



A new approach to increase weld line strength of incompatible polymer blend composites: selective filler addition

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Abstract

A ternary blend composite composed of two immiscible organic phases, polypropylene (PP) and polyamide-6 (PA), and talc as inorganic filler is studied in terms of weld line properties and morphology. Effects of different filler levels as well as compatibilizer on weld line strength under tensile loading condition of each polymeric phase are investigated. Special attention is paid to relate the nature of dispersed domains especially in weld line regions with final performance. It is observed from scanning electron microscopy (SEM) studies that addition of talc filler which is selectively wetted by dispersed PA phase dramatically reduces the elongated domain size in the weld line region and causes to much more homogeneous microstructure. The selective wetting of talc particles by PA phase, therefore, seems to be beneficial in increasing weld line strength via increasing the viscosity of the PA dispersed phase and consequently decreasing the elongated domains at weld line region. Compared to uncompatibilized blend composites, compatibilized ones represent much higher weld line strength for all levels of talc loading.

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1. Introduction

One of the problems of injection molding process which may be unavoidable in most of the multi gate mould designs is ‘weld line’ or ‘knit line’ which is formed when two or more separate melt fronts traveling in different direction meet. Weld line is undesirable when the strength, long term durability and surface quality are concern. It is observed due to multigate molding, existence of pins, inserts or cores within the mould cavity. Variable wall thickness and jetting may lead to weld line because of a sudden change in geometry along the flow path [1–7]. Weld lines can be categorized as cold and hot weld line [1,4]. In previous one, two melt fronts meet head to head and additional flow could not take place after combination. This type leads lowest strength. Hot weld lines (stream lines or melt lines) form when two or more melt streams recombine to each other

such as breaking up and rejoining of flow around pins. An additional flow after recombination occurs.

In unfilled polymers, the weakness of weld line can be explained by incomplete bonding due to inefficient molecular entanglement at the interface, disturbance of molecular orientation parallel to flow direction, inefficient diffusion time, existence of voids and V shape notch owing to entrapped air or contaminants and notch depth [4–6].

On the other hand, loss in weld line strength in filled and reinforced polymers is found to be related with aspect ratio of the dispersed phase [6,8,9]. A serious reduction in strength is observed for polymers reinforced with fibers (glass) or platelets (talc) having high aspect ratio. However, spherical (glass beads or calcium carbonate) additives lead almost identical strength for both specimens with and without weld line.

A limited number of works [10–12] has been done to investigate the effect of weld line on mechanical properties and morphology of immiscible polymer blends. Kim et al. [10] showed that the injection-molded specimens without weld line exhibited traditional skin-core morphology in

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Table 1
Blend and composite compositions

Sample name	PP (wt%)	PA-6 (wt%)	MA-g-PP (wt%)	Talc (wt%)
PP	100.0	–	–	–
PP1	90.0	–	–	10.0
PP2	80.0	–	–	20.0
PP3	70.0	–	–	30.0
PA	–	100.0	–	–
PA1	–	90.0	–	10.0
PA2	–	80.0	–	20.0
PA3	–	70.0	–	30.0
PPPA	75.0	25.0	–	–
PPPA1	67.5	22.5	–	10.0
PPPA2	60.0	20.0	–	20.0
PPPA3	52.5	17.5	–	30.0
PPPAC	60.0	25.0	15.0	–
PPPAC1	54	22.5	13.5	10.0
PPPAC2	48.0	20.0	12.0	20.0
PPPAC3	42.0	17.5	10.5	30.0

which dispersed domains at sub skin layers are elongated toward the flow direction and those at core are not deformed. Elongated domains were reduced by increasing the amount of compatibilizer. But in the presence of weld line and absence of compatibilizer, elongated domains were perpendicular to flow direction at weld line region. Increasing amount of compatibilizer led to fine and isotropic morphology. Similar observation was reported by Fellahi and Fisa [11]. They used a plaque mould with circular insert to create hot weld line or melt line. Reduced thickness of the skin layer, decreased width of weld line region and more isotropic weld line morphology for the blend with compatibilizer were observed. Jarus et al. [12] revealed that by changing the disperse phase viscosity with molecular weight, failure at weld line changes from brittle to ductile nature, since increase in viscosity ratio of blend leads to decrease in elongated disperse domains which is the reason of weakness in strength at weld line.

On the other hand, in a number of engineering applications, the thermoplastic materials of choice are multiphase systems containing polymer blends having a particulate reinforcement as well as weld lines. The weld

line strength in these kinds of injection molded and filled immiscible blend composites needs to be studied.

The aim of this study is to investigate effect of weld line on mechanical and morphological properties of multiphase composites that have two immiscible organic phase, polypropylene (PP) and polyamide-6 (PA), in the presence of inorganic filler; talc. Emphasis is placed on morphology development as a function of both the amount of dispersed phase selective talc addition and the compatibilizer in order to increase the weld line strength of resultant composites. The PP/PA system was chosen because of the extensive existing literature on similar blends, and the significant difference in the chemical character, and therefore affinity to filler, of the two components.

2. Experimental

2.1. Material

PA-6 (Specialamid) with melt index 22 gr/10 min and density 1.12 g/cc was obtained from Gruppo Bonazzi, Italy. PP (Moplen HP 502L) with melt index 8 gr/10 min and density 0.9 g/cc was from Basell, Italy. Maleic anhydride grafted polypropylene (MA-g-PP) containing 1% maleic anhydride (MA) was from Uniroyal Chemical, USA as Polybond 3200, with the density 0.91 g/cc, melting point 157 °C, and melt flow rate 120 g/min 10- μ m mean diameter talc was supplied from OMYA MADENCILIK-Turkey.

2.2. Preparation of polymer blends and composites

The detailed compositions of blends and composites are given in Table 1. Polypropylene and polyamide-6 are indicated as PP and PA, respectively. Immiscible polypropylene and polyamide-6 blend is marked as PPPA, while polypropylene and polyamide-6 blend compatibilized by MA-g-PP is signed as PPPAC. Talc filled composites are denoted as an integer number following the matrix designation.

