

# Influence of PP-g-MA on Morphology, Mechanical Properties and Deformation Mechanism of Copolypropylene/Clay Nanocomposite

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**ABSTRACT:** Copolypropylene/organoclay nanocomposites are prepared by melt intercalation method in this research. Two different routes for addition of compatibilizer are examined, i.e. addition in the twin-screw extruder along with the polymer and the clay powder simultaneously and premixing the compatibilizer with the reinforcement in a batch mixer before addition to the polypropylene (PP) matrix. Morphology, tensile and impact properties and deformation mechanisms of the samples made via two procedures are studied and compared with those of the noncompatibilized system. To study the structure of nanocomposites, x-ray diffraction and transmission electron microscopy techniques are utilized. The deformation mechanisms of different samples are examined via reflected and transmitted optical micros-

copy. The results reveal that introduction of compatibilizer and also the procedure in which the compatibilizer is added to the compound, affect structure and mechanical properties of nanocomposite. The elastic modulus of PP-clay nanocomposite has increased 11.5% with incorporation of compatibilizer. Also, introduction of organoclay without compatibilizer facilitates crazing at the notch tip of PP in 3PB testing. Incorporation of compatibilizer, however, makes difficulties in initiation and growth of crazes at the notch tip. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 3751–3759, 2009

**Key words:** nanocomposites; poly(propylene) (PP); organoclay; mechanical properties; damage zone

## INTRODUCTION

Inorganic fillers are widely used in plastics industry to improve some properties such as stiffness, hardness, heat deflection temperature and shrinkage along with cost reduction. The effects of filler particles on composite properties, mainly depend on their shape, size, surface characterization and extent of dispersion. In general, mechanical properties of polymers reinforced with micro-particles are less than those of nanocomposites.<sup>1</sup> In addition, reaching to desired physical properties such as surface roughness and permeability is difficult when microparticles are used.<sup>1</sup> This is why many researches have been focused on developing nanocomposites in recent years.<sup>2–4</sup> Among different nano-fillers used, layered silicates such as montmorillonite have been considered significantly in recent years.<sup>5–12</sup> Polymers reinforced with montmorillonite reveal improvement

of both mechanical and physical properties including elastic modulus, strength, heat distortion temperature, and permeability.<sup>5</sup>

Use of layered silicates in polymeric matrices was started early in 90s by introduction of PA/nanoclay.<sup>7</sup> Since then, many polymers have been examined in such composites. Among them, polypropylene (PP) has been considered significantly.<sup>12</sup> This is due to high growth rate of this polymer, especially in auto industry.<sup>12</sup> It is well known that in such a non-polar resin, use of a kind of compatibilizer along with high shear stress is required to obtain a reasonable dispersion of clay platelets.<sup>13</sup> Please note that the nonpolar nature of PP may even suppress intercalation of clay in the absence of compatibilizer.<sup>14</sup> In such a case, nanocomposite will not form and improved properties are not obtained. Many efforts have been conducted to improve dispersion of clay in PP using functional oligomers as compatibilizer.<sup>14–22</sup> For this purpose, it is necessary to graft a polar compound such as maleic anhydride (MA) to the main chain of the non-polar polymer. This creates a functional polymer which is miscible in the matrix, while it is compatible with clay as well. The effectiveness of PP-g-MA as compatibilizer in PP/clay system depends on its molecular weight and

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MA content which determines its functionality.<sup>20</sup> The effect of MA content on dispersion of clay in the matrix has been investigated notably, but the results are to some extent contradictory.<sup>14–16,18,20–22</sup> The high amount of MA usually enhances the diffusion of PP chains between the layers, although, it may lead to immiscibility of compatibilizer in the matrix and thus, results in reduction of mechanical properties of composite.<sup>14</sup> Reichert et al.,<sup>18</sup> however, found that at a given clay loading, high MA content, e.g., 4.2 wt %, enhanced mechanical strength higher than that of the low MA content, e.g., 2.9 wt %. Nevertheless, the influence of molecular weight of the compatibilizer is less obvious.<sup>20</sup> Most researchers used low molecular weight PP oligomers to improve the diffusion of chains into clay galleries.<sup>14–16,18</sup> On the other hand, Wang et al.<sup>21</sup> illustrated that although low molecular weight ( $M_w = 9100$ ) oligomer resulted in better dispersion, high molecular weight ( $M_w = 330,000$ ) compatibilizer gave rise to better enhancement in mechanical properties. Kim et al.<sup>22</sup> incorporated four PP compatibilizers with different degrees of MA grafting and three PP matrices with low, medium and high viscosity. They found that the system containing the low viscosity PP and the least MA content compatibilizer resulted in the greatest enhancement of dynamic storage modulus.<sup>22</sup> Therefore, it is claimed that without mentioning the molecular weight of compatibilizer, judging the importance of the viscosity of individual components is hard.<sup>20</sup>

Deformation mechanism of polymer-clay nanocomposites has not been widely investigated. Specially, the kind of deformation mechanism and its role on mechanical behavior of PP-clay nanocomposites have not been studied until recently. In the current investigation, PP-g-MA has been made using the same copolymer used as the matrix. Influence of such a compatibilizer and method of its addition to the matrix on dispersion of nanoclay in PP and mechanical properties and deformation mechanism of the compounds have been investigated.

