

# Reactive Polytetrafluoroethylene/Polyamide 6 Compounds. II. Study of the Reactivity with Respect to the Functionality of the Polytetrafluoroethylene Component and Analysis of the Notched Impact Strength of the Polytetrafluoroethylene/Polyamide 6 Compounds

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**ABSTRACT:** The formation of block copolymers as a result of an *in situ* chemical reaction during the reactive extrusion of electron-beam-irradiated polytetrafluoroethylene (PTFE) and polyamide 6 (PA) was detected indirectly with differential scanning calorimetry and Fourier transform infrared. As expected, the content of the block copolymers in the compound increased as the irradiation dose was increased. The notched impact strength showed an increase in

the PTFE/PA compounds produced with highly irradiated PTFE. This behavior is discussed in the context of the degree of dispersion of the PTFE phase (as reported in part I of this series) and the adhesion changed by the *in situ* reaction. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 98: 1317–1324, 2005

**Key words:** block copolymers; polyamides; polytetrafluoroethylene (PTFE); reactive extrusion; strength

## INTRODUCTION

Compounds of polytetrafluoroethylene (PTFE) as the dispersed phase and polyamide 6 (PA) as the matrix were prepared by reactive extrusion, as described in part I of this series.<sup>1</sup> The crucial point of this study was the characterization of the compound morphology with respect to the functionality of the PTFE components.

The basic principle of the reactive extrusion is the *in situ* transamidation reaction between the polymer components. This article presents proof of the content of the block copolymers formed in the PTFE/PA compounds by this reaction.

In a previous article,<sup>2</sup> the transesterification between amorphous polycarbonate (PC) and semicrystalline poly(butylene terephthalate) (PBT) was verified by differential scanning calorimetry (DSC) through the changes in the crystallization temperature of the PBT component and the glass-transition temperature of the PC-rich phase. In the PTFE/PA compounds under investigation, various irradiated PTFEs were used to study the influence of functionality. Because irradiation results in changes in the melting and crystallization temperatures of PTFE,<sup>3–5</sup> the crystallization

temperature of the PTFE phase is not suitable as proof of the reaction products. In addition, the glass-transition temperatures of both highly crystalline components PA and PTFE are not detectable in the compounds with the required accuracy. Therefore, another method was chosen to prove the *in situ* chemical reaction in the PTFE/PA compounds. The compounds were extracted selectively in hexafluoroisopropanol, an excellent solvent for pure PA but not for pure PTFE. Therefore, the PTFE/PA block copolymers that formed could exist in the soluble PA-rich part and the insoluble PTFE-rich part. After the separation of the compounds in the soluble and insoluble parts, the solvent was removed and investigated. The melting and crystallization processes observed by the DSC method were used to prove the manner in which the PTFE blocks existed in the soluble PA-rich phase, which were dissolved as a block copolymer together with the PA blocks. Analogously, the insoluble PTFE-rich part was investigated with respect to the PA blocks of the block copolymers, which were not dissolved. If the PTFE and PA blocks of the block copolymers were crystalline, one would expect the transition heat of PTFE in the PA-rich part and that of PA in the PTFE-rich part to increase with increasing functionality of PTFE.

In addition, both parts were also investigated by IR spectroscopy to check their chemical composition. In contrast to the DSC results, the IR spectra present both the crystalline and amorphous contents of the compo-

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ment. As a result of the transamidation and formation of block copolymers, bands of PTFE blocks in the soluble PA-rich part as well as bands of PA blocks in the insoluble PTFE-rich part were expected. In principle, the reaction products can also be detected directly with the characteristic copolymer band at  $\approx 1708 \text{ cm}^{-1}$ .<sup>6</sup> Unfortunately, this band is very small because of the low copolymer concentration, and it is partially overlapped by the strong amide band.

Furthermore, the impact strength was investigated as an important material property of the final product. The results of the Charpy notched impact strength of the reactive PTFE/PA compounds are discussed with respect to the degrees of dispersion of PTFE and the improvement in the adhesion between the PTFE particles and PA matrix due to the *in situ* compatibilization by the block copolymers.

## EXPERIMENTAL

### Materials and processing

The matrix material of the PTFE/PA compounds was PA (Miramid SH3, Leuna GmbH, Leuna, Germany). Different PTFE types were used as dispersed components: commercial PTFE Zonyl MP 1600 N as a regulated emulsion polymer, commercial PTFE Zonyl MP 1100 as an irradiated emulsion polymer (DuPont, Wilmington, DE), and PTFE TF 2025 as an irradiated emulsion polymer (Dyneon GmbH, Burgkirchen, Germany). The irradiation dose varied from 500 to 2000 kGy. The composition was 50 wt % PTFE/50 wt % PA ( $\approx 33 \text{ vol } \% \text{ PTFE}/67 \text{ vol } \% \text{ PA}$ ).

