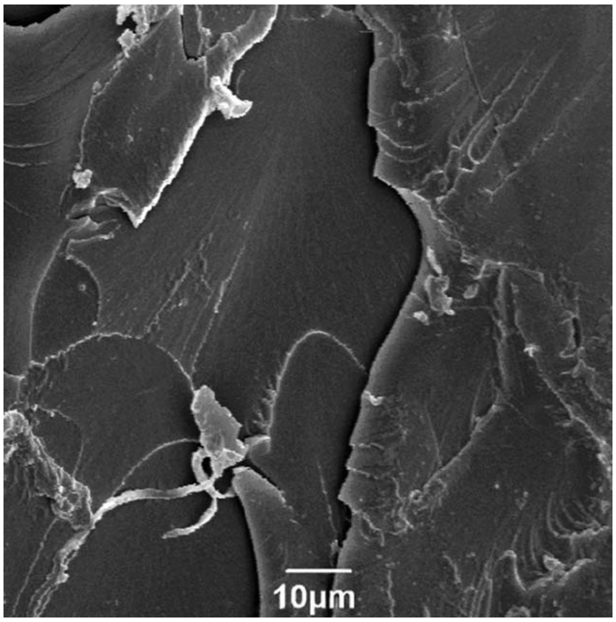

Figure 7 SEM micrographs of epoxy resin containing 5 wt % PC.

deformation indicating rather poor resistance to crack propagation.

It should be mentioned that all SEM micrographs were taken near the crack tip on fractured surfaces after impact tests.

Micrographs of compositions containing 1 and 2 wt % MMT are presented respectively in Figures 5 and 6. One can observe rougher surfaces filled and homogeneous structures with more elongated as well as larger domains with composition having 2 wt % MMT. This might explain the relatively lower mechanical properties of composition containing 2% MMT in comparison with that containing only 1%.

The features showing some plastic deformation might be attributed mainly to the matrix, which will be associated with higher energy absorption during the crack propagation process. Thus the extensive shear yielding of the polymer matrix containing the nanoclay can be chosen as the main fracture toughness improvement mechanism. Other researchers attributed the fracture toughening of EP to the crack arrest and pinning.[21] While in other studies, massive shear yielding was taken as the main mechanism to explain epoxy toughening.[22]

Micrograph of epoxy composition with 5 wt % PC is presented in Figure 7, where a smooth surface with elongated material can be observed. PC seems to show good bonding with the polymer matrix. The toughening may arise from the chemical reactions that most probably took place between EP and the polymeric modifier which was partially solubilised in the resin matrix.

Micrographs of EP modified with 5% of PC and containing 1 and 2% of MMT are shown respectively in Figures 8 and 9. As it can be noticed from Figure 8, there is a homogenous structure with multilayer surfaces made of polymer domains containing MMT particles. This might explain the higher values registered with IS and K_C. It is indeed well accepted and documented in literature that the formation of

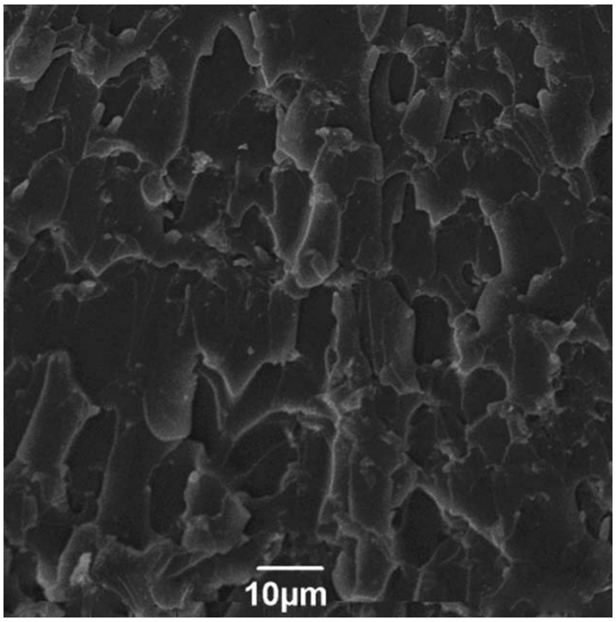

Figure 8 SEM micrographs of hybrid compositions containing 5% PC and 1 wt % MMT.