## EXPERIMENTAL METHOD

### Materials

A kind of co-PP with the MFI (at 230°C, 2.16 kg) of 7 g/10 min and the melting point of 175°C by the trade name of EP-C40R from Arak Petrochemical and an organophilic montmorillonite with the CEC of 110–120 meq/100 g, crystal system of monoclinic, the unit cell molecular weight of 540.46 g/mol and density of 2.5 g/mol with the modifier of Alkylammonium by the trade name of NANOLIN DK4 from FCC were used. To make compatibilizer, MA and

dicumyl peroxide (DCP), as initiator, both from Merck were incorporated in this investigation.

### Preparation of nanocomposites

Nanocomposites with and without compatibilizer were made in this study (Table I). As seen in the table, three series (S1–S3) specimens were made in this study. S1 contains no compatibilizer. In S2 and S3, PP-g-MA has been used as compatibilizer. The difference between these two series is that in S2 all components have been mixed directly in the extruder, while S3 has been made in two steps. In the first step, compatibilizer has been mixed with organoclay in an internal mixer. Mixing with PP has been done in the second step using a twin-screw extruder.

To make nanocomposite without compatibilizer (S1), mixtures of 1.5, 3, and 5 wt % nanoclay with PP were blended in a corotating twin-screw extruder ( $L/D = 20$ ) at a rotor speed of 500 rpm while the temperatures in the successive zones were set at 140, 170, 180, and 190°C.

For preparing the compatibilizer, MA (2 phr) and DCP (0.1 phr) were dissolved in acetone and physically mixed with PP pellets at room temperature. After evaporation of acetone in the air, MA and DCP stick on the granules homogeneously. As mentioned earlier, PP used for making compatibilizer was the same polymer used as the matrix.

S2 samples were blended in the extruder with the same conditions as S1 samples. To pre-mix PP-g-MA and clay for S3 samples, an internal mixer, Brabender PL2000, was used. The mixing condition of speed = 60 rpm,  $T = 175^\circ\text{C}$  and  $t = 4$  min was employed.<sup>23</sup> Final blending of the third series of specimens was done the same as other two series. An injection molding apparatus (the temperatures in the successive zones were set at 140, 170, 180, and 190°C) was incorporated to make tensile and Charpy impact specimens.

### Nanocomposite characterization

Wide-angle X-ray diffraction (XRD) analyzes were conducted using a Bruker Axs generator. An acceleration voltage of 40 kV and a current of 30 mA were applied using Cu-K $\alpha$  radiation. Transmission electron micrographs were taken using a Philips CM200 FEG with accelerating voltage of 200 kV. Tensile tests were performed using a universal testing frame (Hounsfield H10KS) equipped with an extensometer, according to ASTM D638. Notched impact strengths were measured according to ASTM D256 using a 5 J impact hammer from Santam. The dimensions of Charpy samples were 25 × 12.5 × 3.5 mm<sup>3</sup>. All mechanical tests were performed on average number

**TABLE I**  
**Nanocomposites Made in this Study**

System	Description
PP + 1.5 wt % clay	S1- without compatibilizer
PP + 3 wt % clay	
PP + 5 wt % clay	
PP + 1.5 wt % PP-g-MA + 1.5 wt % clay	S2- mixed in the extruder simultaneously
PP + 3 wt % PP-g-MA + 3 wt % clay	
PP + 5 wt % PP-g-MA + 5 wt % clay	
PP + [1.5 wt % PP-g-MA + 1.5 wt % clay]	S3- PP-g-MA and clay were blended in an internal mixer before mixing with PP in the extruder
PP + [3 wt % PP-g-MA + 3 wt % clay]	
PP + [5 wt % PP-g-MA + 5 wt % clay]	

of five specimens. The deformation mechanisms of the materials subjected to tensile loading were studied by a reflected light optical microscope (Olympus PME3) under cross-polarized light. To perform this investigation, the side surfaces of some tensile samples were polished before loading. During loading, the deformation patterns appear on the polished surface and can be observed via reflected optical microscope. Study of deformation behavior at the notch tip was done on thin samples cut from three-point-bent (3PB) specimens using a transmitted light optical microscope (OLYMPUS DL12). To do this experiment, notched Charpy samples were subjected to a certain amount of loading in 3PB mode, in which the plastic deformation was happened at the notch tip of samples. For observation of the deformation mechanism, the samples were cut and polished from both sides to reach the thickness of approximately < 100  $\mu\text{m}$  in the middle of the samples.

## RESULTS AND DISCUSSION

### Structure characterization

The results of XRD for organoclay and PP/clay nanocomposites (5 wt % clay and 5 wt % PP-g-MA in S1, S2, and S3) are shown in Figure 1 and Table II.