Because of the PTFE chain scission, the molecular weight decreased during irradiation, and carbonyl fluoride end groups ( $-\text{COF}$ ) and, after hydrolysis, carboxyl acid end groups ( $-\text{COOH}$ ) were formed. The sum of all these reactive groups related to the  $\text{CF}_2$ —units was functionality  $N_{e,r}$  of PTFE, which increased linearly with the increasing irradiation dose.<sup>7</sup>

The details of the reactive processing of the compounds and the special properties of the irradiated PTFE types (polymerization degree as a measure of the molecular weight,  $N_{e,r}$ , and characteristic values of the particle size) are given in part I.<sup>1</sup>

The mechanical properties were determined with specimens produced by injection molding (BA 500, Battenfeld, Kottlingbrunn, Austria) at mass temperatures similar to those of extrusion. The PTFE contents of the compounds for the investigation of the notched impact strength were varied from 7.5 to 50 wt % (from 4.5 to 33 vol %, respectively).

### Selective extraction

To detect the block copolymers that were produced during the reactive processing, the granules of reac-

tive PTFE/PA were extracted in hexafluoroisopropanol at the ambient temperature for 5 h and then centrifuged. The procedure was repeated four times. Finally, the solvent was removed, and the solids were dried carefully. As a result, insoluble PTFE-rich and soluble PA-rich parts were obtained as solid samples for the DSC, atomic force microscopy (AFM), and Fourier transform infrared (FTIR) experiments.

The selectivity of the extraction experiment was validated by reference experiments on nonreactive PTFE/PA mixtures. With the previously described procedure, the quantitative separation in a soluble part of pure PA and an insoluble part of pure PTFE was verified.

## Methods

### DSC

The DSC measurements were carried out on a DSC Q1000 (TA Instruments, New Castle, DE) from  $-30$  to  $360^\circ\text{C}$  in a nitrogen atmosphere with a scanning rate of  $\pm 20 \text{ K/min}$  and a hold time of 0.5 min at a final temperature of  $360^\circ\text{C}$ . The cycle run was first heating/cooling/second heating. The calibration of the temperature and melting heat was performed with In and Pb standards. The sample mass was 5–7 mg.

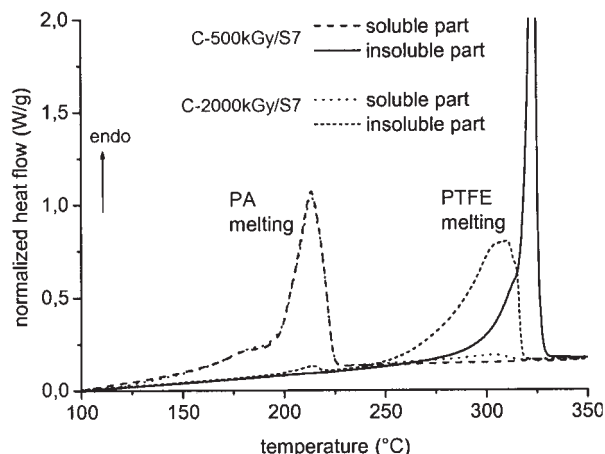
### FTIR spectroscopy

Spectra were measured from  $4000$  to  $600 \text{ cm}^{-1}$  and at a spectral resolution of  $2 \text{ cm}^{-1}$  in the diffuse reflection mode (praying mantis type, Harrick Scientific Corp., Ossining, NY) on an IFS 66 or Equinox 55 spectrometer (Bruker Optics, Ettlingen, Germany) equipped with mercury–cadmium–telluride detector. The samples were mixed with KBr powder, which was also used to record the background spectra. For each spectrum, 100 scans were coadded.

### AFM

The insoluble part of C-2000kGy/S7 was investigated with AFM to show PA blocks enriched on the PTFE particle surface. For the sample preparation, this part was dispersed in hexafluoroisopropanol, deposited on a glass slide, and dried under the ambient conditions. The sample was investigated both at the ambient temperature and after thermal annealing at  $190^\circ\text{C}$  for 4 h. The thermal treatment should permit improved visibility of the crystalline PA phase by partial melting and recrystallization of PA blocks.

The AFM measurements were performed in the tapping mode with a Dimension 3100 NanoScope IIIa (Veeco, Santa Barbara, CA) with Pointprobe silicon SPM sensors (Nanosensors, Neuchatel, Switzerland). More details are given in part I.<sup>1</sup> In the phase images,



**Figure 1** Second heating runs of the insoluble PTFE-rich part and the soluble PA-rich part of the PTFE/PA compounds C-500 kGy/S7 and C-2000 kGy/S7.

bright areas represent PTFE with low adhesion, and dark areas represent PA with higher adhesion.

#### Charpy notched impact strength

The notched impact strength was determined with the Charpy method (ISO Standard 179). The water concentration of the material under investigation had a strong influence on the mechanical properties. The water concentration was only about 1% (after storage for 20 days at 23°C and 50% moisture) instead of the required value of 3%. The test specimens were type 1 (ISO Standard 179). A notch of type A was made mechanically with a special device. The values were determined as average values over 10 specimens; the deviations of the individual specimens ranged from  $\pm 5$  to  $\pm 16\%$ .