All blends were prepared by twin-screw extrusion techniques as described in previous studies [13–15]. PA and MA-g-PP were dried at 80 °C overnight to eliminate the hydrolyzing effect of absorbed water. The filler talc and polymeric ingredient(s) were fed separately into the extruder from the same inlet port with the help of two Brabender DSR 28 volumetric feeders. For PPPA and PPPAC recipes, previously weighted PP, PA, and for some formulations MA-g-PP were dry mixed to prepared desired polymer matrixes described in Table 1. The extruder was an intermeshing co-rotating modular twin-screw extruder with L/D of 28:1. The screw rotation speed was set to 100 rpm. Temperature profile was set to 70 °C for first feed port and 235 °C for the rest. Kneading blocks of 30, 60 and 90° were located at 12 L/D, followed by back mixing element and another 30, 60 and 90° kneading blocks was located at

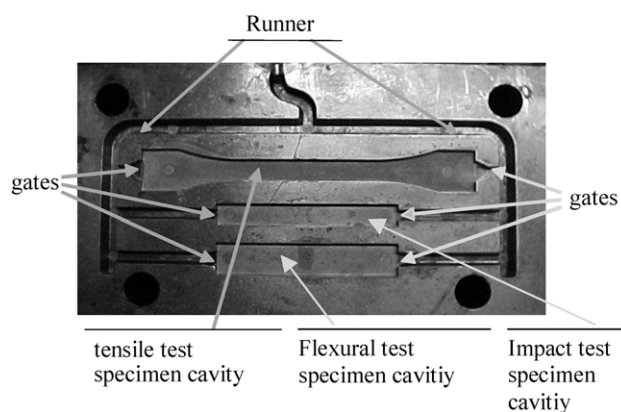


Fig. 1. Injection mould.

Table 2
Tensile properties of normal and weld line specimens

Sample name	Tensile yield stress (MPa)	Tensile yield strain (%)	Young's modulus (MPa)	Stress at break (MPa)	Strain at break (%)	Tensile yield stress (MPa)	Tensile yield strain (%)	Young's modulus (MPa)
PP	34.65 (0.42)	10.35 (0.70)	1686.50 (60.07)	17.79 (0.35)	702.80 (5.3)	27.48 (0.21)	11.20 (0.03)	1328.70 (27.32)
PP1	33.91 (0.09)	6.91 (0.11)	2391.91 (48.03)	20.01 (0.13)	60.20 (0.23)	27.71 (0.56)	3.37 (0.24)	2212.96 (57.36)
PP2	33.05 (0.15)	5.90 (0.07)	2827.21 (18.86)	22.34 (0.68)	13.12 (0.75)	25.57 (0.10)	2.08 (0.07)	2645.49 (46.90)
PP3	32.70 (0.07)	4.58 (0.12)	3283.66 (42.67)	28.27 (0.30)	8.68 (1.39)	22.82 (0.04)	1.30 (0.03)	2935.85 (32.00)
PA	71.17 (0.92)	3.51 (0.05)	3002.69 (37.66)	41.98 (0.18)	170.82 (0.87)	66.51 (0.67)	3.55 (0.05)	2734.34 (33.25)
PA1	75.07 (1.48)	3.40 (0.07)	4678.69 (112.07)	71.16 (3.64)	6.60 (2.59)	59.41 (0.80)	1.88 (0.05)	4664.10 (81.51)
PA2	75.49 (1.46)	3.01 (0.03)	6307.15 (165.95)	75.30 (1.33)	3.25 (0.22)	45.61 (2.39)	0.95 (0.08)	6634.57 (81.82)
PA3	77.32 (0.91)	2.21 (0.25)	8809.19 (153.34)	77.31 (0.67)	2.22 (0.02)	36.41 (0.67)	0.53 (0.02)	8808.43 (86.66)
PPPA	31.12 (0.27)	4.01 (0.04)	2136.80 (15.06)	30.01 (1.3)	12.96 (2.3)	6.84 (1.33)	0.48 (0.08)	1817.50 (218.99)
PPPA1	27.40 (0.44)	3.65 (0.04)	2619.66 (75.96)	26.78 (0.29)	4.49 (0.14)	7.28 (0.86)	0.46 (0.06)	2096.96 (237.54)
PPPA2	24.66 (0.22)	3.24 (0.05)	2741.44 (112.03)	23.98 (0.61)	4.03 (0.30)	9.13 (0.11)	0.43 (0.08)	2721.84 (340.01)
PPPA3	22.80 (0.13)	2.09 (0.11)	3535.05 (66.18)	22.27 (0.31)	2.61 (0.38)	11.61 (0.06)	0.37 (0.02)	3834.61 (185.57)
PPPAC	37.76 (0.23)	5.36 (0.18)	2326.40 (32.78)	14.29 (1.2)	37.5 (5.4)	26.03 (2.80)	1.94 (0.67)	2235.20 (26.72)
PPPAC1	37.26 (0.26)	4.35 (0.09)	2881.22 (44.40)	35.66 (0.27)	7.39 (0.47)	29.19 (0.34)	1.92 (0.01)	2782.79 (84.92)
PPPAC2	36.33 (0.41)	3.59 (0.03)	3554.09 (115.17)	36.08 (0.54)	4.13 (0.20)	25.77 (0.29)	1.22 (0.02)	3179.90 (87.52)
PPPAC3	37.46 (0.40)	2.93 (0.03)	4500.55 (115.09)	37.38 (0.43)	3.21 (0.13)	23.45 (0.32)	0.77 (0.09)	4703.12 (125.46)

20 L/D; just before venting port which was connected to a rotary Edward vacuum pump. The remaining elements were conveying elements. The extrudate was quenched in a water bath, pelletized and then dried.