As seen in Table II, introduction of nanoclay into PP (S1-S3) results in a modest increase in d spacing of (001) and (002) planes. Therefore, based on Table II, one may conclude that the compatibilizer used and method of its addition has had no influence on d spacing of the clay platelets. Figure 1, however, shows a significant difference in intensity of peaks in different nanocomposites. Reduced peak intensity at (001) and (002) planes of nanocomposites can be attributed to the reduced number of layers in clay stacks.<sup>5</sup> Reduction in number of layers by

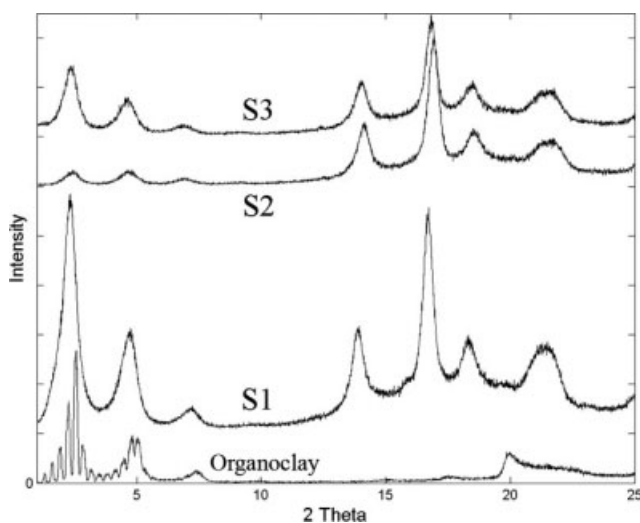
introduction of compatibilizer has been reported before.<sup>24</sup> Using a functional oligomer as compatibilizer can increase the amount of adhesion between layers and polymer.<sup>25</sup> One may conclude that due to increasing the adhesion of layered silicates to matrix, more layers can take off from stacks during application of high shear stress (the rotor speed of extruder was  $\sim 500$  rpm) to system. Subsequently, the numbers of remained layers in each stack decrease and thus, extent of exfoliation increases (i.e. the number of layers in stacks decreases). As stated before, height reduction of peaks related to (001) layers in XRD graphs in the presence of compatibilizer may point out to this matter (Fig. 1). As observed in Figure 1, the S2 specimen, which all three parts have been extruded simultaneously, has the shortest (001) peak and therefore, the extent of exfoliation in this sample may be higher. TEM was utilized to further study the morphology of nanocomposites. Figure 2 represents a typical clay stack in PP/clay (s) nanocomposite where no compatibilizer was incorporated (S1 specimen).

As seen in this figure, an average of more than 10 clay platelets exists in the stack. Figure 3 illustrates TEM micrographs taken from S2 specimen (5 wt %) sample where PP, clay and compatibilizer were mixed simultaneously in extruder. As seen in Figure 3, the typical number of layers per stack is about 3. This observation confirms that despite the compatibilizer used did not influence the distance between the clay layers, it was successful in breaking apart the clay stacks which resulted in reduced number of platelets per stack. Such morphology might be treated as exfoliated structure where instead of single clay layers, stacks with a few layers are dispersed throughout the matrix.

### Mechanical properties

The results of mechanical properties measurements are illustrated in Figure 4.

As seen in Figure 4(a), the elastic modulus increases with increasing the organoclay in all three systems. As stated before, the d-spacing between layers is the same in three systems and thus, the difference between the elastic modulus seen in Figure 4(a) is not due to the different d-spacing. Table II and Figure 4(a) illustrate that addition of compatibilizer improves elastic modulus. This observation can be attributed to better dispersion of clay layers (exfoliated structure) when PP-g-MA is utilized. Apparently, simultaneous addition of clay and compatibilizer (Series 2) is more effective in elastic modulus improvement [Fig. 4(a)] because of better dispersion of clay layers in S2 in comparison with S3 [the peak intensity at 001 planes in S2 is lower than in S3 (Fig. 1)].