## RESULTS AND DISCUSSION

### Detection of the block copolymers

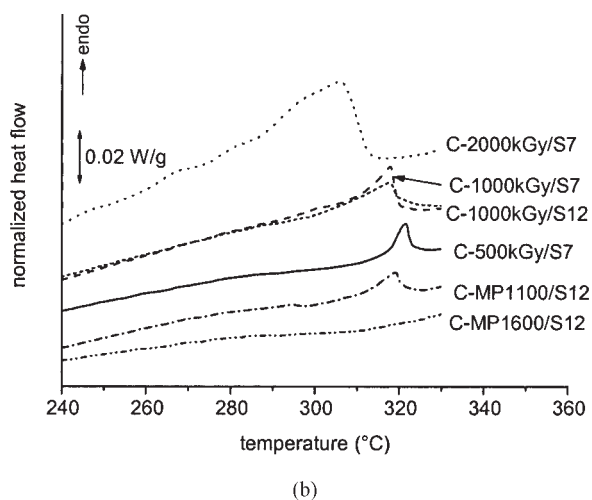
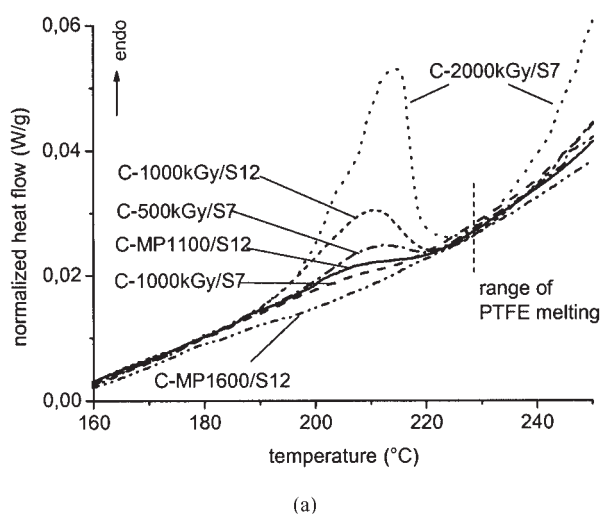
An overview of the heat flows of the insoluble PTFE-rich part and soluble PA-rich part is shown for two compounds (C-500kGy/S7 and C-2000kGy/S7) in Figure 1. The endothermic melting peak of PA in the soluble part is of the same order of magnitude for both compounds. The PTFE melting behavior of the insoluble part shows the same tendency as pure irradiated PTFE, as shown by Häußler et al.<sup>3</sup>

There are clear indications of PA melting in the insoluble PTFE-rich part and of PTFE melting in the soluble PA-rich part. The heat flow curves of both parts of all the investigated compounds are plotted in the temperature ranges of interest in Figure 2(a,b), respectively. No indication of any component other than PTFE or PA can be seen for the extracted parts of

the compound C-MP1600/S12, which was processed with the nonreactive PTFE Zonyl MP 1600 as a reference. This means that in this case the soluble part consists only of pure PA and the insoluble part consists only of pure PTFE.

On the other hand, the melting effects of PA in the insoluble parts and of PTFE in the soluble parts will be more and more characterized by increasing functionality of the irradiated PTFE used. In addition, the well-known variation of the PTFE melting behavior by irradiation<sup>4,5</sup> has been observed for the PTFE blocks in the soluble PA-rich part.

The values of the crystallization heat ( $\Delta H_c$ ) and the extrapolated crystallization onset temperature ( $T_{c,o}$ ) of PA and PTFE in the soluble and insoluble parts are listed in Tables I and II, respectively. In Figure 3, the  $T_{c,o}$  values of the PTFE component are compared with the temperatures obtained for the bulk and the frac-



**Figure 2** Second heating runs of (a) the insoluble PTFE-rich part in the temperature range of the PA melting and (b) the soluble PA-rich part in the temperature range of the PTFE melting of the PTFE/PA compounds.

**TABLE I**  
Characteristic Values of the Crystallization Behavior of the PA Component in the Soluble PA-Rich Part and the Insoluble PTFE-Rich Part of the Compounds

| Compound      | PA in the                  |                |                            |                |
|---------------|----------------------------|----------------|----------------------------|----------------|
|               | Soluble PA-rich part       |                | Insoluble PTFE-rich part   |                |
|               | $\Delta H_c$ (PA)<br>(J/g) | $T_{c,o}$ (°C) | $\Delta H_c$ (PA)<br>(J/g) | $T_{c,o}$ (°C) |
| C-MP1600/S12  | 57.1                       | 185.7          | —                          | —              |
| C-MP1100/S12  | 56.3                       | 183.5          | 0.3                        | 179.5          |
| C-500kGy/S7   | 57                         | 183.3          | 0.1                        | 183            |
| C-1000kGy/S7  | 62.4                       | 186.8          | 0.2                        | 182.7          |
| C-1000kGy/S12 | 57.6                       | 183.5          | 0.8                        | 183            |
| C-2000kGy/S7  | 55.9                       | 185.2          | 2                          | 181.6          |

Cooling rate = 20 K/min.