The composites were dried at 80 °C at least 2 h and then injected into a special double-gated mould. The injection mould had three cavities as shown in Fig. 1. One of them was suitable for tensile test specimens. Other two cavities produced impact and flexural test specimens. Although the last two were not used in this study, it was not closed in order not to affect the runner balance of the mould. The mould had two runner systems feeding two gates for each cavity. Gates were located opposite ends of cavities to form cold weld line in the middle. One of the runners was closed to produce specimens without weld line. Specimens containing weld line were produced by letting both gates of each cavity open. The mould used in the experimental studies was shown in Fig. 1. The injection machine was lab scale Manumold 70/30 injection machine with a 30-ton clamp force, three heading zones set to 230 °C at the barrel and the mould set to 25 °C. Injection pressure and holding pressure were set to 44 and 30 bar, respectively. Injection speed was 125 cm³/s and cycle time was 45 s.

2.3. Characterization

Typical stress strain curves for normal and weld line specimens were obtained at ambient temperature using an Instron 4505 tensile testing machine. At least five specimens were tested with 50-mm/min crosshead speeds for tensile. Yield stress, yield strain, Young's Modulus, stress at break and strain at break were all recorded.

For the ease of comparing the effect of weld line on mechanical properties of molded material, the weld line factor F_{wl} was defined as [1,2];

$$F_{wl} = \frac{\text{Property value of specimen with weld line}}{\text{Property value of specimen without weld line}} \quad (1)$$

Melt flow index (MFI) was determined by using Zwick 4106. Test temperature and load were set to 230 °C and 2.16 kg, respectively.

A scanning electron microscope (Jeol JSM 35C) was used to examine the morphology of the blends. Ten-millimeter long sections containing weld line in the middle were cut from molded samples. A notched with the depth of near half of the specimen thickness was opened perpendicular to weld line. Notched samples were rested within liquid nitrogen for 2 h and broken with a sudden impact. The fractured surfaces were coated with gold.

3. Results and discussion

Some tensile properties of normal (without weld line) and weld line specimens as well as standard deviation

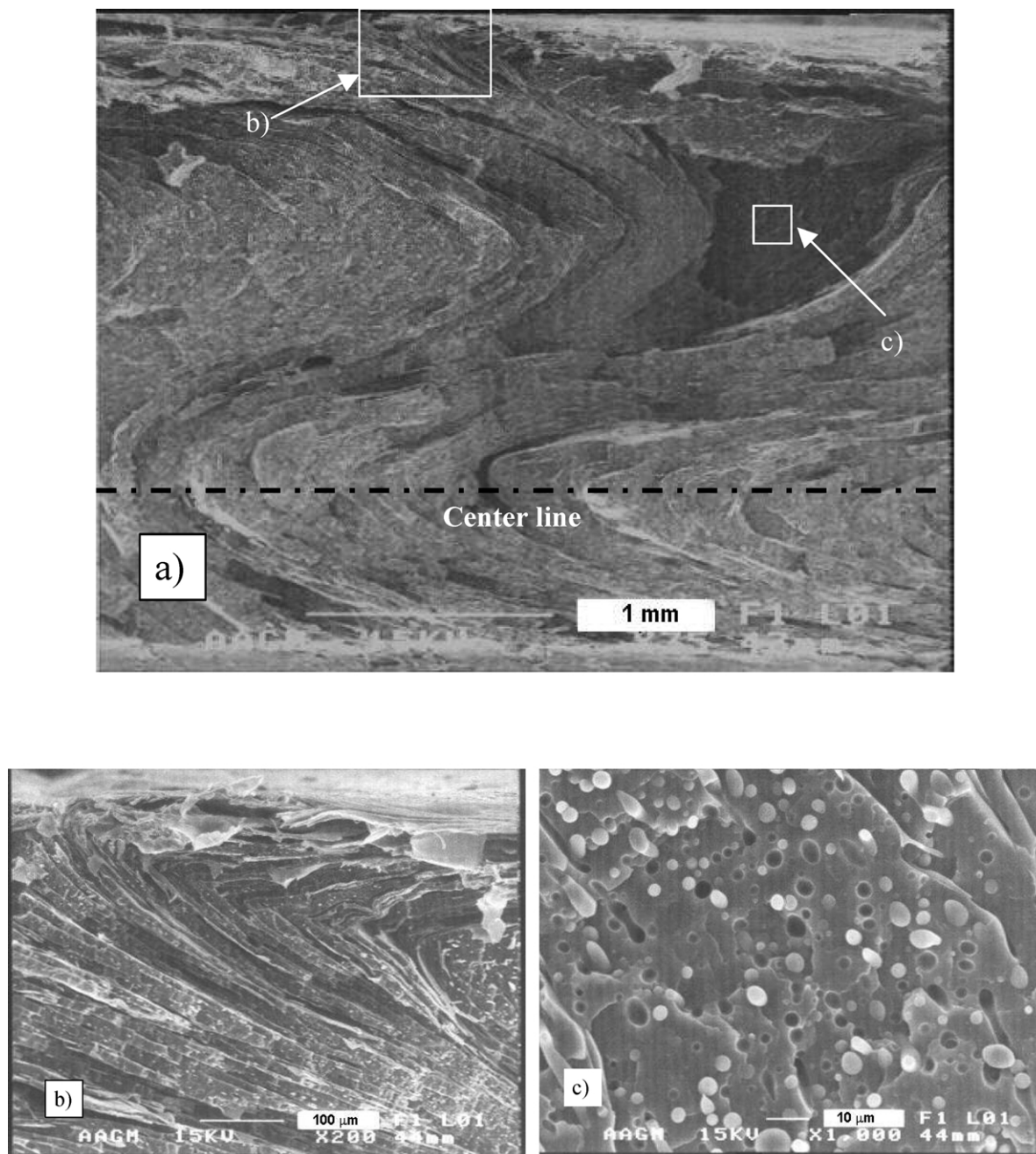


Fig. 2. SEM images of weld line region of PPPA (a), and enlarge views (b, c).

of corresponding measurements are all summarized in Table 2.