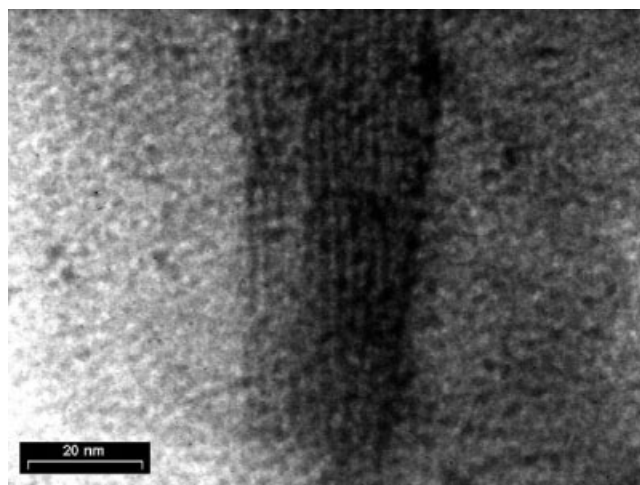


**Figure 1** XRD graphs of organoclay and polypropylene based nanocomposites.

Figure 4(b) presents continuous reduction of yield stress by incorporation of MMT in the absence of compatibilizer. This observation can be explained by the help of what showed in an earlier work of the same group.<sup>26</sup> According to that study, in S1 specimens, due to low adhesion of layers to matrix and relatively high amounts of layers in each stack, layers subjected to applied stress can easily slip on each other and subsequently, molecular chains of polymer can move easier. In other words, organoclay stacks without compatibilizer may act as initiation sites for activating deformation mechanism and as a result, yield stress decreases with increasing the clay content. This phenomenon will be further explained in deformation mechanism section. In the presence of compatibilizer, there are several separated layers of organoclay [based on the XRD graphs and TEM micrographs (Figures 1 and 3)] with good adhesion to matrix. These layers can resist against movement of polymer chains and therefore, the necessary stress for plastic deformation will increase. Increasing the yield stress in S2 series is more pronounced than that of S3 series due to fewer numbers of clay layers in stacks of this sample (Fig. 1). In these systems, despite being the same ratio of

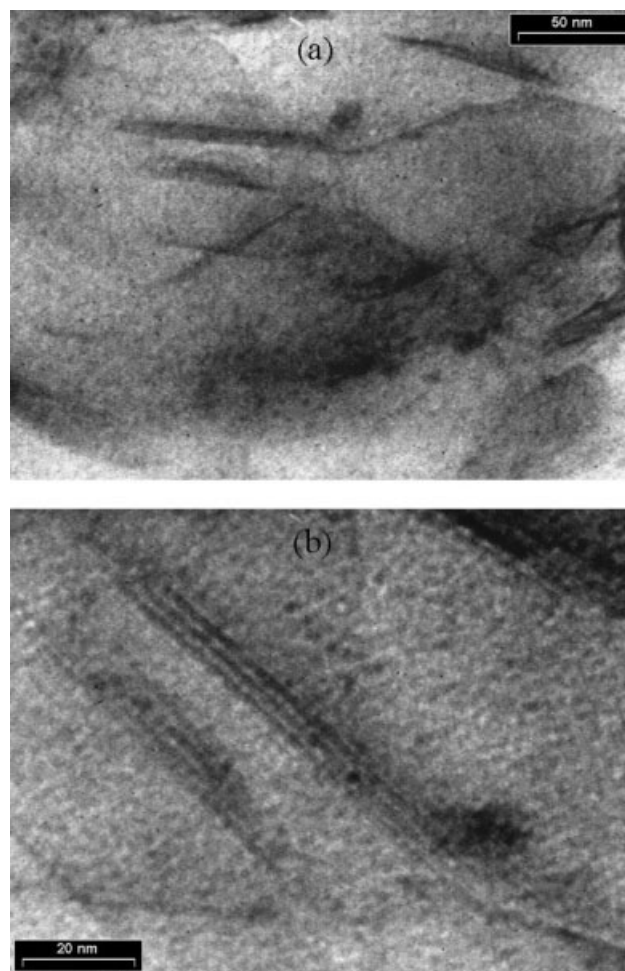
**TABLE II**  
XRD Results of Organoclay and Samples Made

	2 $\theta$		d-Spacing ( $\text{\AA}$ )	
	(001)	(002)	(001)	(002)
Organoclay	2.578	5.035	34.23	17.53
S1 (PP + Clay)	2.363	4.747	37.35	18.59
S2 (PP + PP-g-MA + Clay)	2.408	4.613	36.65	19.13
S3 (PP + [PP-g-MA + Clay])	2.453	4.622	35.98	19.09

