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Modification of fracture toughness of isotactic polypropylene with a combination of EPR and $CaCO_3$ particles

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The main goal of this study is to investigate the role of both ethylene-propylene rubber (EPR) and calcium carbonate ($CaCO_3$) on fracture toughness and fracture mechanism of isotactic polypropylene (iPP). To do so several hybrid composites with different ratios of EPR/$CaCO_3$ were examined. Three and four point bending tests were carried as well as microscopic evaluation. The results of three-point bending test show that plane strain fracture toughness (K_{IC}) of PP/$CaCO_3$ can be improved by addition EPR content. Since the trend of the fracture toughness values is nearly similar to what expected by the rule of mixtures, thus one may conclude that the existence of both $CaCO_3$ and EPR have not significance effect on synergistic toughening. The results of 4-point bending test show that the dominant mechanism of fracture in PP-$CaCO_3$-EPR studied in this work can be a craze-like type damage which is included with both highly localised dilatational shear bands due to EPR particles and some craze owning to $CaCO_3$.

1. INTRODUCTION

Polypropylene (PP) is the third most important plastic from a sales tonnage standpoint. Since PP can be synthesized from low-cost petrochemical raw materials it is one of the lowest in cost among commodity thermoplastics. Although PP is extensively used in many fields of applications, but its utilization has been limited because of its poor impact strength. One of the conventional methods for overcoming this drawback is blending with other polymers, especially with elastomers such as Ethylene-Propylene Rubber (EPR) [1-4]. However EPR particle diminish tensile properties of PP such as yield strength and Young's modulus. To compensate the effect of EPR content, addition of rigid fillers such as calcium carbonate ($CaCO_3$) is recommended [2-3]. The produced blends so called hybrid composites. The mechanical properties of hybrid composites are dictated not only by their composition, but also by their phase morphology [2-7]. According to the literature, two kinds of phase morphologies are normally found. Either the rubber particles and rigid particles are dispersed in the PP matrix separately, or the rigid particles are encapsulated by rubber particles [2-5]. Earlier work showed that the most important factor that can determine phase morphology is surface treatment of the rigid particles [6-7]. Most previous studies have focused on the relationship between phase morphology and mechanical properties in PP/rubber/ filler systems [3, 5-9]. To our knowledge, there is no known significance effort focusing on using three and four point bending tests to investigate the fracture mechanism of PP-$CaCO_3$-EPR hybrid composites. Thus the main goal of this research is to elucidate the fracture mechanism of PP-$CaCO_3$-EPR using three and four points bending test.

2. EXPERIMENTAL PROCEDURES

PP/20wt%$CaCO_3$ pellets with the average particle size 5 μm were obtained from Niroomand Polymer Company. PP/20wt%EPR pellets with the average particle size about 3.5 μm were produced from the Montell Company. Also pure PP (080) was incorporated by Imam Khomeini Petrochemichal Complex. To obtain ternary blends, the pellets were mixed and extruded in a single-screw extruder. To study possible synergistic effect, the total weight fraction of the additives was kept constant and pellets with $CaCO_3$/EPR ratios of 0/20, 5/15, 10/10, 15/5, and 20/0 were produced. Standard fracture specimens were made through injection molding. To determine fracture toughness, single edge notched 3-point bending test (3PB) was carried out. To elucidate the fracture mechanism of PP/$CaCO_3$/EPR 4-point bending test (4PB) was employed. An Olympus PME3 optical microscope was employed to examine the polished surfaces of 4-point bending specimens under bright and cross- polarized light conditions. A Jeol JSM-5600LV scanning electron microscope (SEM) was used to clarify the dominant fracture mechanism of the studied materials.

3. RESULTS AND DISCUSSION

Figure (1) shows value of K_{IC} for pure PP and variations of fracture toughness of PP-EPR-CaCO3 hybrid composite versus $CaCO_3$/EPR. Also for comparison, the variations of fracture toughness of hybrid composites, which obey the rule of mixtures, has been shown by dash line. The results show that the value of K_{IC} for pure PP is about 1.9 MPa m ^{½}. This value is different from what proposed by other investigators [2-3]. This is because; fracture toughness can be affected by molecular weight and crystallinity structure. For example Hoffmann *et al*. [2] observed a raise in fracture toughness as molecular weight increase. Friedrich [3] showed that fracture toughness depends on spherulite size. While spherulite diameter increases a decrease in fracture toughness can be observed, though this effect will be negligible as molecular weight increased [2-3]. As it can be seen, the fracture toughness of PP/20$CaCO_3$ is lower than that of pure PP. In the other word addition of calcium carbonate led PP toward brittle behaviour, because of its big size. It is worth noting that the debonding energy is decreased with increasing the particle size [10].

Looking at in more details in Figure (1) shows that the trend of the fracture toughness values is nearly similar to what expected by the rule of mixtures, therefore one may conclude that the existence of both $CaCO_3$ and EPR have not significance effect on synergistic toughening.

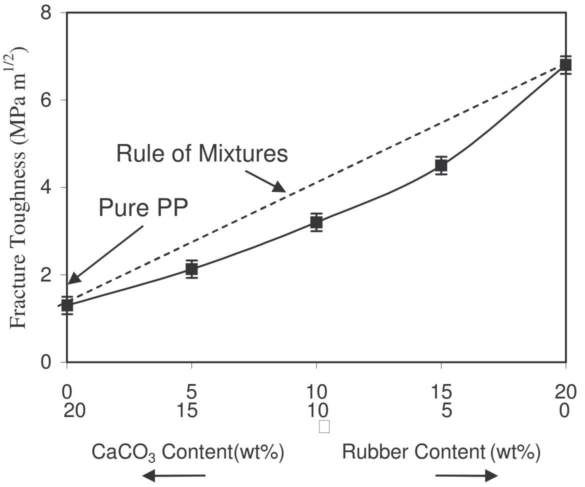

Figure 1. Variations of KIC of PP hybrid composite versus CaCO3/EPR ratio

In order to find the mechanism of fracture, it is important to consider the results of 4PB tests. Figures (2-a) and (2-b) show optical micrographs, taken from polished surfaces of PP-10$CaCO_3$-10EPR hybrid composite after 4PB testing at different magnifications. As expected, there is a typical damage pattern around the crack tip. Observation of the damage zone in front of the crack tip under cross-polarised light, illustrates that there is evidence of birefringence in matrix (not shown). This shows that for this material, in front of pre-crack, shear yielding is present inside the damage zone. Also some elongated voids are observed, those are probably formed due to cavitation of rubber particles or debonding at the interface [10-11].