As talc content increases, slightly decreasing trend of tensile strength and weld line strength for PP are observed. As increase in filler content, the deformation become more brittle and the neck formation was not observed. On the other hand, tensile strength of PA increases with the filler content probably due to interaction between polar moieties of PA and OH groups of talc edges. However, weld line strengths of both PP and PA show decreasing trend with the filler content most probably due to the orientation of talc

plates perpendicular to flow direction at the weld line as also pointed out by some authors [6,8,9].

Viscosity of PP is lower than PA at high temperature and high shear rates peculiar to extruder. In addition to this, PP is major component in the selected composite (75/25). Thus, the PPPA blend must have a continuous PP phase in which PA is dispersed. It is clear to see from Table 2 that the tensile strength of this blend is of the same magnitude as the PP and does not exhibit significant reduction. This result may be attributed to the fact that dispersed immiscible phase forms elongated domains which are parallel to the flow direction at large sub skin region. When an external force is

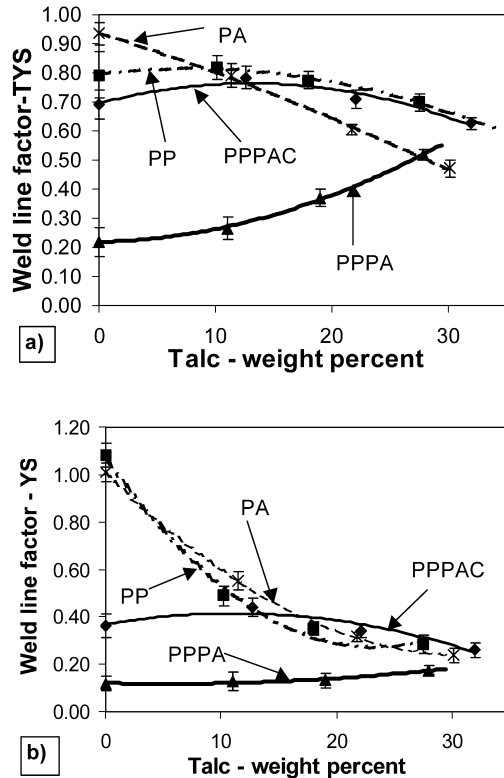


Fig. 3. Weld line factors of blends and blend composites (a) for tensile yield stress (b) for tensile strain.

applied to the matrix and these elongated disperse phase, they behave as separate but parallel springs and share the applied load as indicated previously [13–15].

On the other hand, when a weld line is present, fracture at weld line decreases both the tensile yield strength and strain significantly. That reduction can be explained by morphological observations. Uncompatibilized, unfilled blend; i.e. PPPA, has wide weld line region even detected by naked eyes. The topographic micrograph of cryogenically fractured surface of the weld line region of PPPA is shown in Fig. 2a. Here due to its electron rich character, PA phase appears as lighter regions than PP does. Shape of weld line

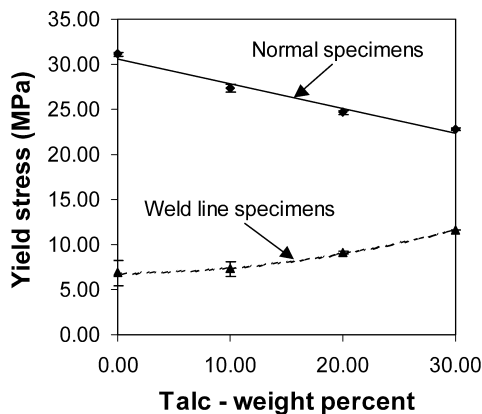


Fig. 4. Tensile yield stress versus filler level of normal and weld line specimens of PPPA composites.

Table 3

Effect of talc on melt flow index (MFI) of PP and PA composites

Sample name	MFI (gr/10 min)
PP	8.23
PP1	4.87
PP2	4.46
PP3	4.00
PA	22.1
PA1	21.63
PA2	14.10
PA3	7.44
PPPA	11.15
PPPA1	9.97
PPPA2	4.11
PPPA3	1.64

Data in parenthesis represent standard deviations. Except unfilled PP all weld line specimens break off as soon as the yield tensile strength is reached.

is not straight but shows an oxbow (second order distortion) shape, which is consistent with the result reported by Kim et al. [10] and Jarus et al. [12]. Weld line region consists of several curvatures lying parallel to each other. That curvatures are formed by elongated or stretched, thin, macro PA domains (Fig. 2b). When two concave-up flow fronts are met within the mould, due to high injection pressure and speed, their concave-up shapes are deformed to oxbow shape. Between the two fronts, there is a quite wide region of spherical PA domains dispersed through PP matrix as shown in Fig. 2c. Those spherical PA domains are also observed between parallel curvatures formed by stretch PA, but that spherical domain regions are thin as compare with the flow front meet region. Orientation of elongated domains parallel to flow direction is changed to orientation of elongated domains perpendicular to flow direction. Thus above-mentioned springs cannot share the applied force. That caused to weak mechanical properties and very low weld line factors for both tensile yield strength and strain for unfilled blend (Table 2 and Fig. 3).

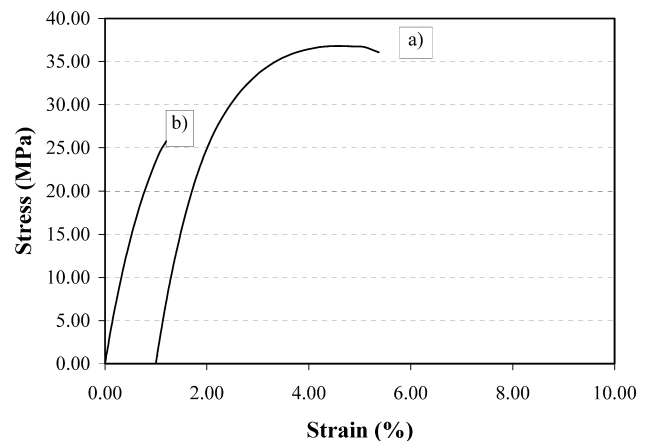


Fig. 5. Stress strain curves of (a) normal specimen of PPPAC2 (b) weld line specimen of PPPAC2.