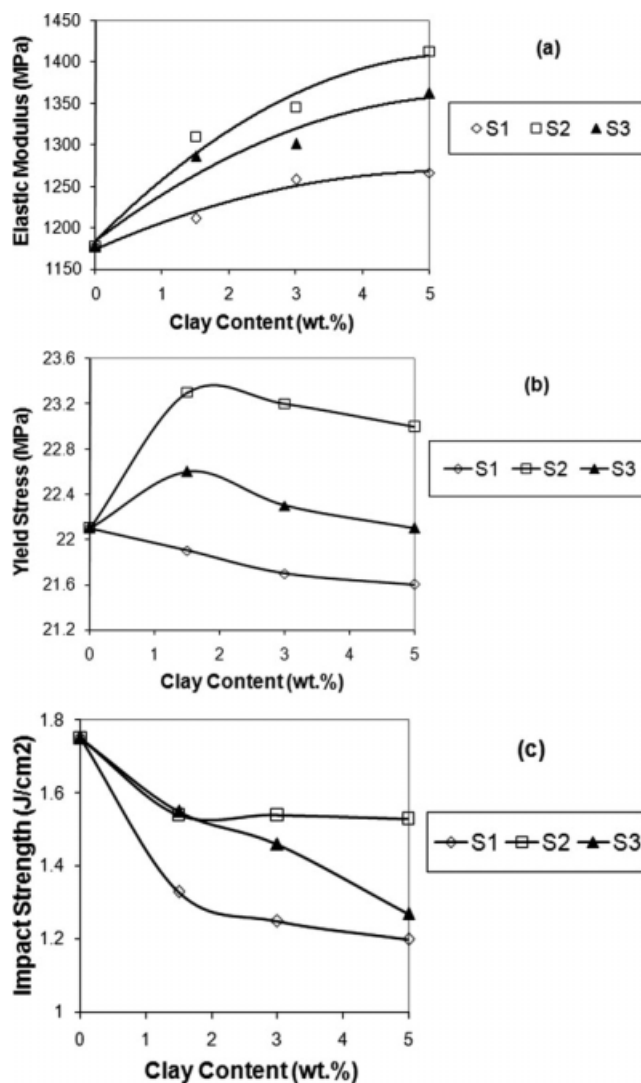


**Figure 2** The TEM micrograph of PP + 5 wt % clay, without compatibilizer (S1).

compatibilizer to organoclay in all samples, increasing the amount of organoclay may increase the amount of layers in each stack and consequently, a slight reduction in yield stress can be reasonable.



**Figure 3** The TEM micrographs of S2 specimen (5 wt %) at two different magnifications.



**Figure 4** Variation of (a) elastic modulus, (b) yield stress, and (c) impact strength versus weight percent of clay.

Decreasing the impact strength of PP with addition of organoclay (with/without compatibilizer) is shown in Figure 4(c). As seen in this figure, existence of compatibilizer decreases the reduction of the impact strength of samples. It means that increasing the adhesion between layers and matrix and decreasing the amount of layers in stacks can improve this property of nanocomposites. However, for understanding the relationship between mechanical properties such as yield stress and impact strength and morphology of samples, it is necessary to investigate deformation mechanism during loading. The following section has been devoted to this subject.

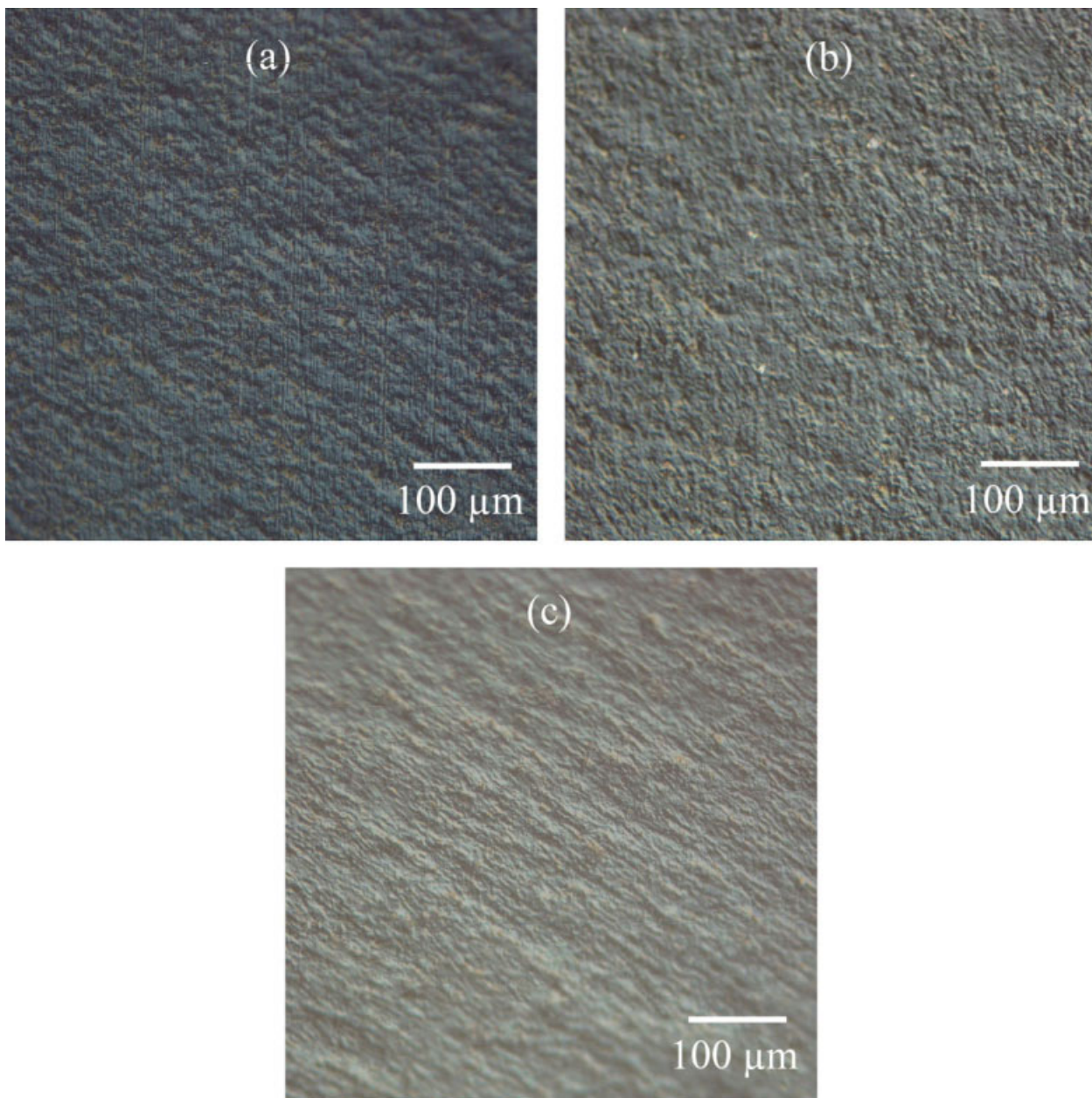
### Deformation mechanism

To investigate the influence of organoclay and compatibilizer on deformation behavior of PP, three