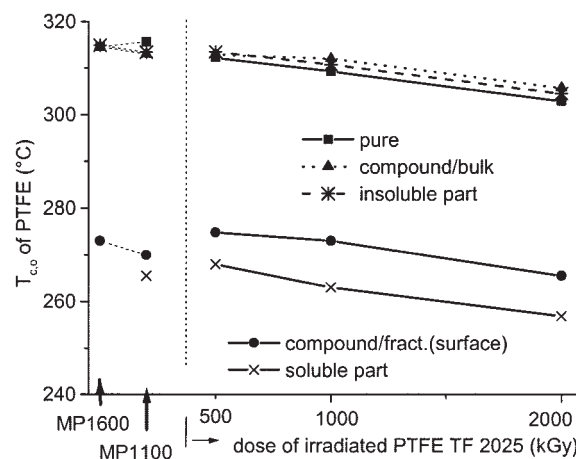
tionated crystallization step in the granules of the compound.<sup>1</sup> The values found in the insoluble PTFE-rich part are comparable in magnitude to those of pure PTFE and PTFE particles with bulk behavior.<sup>1</sup> On the other hand, PTFE in the soluble PA-rich part shows a behavior similar to that of particles crystallized by the so-called fractionated crystallization step<sup>1</sup> at lower temperatures than those of the bulk phase. The temperatures are only somewhat lower than those in the granules. It is possible that the block copolymer formation influences the crystallization temperature and causes the lower values. This problem can be clarified only by further investigation.

The complete mid-IR spectra of the insoluble and soluble parts are given in Figures 4(a) and 5(a). The baseline-corrected and normalized range of the amide I and II bands of the PA blocks in the insoluble PTFE-rich part is shown in Figure 4(b). For a better analysis of the CF<sub>2</sub>— stretching bands of PTFE blocks in the soluble PA-rich part, the difference spectra (spectra of the soluble PA-rich parts minus the spectrum of pure

**TABLE II**  
Characteristic Values of the Crystallization Behavior of the PTFE Component in the Soluble PA-Rich Part and the Insoluble PTFE-Rich Part of the Compounds

| Compound      | PTFE in the                  |                |                              |                |
|---------------|------------------------------|----------------|------------------------------|----------------|
|               | Soluble PA-rich part         |                | Insoluble PTFE-rich part     |                |
|               | $\Delta H_c$ (PTFE)<br>(J/g) | $T_{c,o}$ (°C) | $\Delta H_c$ (PTFE)<br>(J/g) | $T_{c,o}$ (°C) |
| C-MP1600/S12  | —                            | —              | 76.3                         | 314.9          |
| C-MP1100/S12  | 0.7                          | 265.5          | 70.8                         | 313.4          |
| C-500kGy/S7   | 0.4                          | 268            | 71.2                         | 313.5          |
| C-1000kGy/S7  | 1.1                          | 263            | 67.4                         | 310.7          |
| C-1000kGy/S12 | 0.7                          | 263            | 71                           | 310.9          |
| C-2000kGy/S7  | 2.9                          | 257            | 59.4                         | 304.5          |

Cooling rate = 20 K/min.

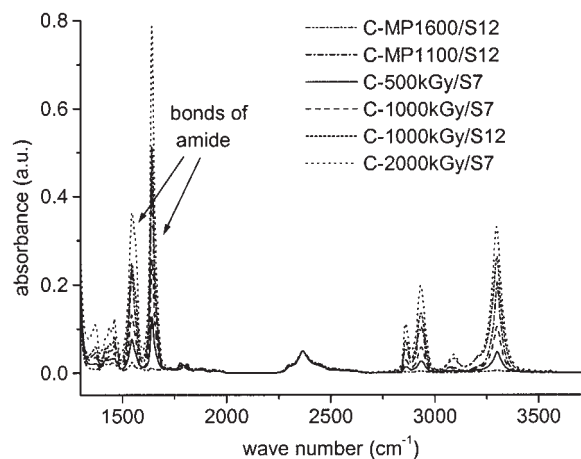


**Figure 3** Comparison of the PTFE  $T_{c,o}$  values in the initial state of the PTFE micropowder in the compound granules and in the soluble/insoluble parts of the compounds versus the PTFE type and irradiation dose, respectively (cooling rate = 20 K/min).

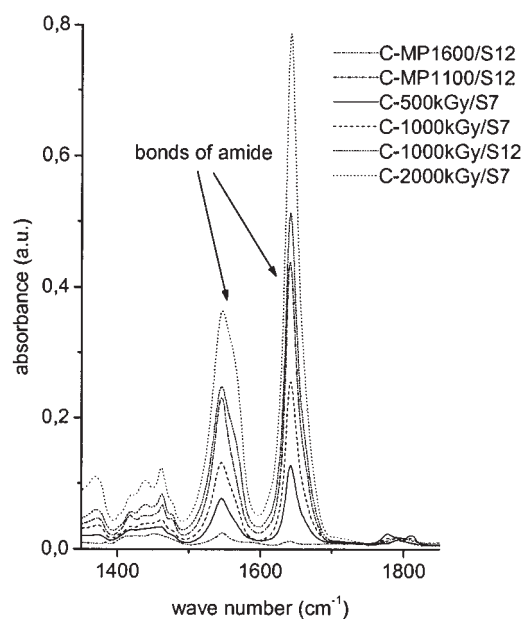
PA) have been calculated [Fig. 5(b)]. Similarly to the DSC results, the increasing content of PA blocks in the insoluble part and of PTFE blocks in the soluble part is clearly detectable at higher irradiation doses and shear rates.