With respect to increase in filler content, reduction of both tensile yield strength and yield strain are observed in the PPPA blend composites without weld line. Recently, effect of inorganic filler phase (talc) on mechanical and morphological properties of binary immiscible polymer blends having no weld lines was studied in detail by our group [15]. It has been found that the filler; talc, selectively dispersed in polar PA phase and weak interaction of continuous PP phase with both PA and talc dominate yield properties. Moreover, this drawback in blend composites can be eliminated by using a compatibilizer, MA-g-PP. It is well known that MA-g-PP as compatibilizer leads to chemical linkage between PP and PA phase causing much uniform morphology as indicated previously. Extra strong

interaction between the anhydride or carboxyl groups of MA-g-PP and amine end group of PA causing amide bridges exist together with the special interaction between edge hydroxides of talc and amine group of PA possibly through H-bonding [16]. As a result, co-continuous morphology can be changed to a well-dispersed two phase particle-in-matrix morphology via compatibilization which then resulted in an increased final performance of the resultant blend composites as reported in above mentioned study.

Although there is a small increase in weld line strength of PPPA1 blend composites relative to that of PPPA blend with a quite large standard deviation, as the filler level increases further it is observed that weld line strength of uncompatibilized blend composite increases, significantly. Standard

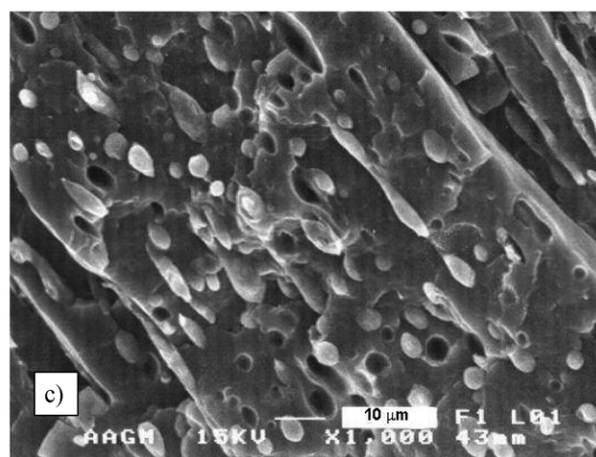
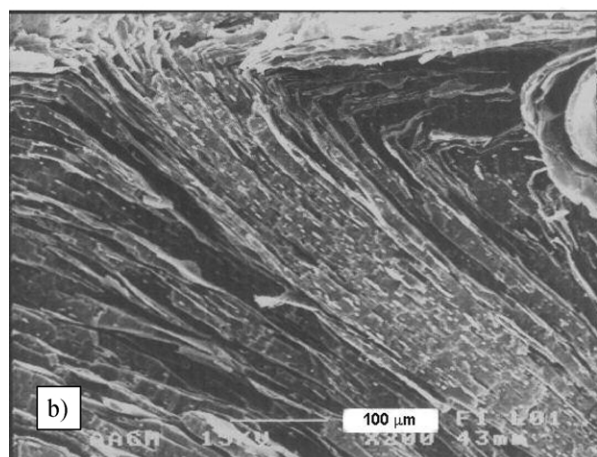
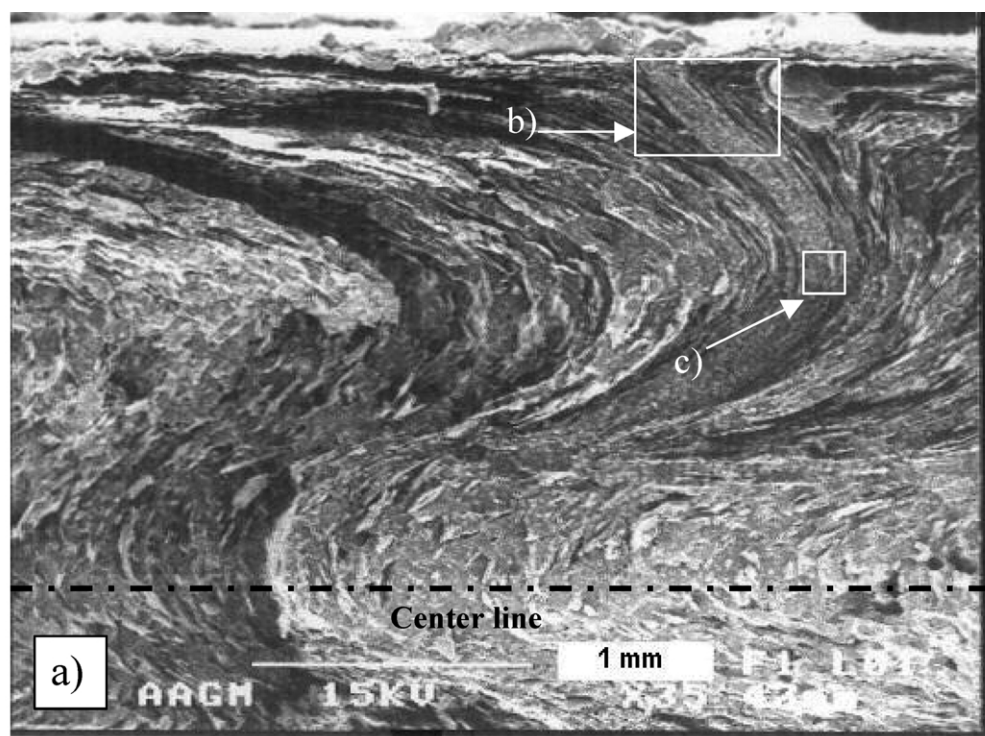


Fig. 6. SEM images of weld line region of PPPA1 (a), and enlarge views (b, c).

deviation for corresponding measurements become smaller which indicates that increasing trend of weld line strength of uncompatibilized blend by addition of talc is statistically significant. Addition of 30% talc to PPPA blend results in 69.7% increase in weld line strength. In other words, most interestingly, the addition of talc filler seems to have positive effect on the weld line strength of resultant composites (Fig. 4), although both individual parents exhibit decreasing trend in weld line strength for each filler loading degrees. Moreover, the difference between the tensile strength of the PP/PA blend composites without and with weld line is not very significant especially at higher filler loading degrees.