tensile specimens including neat PP, PP + 5 wt % clay and PP + 5 wt % PP-g-MA + 5 wt % clay were polished and subjected to tensile loading until the yielding point. As mentioned in experimental procedure, the pre-polished surfaces were then examined via reflected optical microscopy. The results are shown in Figure 5.

The PP used in this research is a kind of hetero-phasic copolymer and consists of almost 8 wt % of polyethylene. The morphology of this kind of co-PP consists of PP as matrix and the rubbery phase of EPR which is usually dispersed in the matrix with uniform and small size morphology.<sup>27</sup> These rubber particles act as stress concentrators and thus, very fine shear bands form on the surface. As seen in Figure 5(a), shear bands are so fine that almost cannot be seen in optical microscopy. If cross-polarized and DIC filters are not used for investigating the deformation mechanism of sample, the signs of deformation mechanism will be scarcely visible on the surface. Introduction of organoclay with/without compatibilizer have different influences on deformation mechanism of matrix as described in the following. As stated before, the structure of nanocomposite containing organoclay without compatibilizer consists of a relatively high amount of layers in each stack which their adhesions to the matrix are unsuitable. On the other hand, it is well known that the dominant deformation mechanism in PP under static loading condition (at low strain rates) is shear yielding.<sup>28,29</sup> In other words, the polymer molecules tend to align in the direction of 45° to direction of static loading. It seems that the existence of silicate layers (with the condition which stated before) can facilitate slippage of polymer chains under loading. Therefore, it is expectable that the deformation mechanism of this sample is as fine as the neat sample [Fig. 5(b)]. When compatibilizer is used, due to strong adhesion of layers to the matrix and high amount of separated layers in polymer, the movement of molecular chains subjected to applied stress will be more difficult and thus, the system will need additional stress for occurring deformation in comparison to the pure PP. Difficulties in shear deformation by introduction of nanoclay may cause transformation of shear yielding from fine to coarse [compare Fig. 5(a-c)]. In another word, if something prevents the movement of polymeric chains, the suitable sites for initiation of deformation will decrease and thus, coarser bands form. The surface roughness caused by shear yielding mechanism, can be observed more clearly in Figure 5(c). Increasing the yield stress of the samples with compatibilizer [Fig. 4(b)] may corroborant the slippage difficulty of polymer chains in this system.

To investigate the effect of organoclay on crazing behavior in PP, it is necessary to apply the load at

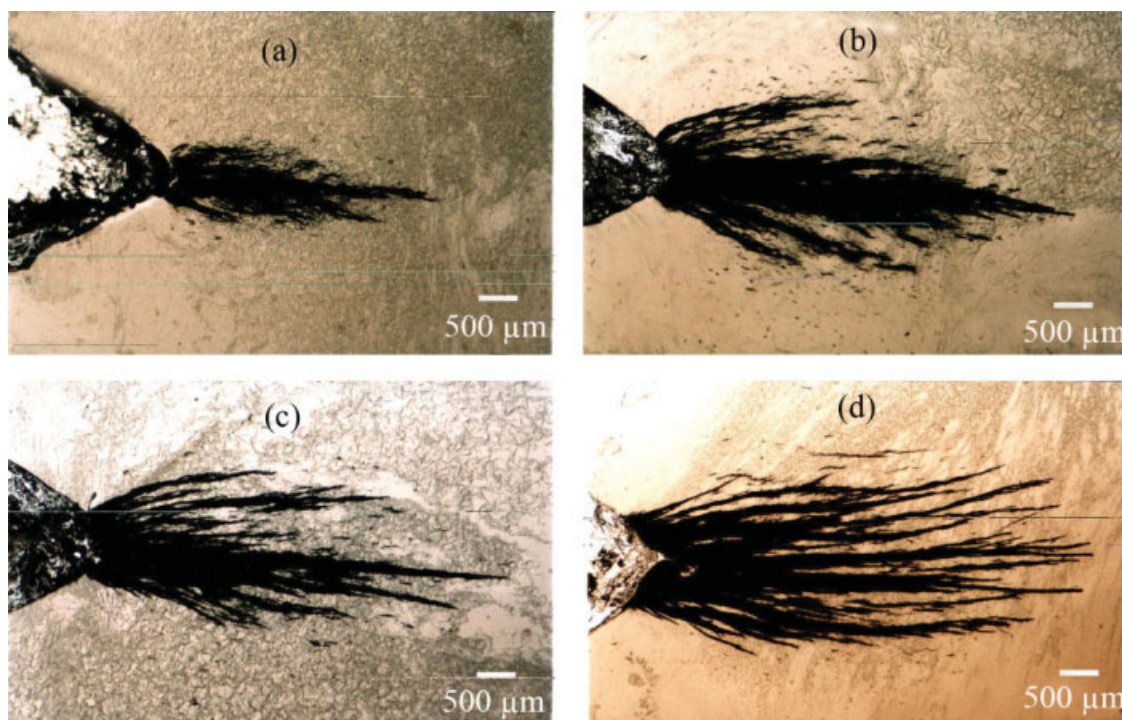


**Figure 5** Shear yielding mechanisms of samples: (a) neat PP, (b) S1 (5 wt %), and (c) S2 (5 wt %) subjected to vertical loadings, observed under cross-polarized, and DIC filters. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