The analysis of the behavior of the reactive —COOH groups in irradiated PTFE at 1810 and 1778 cm<sup>-1</sup> provides interesting aspects. The intensity of these bands determined in the initial state of the PTFE micropowder can be used as a measure of  $N_{e,r}$  of the PTFE micropowder. Only in the spectra of the insoluble parts of C-500kGy/S7 and C-1000kGy/S7 can residual reactive groups be observed [Fig. 4(c)]. Apparently, not all —COOH groups could react during processing in these two compounds. Both compounds were produced with the S7 screw configuration. No remarkable IR intensities of the reactive groups were detected in the spectra of the other samples. That means that all of them reacted during the reactive extrusion. Furthermore, the C-MP1100/S12 and C-1000kGy/S12 compounds, produced with the S12 screw configuration with the higher shear rate, did not have any residual reactive groups.

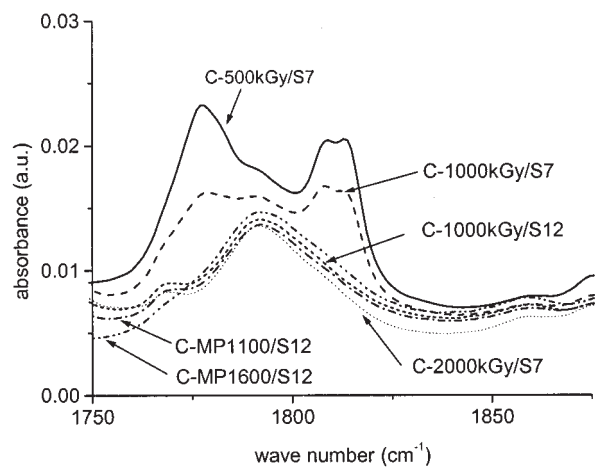
The results can be explained in the following manner: The probability of a reaction during processing is determined not only by the initial functionality but also by the PTFE particle size distribution in the compound because only reactive groups on the PTFE particle surface have the spatial order to react with PA. Accordingly, the particle distribution in the initial state and the effectiveness of the breakdown process, which determine the dispersion degree of the PTFE phase in the compound, have to be considered when the chemical reactivity is analyzed. The results of the PTFE/PA compounds produced with irradiated PTFE



(a)

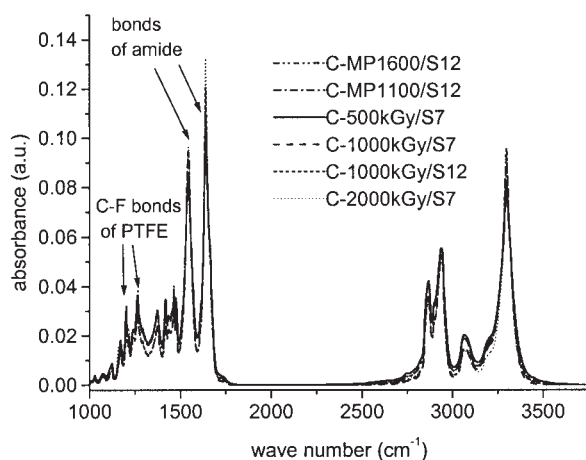


(b)

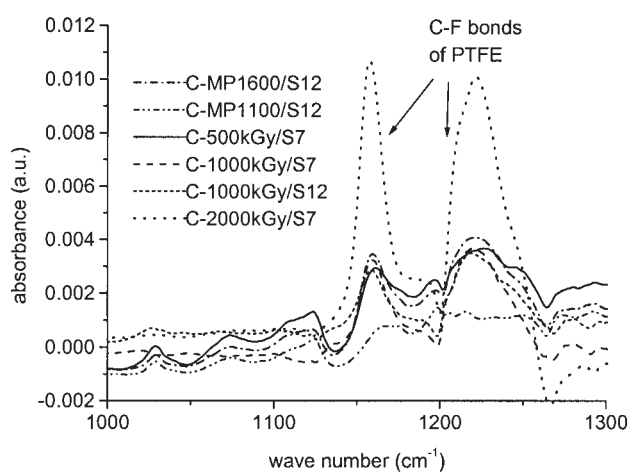


(c)

**Figure 4** FTIR spectra of the insoluble PTFE-rich parts of all the compounds: (a) overview, (b) range of the amide bonds, and (c) range of the reactive bonds of the PTFE part.



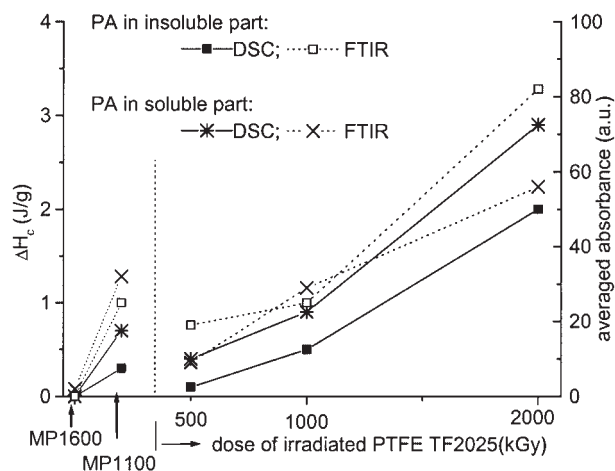
(a)



(b)

**Figure 5** FTIR spectra of the soluble PA-rich part of all the compounds: (a) overview and (b) difference spectra calculated with the spectra of pure PA in the range of the C—F bonds.