The presence of filler in an immiscible polymer blend

system may affect the compatibility. Nesterov and Lipatov [17] indicated theoretically that the introduction of filler that has interaction with one or both component of binary polymer mixtures increase the thermodynamic stability of the ternary systems. Moreover, it is indicated by Jarus et al. [12], in order to eliminate the elongated and oriented domains parallel to weld line that cause poor strength, increase in the viscosity of dispersed phase can be a key parameter. Similarly, the selective wetting of talc particles by PA phase seems to be beneficial to weld line strength via increasing the viscosity of the PA dispersed phase and consequently decreasing the aspect ratio of the elongated domains. Effect of talc content on MFI, an indirect measure of viscosity in molten state, of the PA phase of PPPA blend

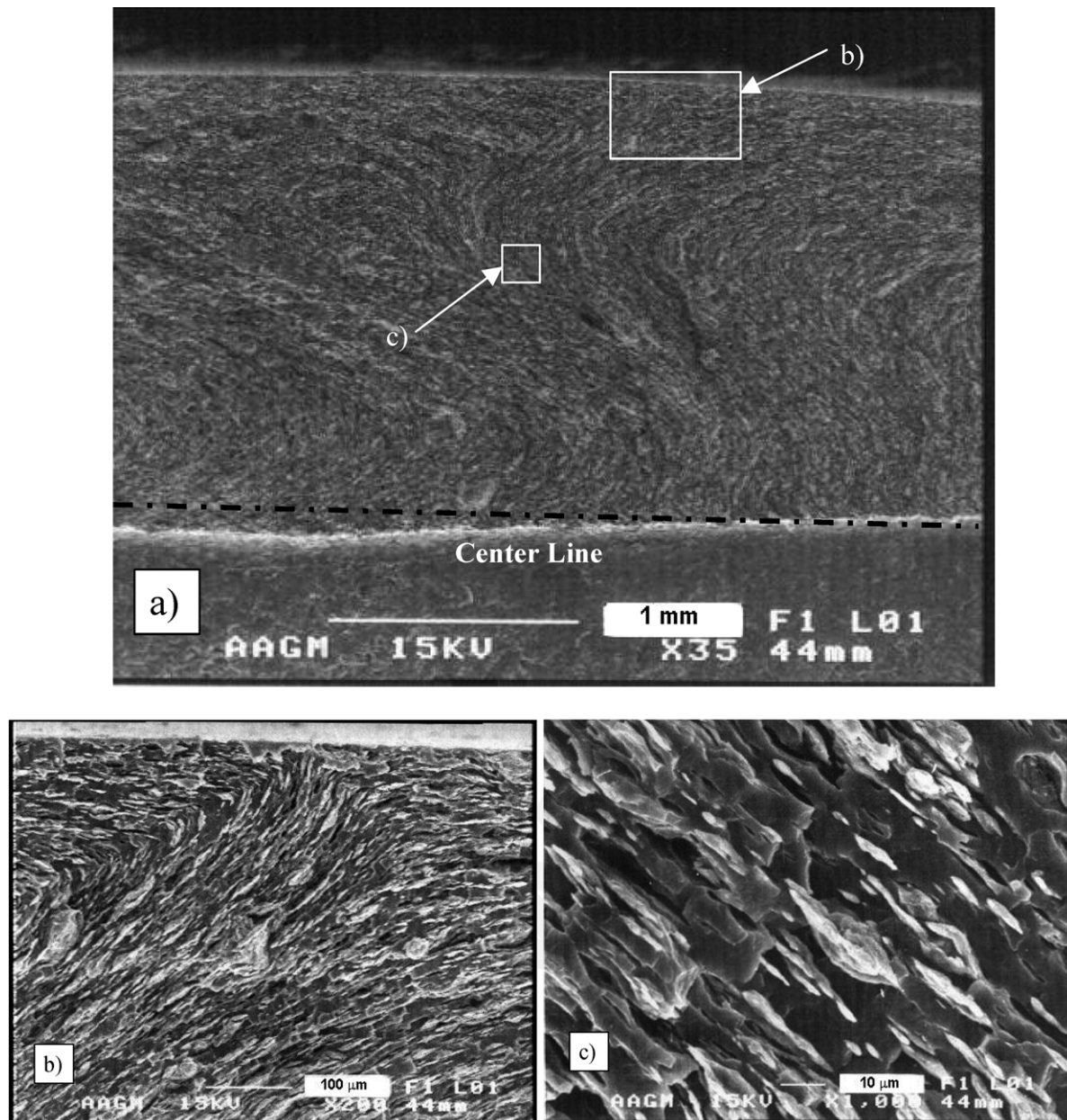


Fig. 7. SEM images of weld line region of PPPA2 (a), and enlarge views (b, c).