higher rates. This is due to the fact that PP undergoes crazing at higher strain rates.<sup>30</sup> This situation has been provided by loading of single-edge-notched specimens loaded under three-point bending (3PB) condition. The deformation mechanisms at the notch tip of samples subjected to 3PB loading are illustrated in Figures 6–8.

The dominant deformation mechanism of PP at the notch tip in flexural loading is crazing due to high strain rate.<sup>30</sup> This is consistent with the observations made in this study (Figs. 6–8). Comparing Figures 6–8 reveals that the larger damage zones form when compatibilizer is not incorporated (Fig. 6). This means

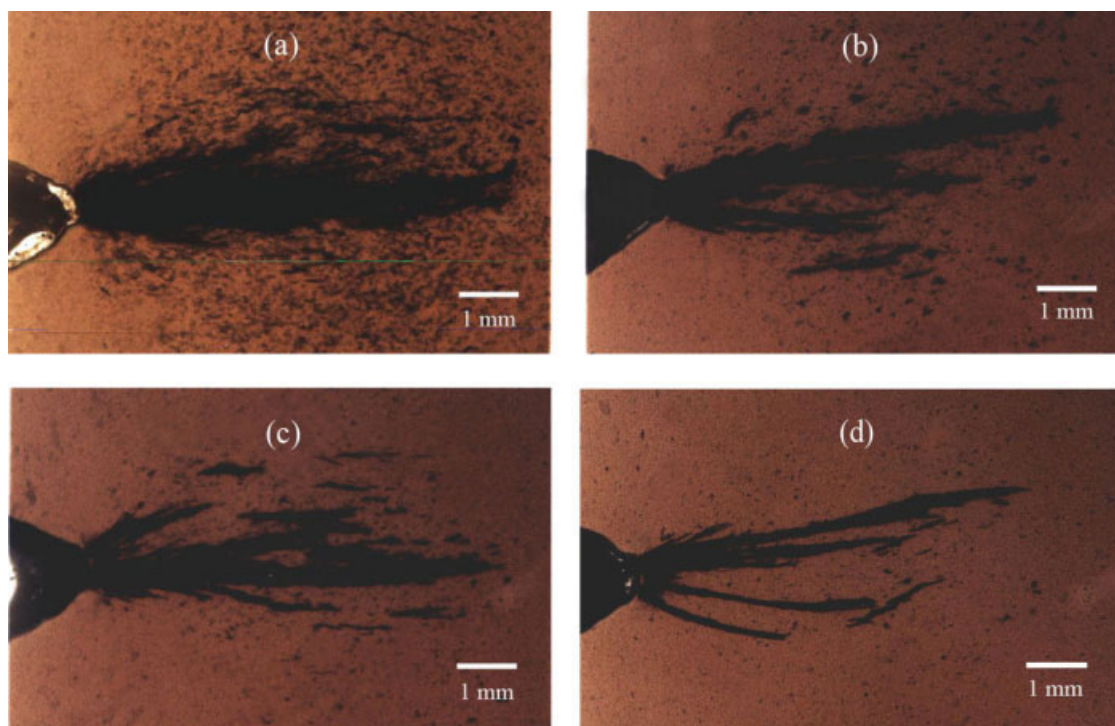
that incorporating organoclay without compatibilizer facilitates crazing at the notch tip. As seen in Figure 6, increase in clay loading enlarges the damage zone size, since the number of craze initiation sites increases with clay content. This phenomenon is not observed in the samples containing compatibilizer. Figures 7 and 8 illustrate that the deformation zone at the notch tip becomes smaller and localized with increasing the amount of organoclay and compatibilizer in both S2 and S3 series. This shows that the introduction of organoclay with compatibilizer prevents the initiation and growth of crazes. As stated before, silicate layers with almost good adhesion to