have to be discussed in more detail because the particle size distributions in the initial state of the irradiated PTFE micropowder are of the same magnitude (see Table II in Pompe et al.<sup>1</sup>). At first, the results of the C-1000kGy/S7 and C-1000kGy/S12 compounds permit the analysis of the influence of the shear rate on the particle size. The S12 screw configuration has a higher shear rate than S7. The degree of fractionated crystallization increases from 74% for C-1000kGy/S7 to 95% for C-1000kGy/S12.<sup>1</sup> Hence, the average particle size decreases as the shear rate increases. Second, the influence of the fracture energy of the agglomerates can be detected by a comparison of the compounds produced with the same S7 screw configura-



**Figure 6** Comparison of the DSC ( $\Delta H_c$ ) and FTIR results for the respective components of the insoluble and soluble parts of the compounds versus the PTFE type and irradiation dose, respectively.

tion and with various irradiated PTFEs. Increasing the irradiation dose results in a decreasing particle size as a result of the decreasing bulk fracture energy.<sup>1</sup> When the PTFE particle size is small enough, all reactive groups of the PTFE component have, with respect to the spatial order, the chance to react. Consequently, no reactive groups were found in these compounds.

A generalized conclusion can be drawn: The whole functionality of the PTFE micropowder can only be used to improve compatibility by the *in situ* reaction if an adequate screw configuration guarantees sufficient effectiveness of the breakdown process of the agglomerates. The average particle size has to be so small that most of the reactive groups of the PTFE part will be situated on the surface of the PTFE particle and a reaction with the neighboring PA molecules can take place.

In Figure 6, the  $\Delta H_c$  values (see Tables I and II) and IR absorbance are summarized with respect to the PTFE type and the irradiation dose, respectively. The irradiation dose is a measure of the PTFE functionality. For the characterization of the PA block content in the insoluble PTFE-rich phase, the average value of the absorbance of both amide bands [see Fig. 4(b)] has been used. The PTFE content in the soluble PA-rich phase is described by the average value of the absorbance of the  $\text{CF}_2$  bands [see Fig. 5(b)]. The DSC and IR methods show the same tendency of an increase in the PA content in the insoluble part and of the PTFE content in the soluble part as the irradiation dose is increased up to 2000 kGy. This confirms the formation of block copolymers with a higher content due to the increasing functionality of the PTFE micropowder used.

Finally, the composition of the insoluble PTFE-rich part and soluble PA-rich part of the C-2000kGy/S7

compound can be estimated with the values of the melting heat [ $\Delta H_f^{\text{meas}}$  (J/g)]. The content of the crystalline part [ $m_{\text{cryst}}$ (component)] is calculated as follows:

$$m_{\text{cryst}}(\text{component}) = \Delta H_f^{\text{meas}}(\text{J/g}) / \Delta H_f^0(\text{component}) \quad (1)$$

where  $\Delta H_f^0$  is the melting enthalpy for 100% crystalline polymer [ $\Delta H_f^0(\text{PA}) = 230 \text{ J/g}^8$ ] and PTFE [ $\Delta H_f^0(\text{PTFE}) = 82 \text{ J/g}^9$ ].

Under the assumption that the crystallinity of PA [ $\alpha_{\text{cr}}(\text{PA})$ ] and PTFE [ $\alpha_{\text{cr}}(\text{PTFE})$ ] in the soluble and insoluble parts is the same, as determined in the granules, the whole content of the component [ $m(\text{component})$ ] can be estimated as follows:

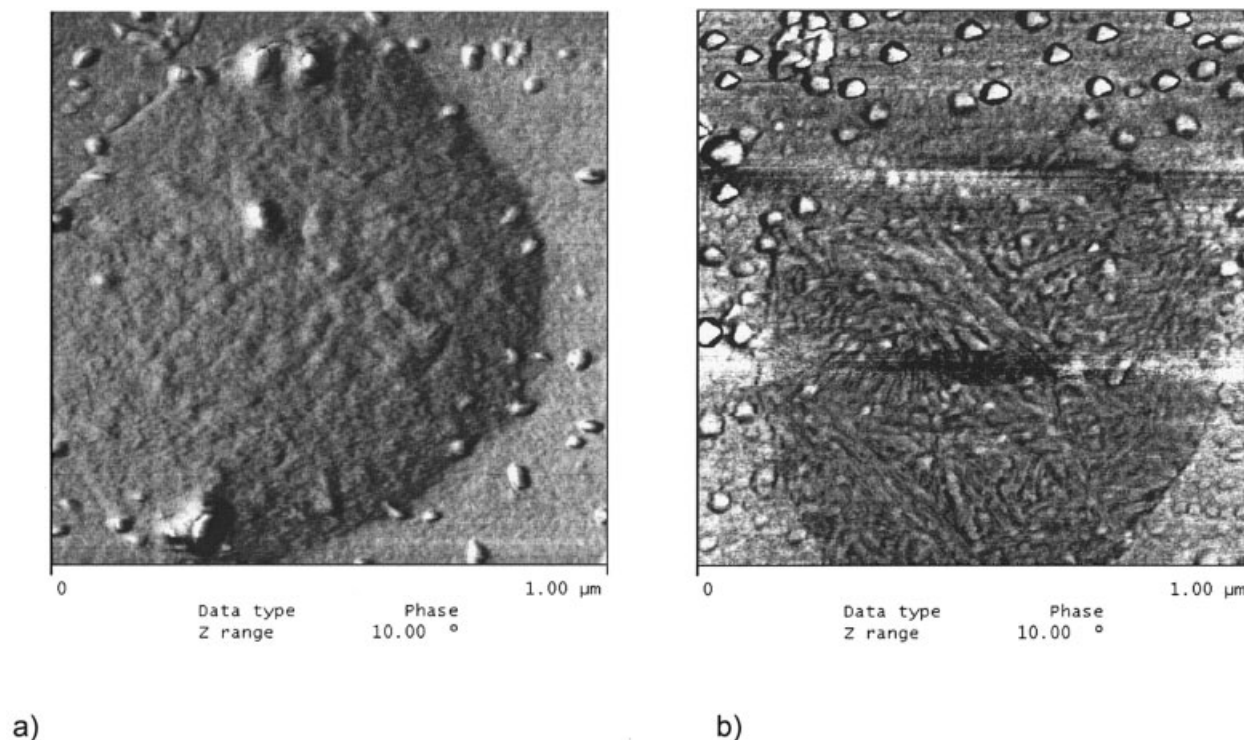
$$m(\text{component}) = m_{\text{cryst}}(\text{component}) / \alpha_{\text{cr}}(\text{component}) \quad (2)$$