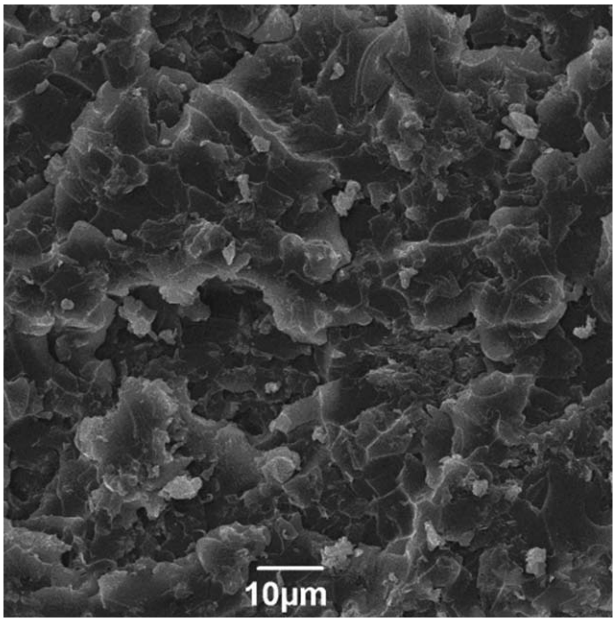

Figure 9 SEM micrographs of hybrid compositions containing 5% PC and 2 wt % MMT.

TABLE III
Glass Transition Temperatures of Compositions Containing Different Amount of Montmorillonite and Polycarbonate

Montmorillonite content (wt %)	Polycarbonate content (wt %)	Glass transition temperature (°C)
0	0	50
1	0	40
2	0	34
0	7.5	66
1	7.5	62
3	7.5	61

second phase within polymer matrix is associated with higher energy absorption during the crack propagation processes either under low speed (K_C) or high speed (IS) conditions.

However, the micrograph in Figure 9 seems to exhibit a quite different structure. One can observe a kind of more condensed stratified structure made from MMT particles surrounding the polymer matrix. The larger amount of MMT (2% in this composition) may create the smaller dimension of polymer domains. The SEM micrograph shows the phase separation, large amount of free volume between the domains, which could be the reason behind decreasing the fracture toughness. This hybrid composition exhibits lower values of both IS and critical intensity factor in comparison with hybrid composite containing 1% of MMT.

Thermal analysis

Further investigations of the cured epoxy based nanocomposites as well as pristine EP were carried out to check the occurrence of any chemical reaction and the possible shift of the glass transition temperature. From Table III showing glass transition temperatures of compositions containing MMT and PC, it can be noted that the addition of nanoclay to EP resulted in a decrease of the glass transition temperature (T_g). However, this latter was increased by the incorporation of PC to nonmodified or MMT modified EP.

The decrease of the glass transition temperature due to nanoclay addition was already reported by other research groups and related to the possible formation of interphase layer and plasticization of the polymer matrix.[23–25]

However, some other investigations indicated an increase of T_g and attributed mainly to the restriction of the polymer chain mobility and reduction in free volume.[14,26,27] An increase of about 10°C was observed by Basara et al. in an epoxy based nanocomposite with 9 wt % of Cloisite 30B.[14]

As it can be logically expected from the mentioned studies, the glass transition temperature of EP remained relatively unchanged with clay addition as reported by Balakrishnan et al.[28]

CONCLUSIONS

From the obtained results it is possible to conclude that the addition of PC and MMT led to a significant improvement of EP Izod IS, K_C as well as flexural strength and toughness. The addition of 5 wt % PC to EP resulted in increased IS and K_C parameters respectively by 150% and 90% in comparison with neat nonmodified polymer matrix. Whereas, more than twofold increase of IS was obtained with 7.5 wt % PC.

The K_C and IS were increased maximally by respectively 1 wt % and 2 wt % of nanoparticles addition corresponding to ~ 90% and 100% in comparison with nonmodified EP toughness.

Hybrid compositions containing specific amount of both polymeric modifier and nanoparticles, exhibited improvement of IS and resistance to crack propagation. In fact, hybrid composition based on 1 wt % MMT and 5 wt % PC was characterized by maximum enhancement of K_C, IS and flexural strength values.

SEM micrographs representing epoxy compositions with MMT show a stratified and elongated structure with the presence of plastic yielding. The mechanism of mechanical properties enhancement might be due to extensive yielding of EP as well as to some chemical reactions occurring between the nanoclay and the polymer matrix. However, DSC analysis showed a decrease of the glass transition temperature induced by nanoclay addition, which might be related to the possible formation of interphase layer and plasticization of the polymer matrix.

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