For further elucidation of the mechanism of fracture SEM studies have been carried out on 4PB samples. Figures (3-a) and (3-b) show the SEM micrographs of the damage zone near a crack tip in PP-10GF-10EPR hybrid composite at different magnifications. As it can be seen, the damage zone inside of matrix is composed of crazes and some voids due to debonding. Briefly the dominant mechanism of fracture in PP-$CaCO_3$-EPR studied in this work can be a craze-like damage which is included with both highly localised dilatational shear bands due to EPR particles and some craze owning to $CaCO_3$.

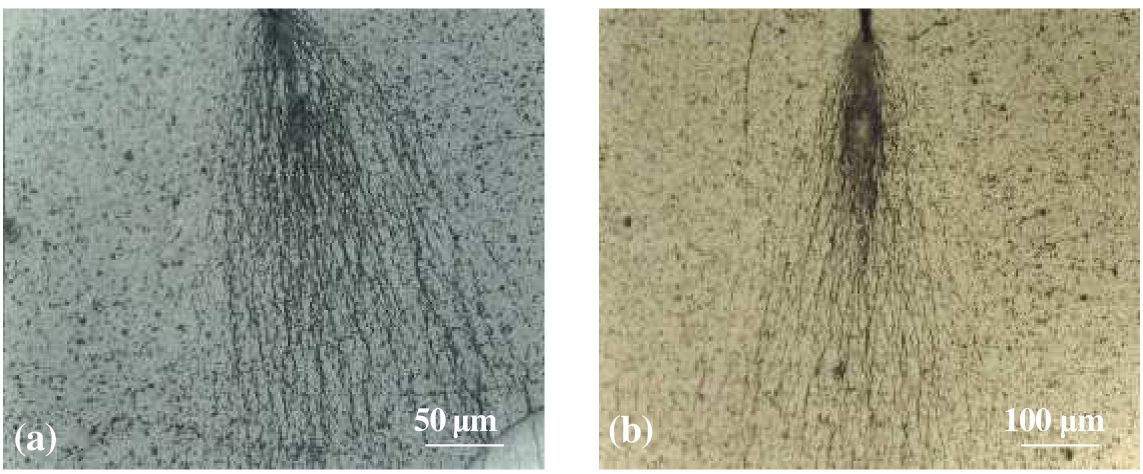

Figure 2. Optical micrographs of 4-point bending test at different magnifications

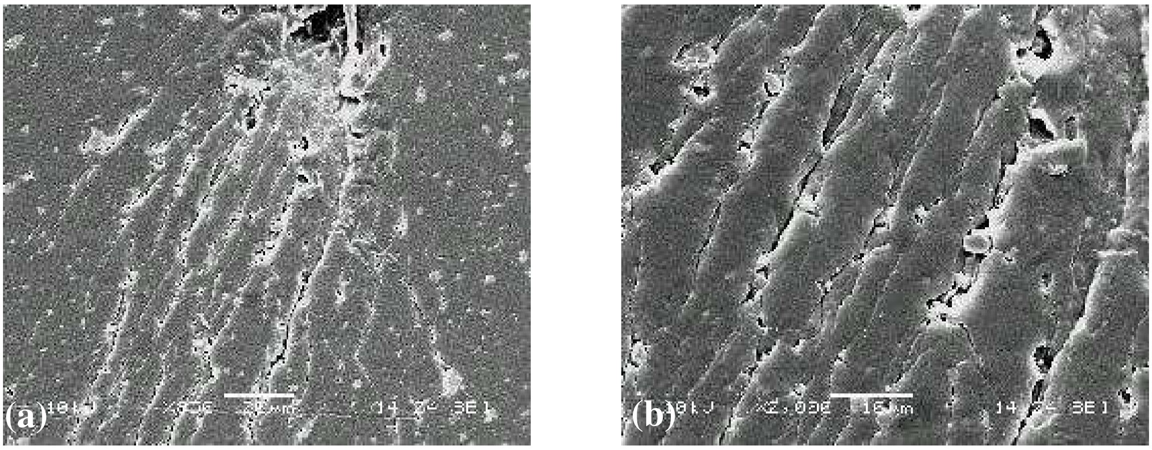

Figure 3. SEM micrographs of 4-point bending test at different magnifications

4. CONCLUSION

To understand fracture mechanism of PP-$CaCO_3$-EPR hybrid composites, three-point and four-point tests, optical and scanning electron microscopy were carried. The results are remark as bellow.

1) The existence of both $CaCO_3$ and EPR has not significance effect on synergistic toughening of PP-$CaCO_3$-EPR hybrid composite studied in this work.

2) The results of 4-point bending test illustrate that the damage zone which is formed in front of pre-crack included craze and there is evidence of shear yielding.

3) The dominant mechanism of fracture in PP-EPR- $CaCO_3$ reported to be a craze-like type of damage, which is believed to be combination of craze because of being $CaCO_3$ and highly localised dilatational shear banding owing to EPR.

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