With  $\alpha_{\text{cr}}(\text{PA}) = 28\%$  and  $\alpha_{\text{cr}}(\text{PTFE}) = 73\%$ , the composition of the insoluble part was determined to be 97 wt % PTFE/3 wt % PA, and that of the soluble part was determined to be 5 wt % PTFE/95 wt % PA. Unfortunately, the real contents of the block copolymers cannot be estimated because the molecular weight of the block copolymers is unknown.

To visualize the PA blocks in the insoluble PTFE-rich part, which should be arranged on the surface of the PTFE particles, AFM images were acquired of PTFE particles in the insoluble part of C-2000kGy/S7. Figure 7(a,b) shows the phase images of one PTFE particle without and with thermal treatment at 190°C for 4 h. The thermal treatment at 190°C permits a recrystallization of the PA part. The nicely visible lamellae of crystalline PA on the PTFE particle surface in Figure 7(b), as the result of this recrystallization process, confirm the presence of PA blocks.

### Notched impact strength of the compounds

The notched impact strength is dependent on the composition and the PTFE type used in the reactive PTFE/PA compounds. Some results were already published for C-2000kGy/S7 by Lehmann et al.<sup>6</sup> This article discusses the results of the compounds with different PTFE types. The point of interest is the influence of the morphology and compatibilization of the PTFE and PA components on the fracture behavior. The notched impact strength of the pure matrix polymer PA [ $a_{c,A}(\text{pure PA})$ ], determined with the same water content and the same method, is used as a reference value. Figure 8 plots the measured notched impact strength of the compound [ $a_{c,A}(\text{compound})$ ] with respect to  $a_{c,A}(\text{pure PA})$  versus the PTFE content of the compounds produced with different PTFE types. The deviations vary from  $\pm 5$  to  $\pm 16\%$ .

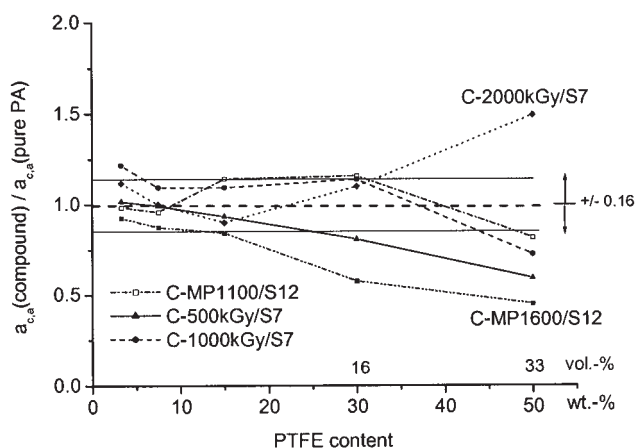


**Figure 7** AFM phase images of the PTFE particles in the insoluble part of the C-2000 kGy/S7 compound in the (a) untreated and (b) thermally treated states.

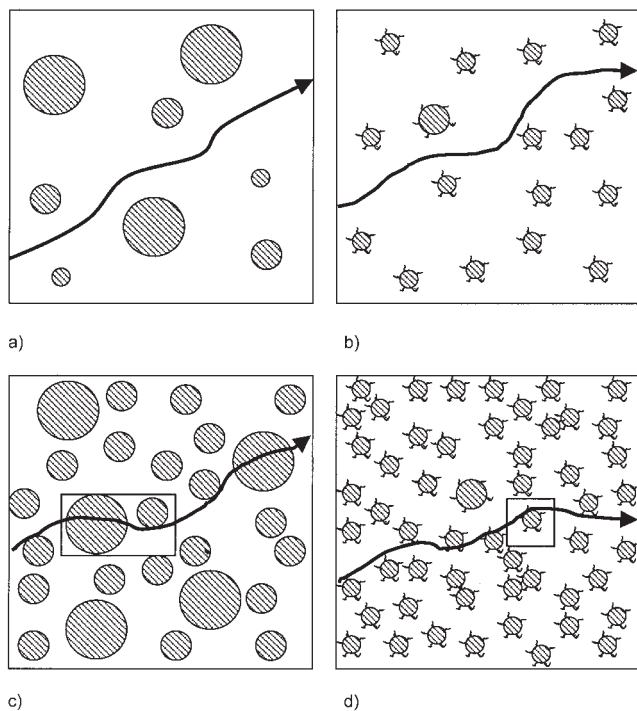
A value of the related notched impact strength of about 1 means that the behavior is governed by the PA matrix. The analyzing method is not sensitive in the range of  $1 \pm 0.16$ . This range corresponds to the range of the deviations. It seems that the matrix is responsible for the behavior observed in most of the compounds. The expected influence of the special morphology and the adhesion between the dispersed PTFE phase and PA matrix on the notched impact strength is observed only for compounds with PTFE