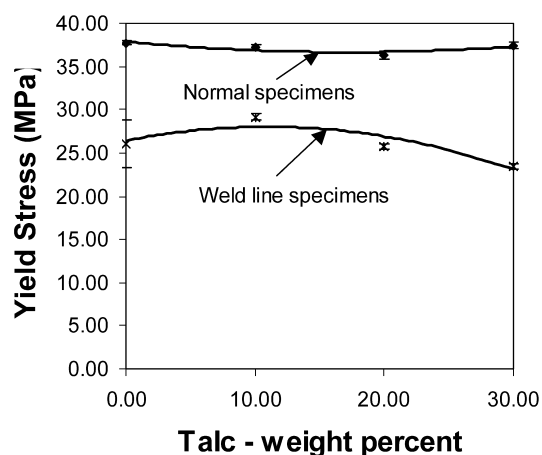


Fig. 8. Tensile yield stress versus filler level of normal and weld line specimens of PPPAC.

is well pronounced in Table 3. Introduction of filler causes to decrease in MFI or in other words to increase in viscosity of both PP and PA matrixes, but this decreasing trend in MFI is much more extensive for PA case. Addition of 10% (by weight) talc into PA does not affect the MFI of resultant composite severely, however, as the filler level increases further, reduction in MFI is much more severe. Since talc is selectively wetted by PA phase of PPPA composite blend as indicated previously [15], it mainly causes to decrease MFI or increase viscosity of PA phase which in turn viscosity of PPPA composite.

When the fracture properties given in Table 2 is examined, it can be said that trend of fracture properties of all composites without weld line with respect to increase in the filler level resemble to those of yield properties. Except unfilled PP, fracture properties of all weld line samples are identical to yield properties, since they break off suddenly at weld line region as soon as the stress level reaches to a certain level (Fig. 5).

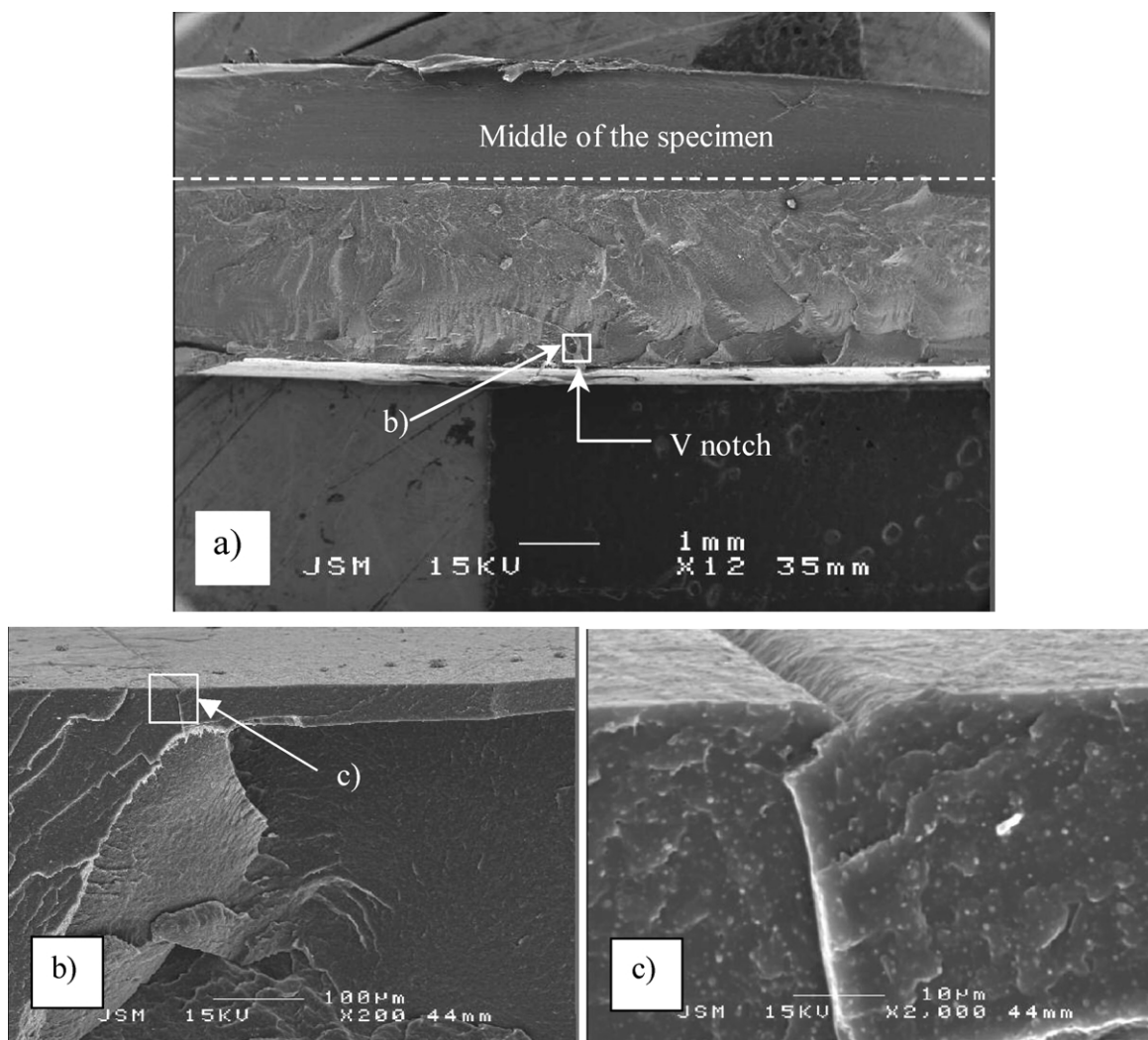


Fig. 9. SEM images of weld line region of PPPAC (a), and enlarged views (b, c).

Figs. 6 and 7 together with Fig. 2 indicate the change of morphology at the weld line region of PPPA blend and composites due to viscosity increase of dispersed PA phase with addition of talc.

When 10% talc is added into uncompatibilized PPPA blend, the width of weld line region is reduced slightly as shown in Fig. 6a. Spherical PA/talc domains (Fig. 6c) are located at the region where the two flow fronts met. Elongated PA/talc domains are surrounding that region (Fig. 6b). Unlike unfilled PPPA blend, less spherical PA domains are detected between elongated domains far from weld line. Slight change in weld line morphology may be related with small decrease in MFI of PA phase of PPPA blend composite by addition of 10% talc as indicated in Table 3.