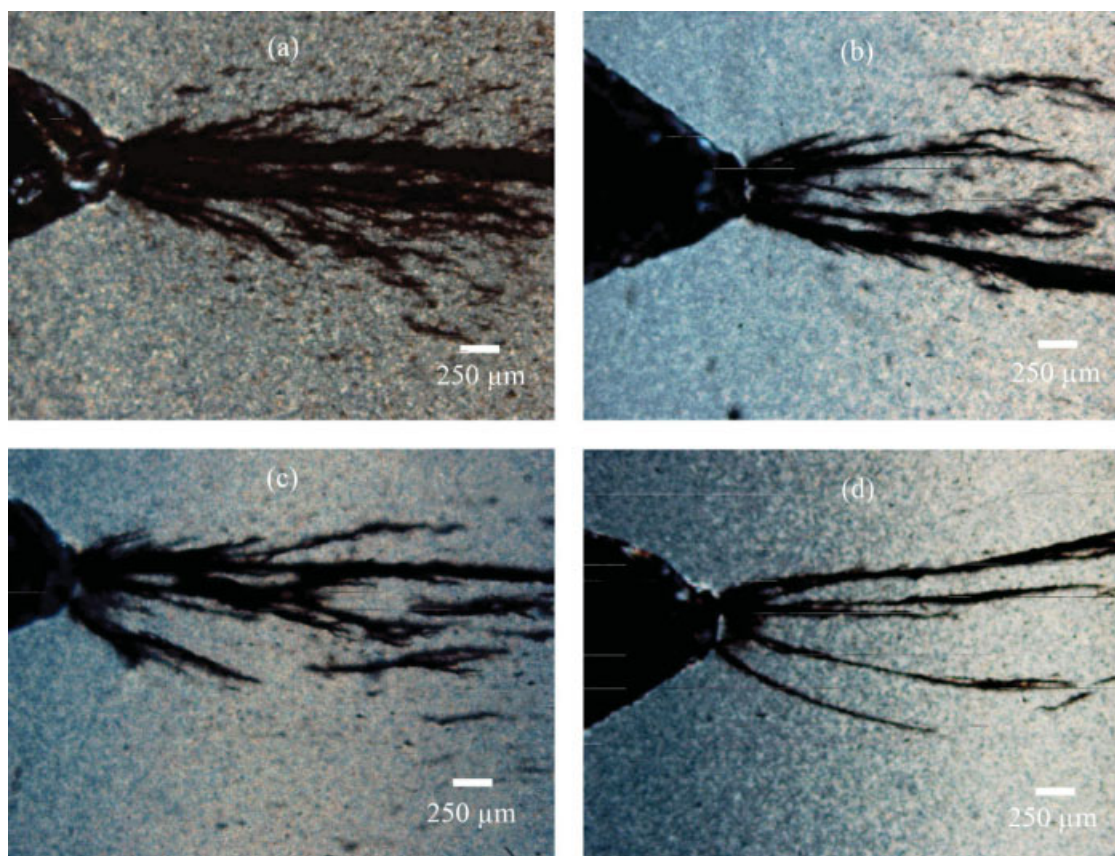
**Figure 6** Transmission optical micrographs showing damage zones at the notch tip of S1 specimens subjected to the load of  $F = 215$  N: (a) neat PP, (b) 1.5 wt %, (c) 3 wt %, and (d) 5 wt %. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

the matrix and relatively high aspect ratio (relatively low amount of layers in each stack), can act as obstacles for slippage of polymer chains. Therefore,

one may conclude that introduction of organoclay with compatibilizer can increase the yield stress of PP in the condition of occurring the shear yielding



**Figure 7** Transmission optical micrographs showing damage zone at the notch tip of S2 specimens subjected to the load of  $F = 170$  N: (a) neat PP, (b) 1.5 wt %, (c) 3 wt %, and (d) 5 wt %. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 8** Transmission optical micrographs showing damage zone at the notch tip of S3 specimens subjected to the load of  $F = 170$  N: (a) neat PP, (b) 1.5 wt %, (c) 3 wt %, and (d) 5 wt %. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

(tensile test) and also can limit the deformation zone in the condition of happening the crazing mechanism (3PB). In another word, both deformation mechanisms, shear yielding and crazing, need moving of polymer chains for occurring. Therefore, everything that prevents moving of polymer chains (here organoclays with compatibilizer) can postpone or limit deformation mechanism of the sample. This limitation reveals in increasing the yield stress in static tensile loading and decreasing the damage zone in 3PB loading.

### CONCLUSIONS

Co-PP/clay nanocomposites were prepared via melt intercalation in an intermeshing corotating twin-extruder and the following results were obtained:

- The existence of compatibilizer and its mixing method does not alter the d-spacing between the layers, but affects the adhesion of the layers to the matrix and also the number of layers in stacks.
- Incorporation of compatibilizer increases modulus of elasticity, yield stress and impact strength of nanocomposites.

- The sample, which all three parts (PP, organoclay and PP-g-MA) were mixed in extruder simultaneously (S2 series), shows the best mechanical properties. In fact, premixing of organoclay and compatibilizer does not provide better dispersion of nanoclay in the matrix.
- The shear yielding occurs in tensile testing of pure polymer and its nanocomposites. The surface roughness (The roughness of the polished surface after deformation) increases in nanocomposite specimens when compatibilizer is incorporated.
- Introduction of organoclay without compatibilizer facilitates crazing at the notch tip of PP in 3PB testing. Incorporation of compatibilizer, however, makes difficulties in initiation and growth of crazes at the notch tip. This is due to the improvement of adhesion at interface of clay platelets and the matrix that results in further increase of yield stress.

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