concentrations greater than or equal to 15 vol %. The compounds with the nonreactive PTFE Zonyl MP1600 show notched impact strengths that are lower than those of the PA matrix; the decrease approximately corresponds proportionally to the PTFE content. On the other hand, a clear increase can be observed for C-2000kGy/S7 produced with the PTFE type of the highest functionality.

With the results of the morphology and reactivity, the behavior of the notched impact strength is discussed in the following. A scheme of various compounds is presented in Figure 9. The PTFE content [9 vol % in Fig. 9(a,b) and 33 vol % in Fig. 9(c,d)] and the PTFE type [PTFE Zonyl MP1600 in Fig. 9(a,c) and PTFE irradiated with 2000 kGy in Fig. 9(b,d)] were varied. For a low PTFE content, the distance between the PTFE particles is large. Independently of the improved adhesion between PTFE and PA by the formation of block copolymers in the case of the PTFE type irradiated by 2000 kGy, the behavior of the notched impact strength is determined by the PA matrix. With increasing PTFE content, this distance decreases, and the influence of the PTFE phase increases. In the C-MP1600/S12 compound, the PTFE particle size is large and varies strongly, and the compatibility is not improved by block copolymers. Therefore, two possibilities exist for the fracture spreading. The fracture spreads both in the interface without adhesion and through the large PTFE agglomerates, which are an



**Figure 8**  $a_{c,A}(\text{compound})/a_{c,A}(\text{pure PA})$  ratio versus the PTFE content for different PTFE types [ $a_{c,A}(\text{compound})$  and  $a_{c,A}(\text{pure PA})$  were determined at the same water content].



**Figure 9** Scheme of the notched impact strength behavior with respect to the morphology of the dispersed PTFE phase and the adhesion between PTFE and PA (a,c) for compounds with PTFE Zonyl MP1600 and (b,d) for compounds with PTFE irradiated with 2000 kGy: (a,b) 9 and (c,d) 33 vol %.

association of primary particles (see part I<sup>1</sup>). The scheme of this behavior is plotted in Figure 9(c). The cryofractures of C-MP1600/S12 presented in Figure 1(a) in Pompe et al.<sup>1</sup> show that the fracture can be going through the PTFE particle.

The other extreme is the compound C-2000kGy/S7 with the finest dispersed PTFE phase (see Pompe et al.<sup>1</sup>) and the highest content of the block copolymers formed during the processing. The good adhesion in the interface results in values of the notched impact strength that are higher than that of the PA matrix, as schematically shown in Figure 9(d).

For some compounds, investigations of the notched impact strength were made with various water contents (dry after injection, 1%, and 3%). As expected, the values of the notched impact strength increased with increasing water content. However, when the values of the compound were related to  $a_{c,A}$  (pure PA) determined by the corresponding water content, an interesting effect was found. The change in the notched impact strength with increasing PTFE content for the nonreactive and highly reactive PTFE was enhanced with increasing water content. An explanation for this interesting phenomenon cannot be given at present.

## CONCLUSIONS

The characterization of reactive PTFE/PA compounds with a combination of DSC and FTIR spectroscopy has

been described with respect to the PTFE functionality. The PTFE/PA block copolymers formed *in situ* during the reactive processing have been indirectly verified. It has been shown that the varying functionality of PTFE due to the variation in the irradiation dose is the most important parameter in determining the contents of the block copolymers and also the degree of the dispersion of the PTFE phase (part I<sup>1</sup>).

From a study of the influence of the shear rate on the residual reactive —COOH groups in the PTFE component in the compound, the following conclusion can be drawn: The whole functionality of the PTFE micropowder can only be used to improve the compatibility by an *in situ* reaction if an adequate screw configuration guarantees sufficient effectiveness in the breakdown process of the agglomerates. The average particle size has to be so small that most of the reactive groups of the PTFE part will be situated on the surface of the PTFE particle and the reaction with the surrounding PA molecules can take place.

An influence of the PTFE phase on the notched impact strength has been observed only in compounds with high PTFE concentrations ( $\geq 15$  vol % = 30 wt %). With the nonreactive PTFE type, the notched impact strength of the compound is smaller than that of the PA matrix. On the other hand, the notched impact strength values are higher than the values of the PA matrix when PTFE of high functionality (irradiation dose  $\geq 1000$  kGy) is used in the compound processing. A simple model using the results of the PTFE particle size and the block copolymer content with respect to the functionality of the PTFE type can be used to explain the observed behavior.

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