The microstructure of weld line region is completely changed when the filler level is reached to 20%. As it is seen from Fig. 7a, the width of weld line region of PPPA2 is reduced dramatically. The curvature of weld line is hard to detect. All elongated and spherical domains of PA/talc are disappeared. Instead of them much more homogenized plate-like morphology of PA/talc domains are prevailed (Fig. 7b and c). In the region of two flow fronts met, the plate like PA/talc domains with a low aspect ratio are oriented in a similar way of elongated domains of PPPA (Fig. 7c). However, that region is very narrow. With 30% loading of filler, the structure reaches to a much more homogeneous morphology even in the weld line region. Severe change in morphology of weld line region for 20 and 30% talc loadings can be predicted from Table 3, since addition of 20 and 30% talc results in much more severe reduction in MFI of PA phase of PPPA. That is also believed to be the reason of increasing the weld line strength of resultant composites.

On the other hand, compatibilized and filled composites exhibit nearly constant tensile strength compared to uncompatibilized ones. Extra strong interaction between the anhydride or carboxyl groups of MA-g-PP and amine end group of PA and the special interaction between talc and amine group of PA lead to higher tensile properties compared to uncompatibilized composites for all level of talc. When a weld line is present, yield stress and strain increases up to 10% talc level, and then decreasing trend is recorded as shown in Table 2. Although, increase in the selective filler level can facilitate an increase in weld line strength of uncompatibilized PPPA blend, improvement of weld line strength by addition of compatibilizer is much more evident. However, when the selective filler and compatibilizer are introduced into the PPPA blend at the same time, reduction of weld line strength after a certain level of filler is interesting. It is much probable that selectivity of amine groups of PA to talc surface is higher than the anhydride groups of MA-g-PP. When talc level is increased above 10%, the abundant OH groups of talc edges overwhelms over anhydride groups of compatibilizer and causes to reduce the number of linkages between PP and

PA. But anyway, compared to uncompatibilized blend composites, compatibilized ones represent much better weld line strength for all level of talc loading as indicated in Fig. 8. In spite of decreasing trend after certain level of filler loading, the beneficial effect of both selective talc particles and compatibilizer can be observed in weld line factors of the resultant blend composites (Fig. 2).

A short weld line can be observed only at the sub-skin region of compatibilized PPPA blend (PPPAC) as it is seen in Fig. 9a and b. In addition to this, oxbow shape of weld line observed in the uncompatibilized blend cannot be identified easily. At the center of the specimen, any sign of weld line cannot be seen (Fig. 9a). Even at the skin and sub-skin region where weld line is detected, the shapes of dispersed PA domains are spherical (Fig. 9c).

Although only spherical dispersed PA domains are observed in the compatibilized blend, as filler introduced, these elongated domains in uncompatibilized composites at the weld line region are observed only at sub-skin region for all filler levels. Shape of dispersed phase near the centre region is much probably related with the plate-like shape of talc which is encapsulated by dispersed PA phase. As shown in Fig. 10, all talc filled compatibilized blend composites

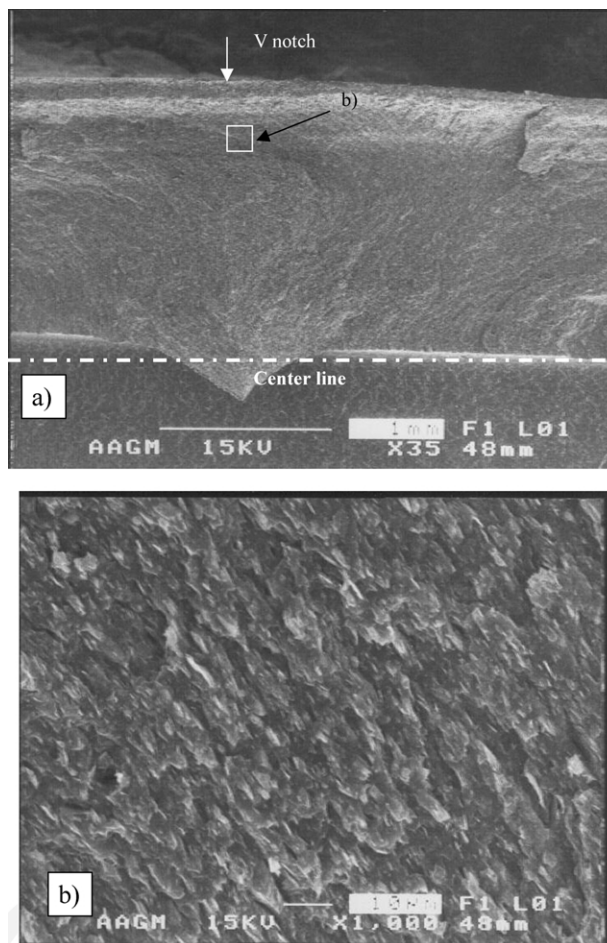


Fig. 10. SEM images of weld line region of PPPAC3 (a) and enlarge view (b).

exhibit nearly identical weld line morphology. Both the thickness of sub-skin region and the length of the elongated domains are very small compared to even 30% talc filled uncompatibilized PPPA.

4. Conclusions

Although weld line strengths of both PP and PA show decreasing trend with the filler content, the addition of talc filler seems to have positive effect on the weld line strength of resultant PPPA blend composites. SEM studies reveals that selective wetting of talc particles by PA phase gives rise to decrease both in width of weld line region and the length of elongated dispersed PA phase along the weld line via increasing the viscosity of dispersed phase.

It is also clear that the difference between the tensile strength of the PPPA blend composites without and with weld line is not very significant especially at higher filler loading degrees.

Compared to uncompatibilized blend composites, on the other hand, compatibilized blend composites represent much higher weld line strength for all levels of talc loading.

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