

# **Localisation of carbon nanotubes in polyamide 6 blends with non-reactive and reactive rubber**

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## **Abstract**

Blending of two immiscible polymer matrices can be an effective way to combine favourable properties of both blend partners. The additional incorporation of multiwalled carbon nanotubes (MWCNTs) in such thermoplastic blends may further enhance the blend properties and especially generate electrical conductivity.

In the present study, 20 wt.% of non-reactive rubber and maleic anhydride functionalized rubber were melt blended with polyamide 6 and 3 wt.% MWCNTs by using different incorporation strategies. For the blends containing non-reactive rubber, the MWCNTs were always localised selectively in the thermodynamically preferred polyamide phase as shown by TEM images and electrical measurements. Interestingly, the different strategies resulted in different localisation behaviours of the MWCNTs in case of the reactive rubber. These findings demonstrate the significant influence of maleic anhydride groups of the rubber component on localisation of MWCNTs in the different blend phases which results in different values of electrical volume resistivity of the blends.

## Keywords

Carbon nanotubes localisation, Polymer-matrix blend, electrical resistivity

## 1. Introduction

Blending of two immiscible polymer matrices can be an effective way to combine favourable properties of both blend partners. The incorporation of rubber in polymers is a concept to enhance the toughness of the blends which was described in the literature also for polyamides [1-4]. The additional incorporation of multiwalled carbon nanotubes (MWCNTs) in such thermoplastic blends may further enhance the blend properties and especially generate electrical conductivity.

Wang et al. [5] described the addition of ultra-fine rubber particles of carboxylic styrene-butadiene latex to PA6/MWCNT composites. For PA6/MWCNT composites an electrical percolation threshold at 6 phr MWCNT was found. However, if the PA6/MWCNT composites were filled with 16 phr rubber the percolation threshold decreased to 3 phr MWCNT. By variation of the rubber content for PA6/4 phr MWCNT/rubber composites an electrical percolation threshold at 8 phr rubber was found. Also González et al. [6] described an decrease of the percolation threshold after rubber addition (styrene/ethylene-butylene/styrene triblock copolymer, SEBS) of 5-30%. The findings of both studies based on the volume exclusion effect.

In the literature same examples for reactive blending were found. The preparation of a polyamide 12 (58 wt.%) / maleated polyethylene-octene rubber (POE-g-MA, 40 wt.%) / graphene (2 wt.%) blend using different mixing strategies was described by Yan et al. [7]. It was found that the simultaneously melt mixing of all 3 components leads to randomly dispersed graphene sheets in both polymers, whereas the most sheets tend to distribute in PA12. The electrical conductivity was similar to the one of binary PA12/ 2 wt.% graphene

composite. The graphene sheets are preferentially located in PA12 if the PA12/graphene composite was blended with the rubber. For such blend the highest conductivity was found. If the polyamide was melt mixed with a rubber/graphene composite the graphene was selectively located in the rubber and the lowest conductivity of the blend was obtained due to the fact that the graphene is not able to form a conductive network in the PA12 matrix. The MWCNT localisation in PA6/POE-g-MA/MWCNT (75/20/5) blends is shown by Dasari et al. [8]. Such blends were prepared by twin-screw extrusion and simultaneously blending of all 3 components. The majority of the MWCNT are selectively located in the continuous PA6 matrix and a few MWCNT are in the rubber component. The electrical conductivities of the ternary blends are 1 decade higher than the binary composites without rubber at similar MWCNT loading indicating the volume exclusion effect.

That the blending sequence significantly influenced the blend properties like mechanical properties, microstructure, and filler location was also shown by Dasari et al. [9] for PA66/organoclay/SEBS-g-MA blends.

The role of maleic anhydride group in blend components for the nanotube localization was described from Gültner et al. [10] for PC/SAN (60 wt.%/40 wt.%) blends containing 0.5 wt.% amino-functionalized MWCNT. It was found that the MWCNT localization was independent on the mixing strategy in the PC component if the blend was non-reactive (without a maleic anhydride functionalized component). The addition of a MA-functionalized component which was miscible only with the SAN component leads to a MWCNT localization in the SAN component. It was concluded that a chemical coupling or strong interactions between MWCNT and MA-functionalized component were the driving force for the MWCNT localisation behaviour. Additionally, the content of the MA-functionalized component at a constant MWCNT concentration was varied. It was shown that a certain critical ratio of the MA-functionalized component and MWCNTs was required to achieve selective MWCNT localization in the SAN component.

The electrical percolation threshold for PA6/MWCNT composites in the literature varied by the preparation conditions. So, Kodgire et al. [11] and Krause et al. [12] found an electrical percolation threshold of 2-3 wt.% MWCNT on compression moulded PA6 films and Meincke et al. [13] found an electrical percolation threshold of around 5 wt.% on injection moulded test bars.

In the present study, non-reactive rubber and maleic anhydride functionalized rubber were melt blended with polyamide 6 using four different mixing strategies. The influence of the maleic anhydride functionalization of the rubber, the mixing strategy on the electrical properties, and the nanotube localisation were investigated.

## **2. Experimental part**

### **2.1 Material**

As polyamide 6 (PA6) matrix ULTRAMID<sup>®</sup> B27E (BASF SE, Germany) and as PA6-masterbatch PLASTICYL<sup>™</sup> PA1503 (Nanocyl S.A., Sambreville, Belgium) containing 15 wt.% Nanocyl<sup>™</sup> NC7000 (multiwalled carbon nanotubes, MWCNTs) were used. Two different rubbers were used for the blends: the non-reactive rubber AFFINITY<sup>™</sup> EG8200G (ethylene copolymer, Dow Chemical Company) and the maleic anhydride functionalized rubber EXXELOR VA1803 (ethylene copolymer, Exxon Mobil Corporation) with a maleic anhydride content in the range of 0.5 to 1.0 wt.%. The commercially available multiwalled carbon nanotube materials Nanocyl<sup>™</sup> NC7000 (Nanocyl S.A., Sambreville, Belgium) were employed. For Nanocyl<sup>™</sup> NC7000 the carbon purity is given as 90 % [14] and the bulk density was measured to be 66 kg/m<sup>3</sup>, and the mean agglomerate size was larger than 675 µm [15]. The outer diameter was determined to be 10±3 nm [16]. The nanotube length distribution as determined by TEM was characterized by a x<sub>50</sub>-value of 1341 nm and a x<sub>90</sub>-value of 3314 nm [17].

The MWCNTs were annealed to reduce the degree of the functional groups on the surface of the nanotubes at 2600°C in argon atmosphere for one hour (performed at the Leibniz Institute for Solid State and Materials Research Dresden).

## 2.2 Methods

The polyamide 6/rubber/MWCNT blends were produced using a small-scale DSM Xplore 15 twin screw microcompounder (DSM Xplore, Netherland) with a chamber volume of 15 cm<sup>3</sup>. The mixing conditions were 260°C melt temperature, 80 rpm mixing speed, and 3 min mixing time. The extruded strands were compression moulded (260°C, 50 kN) into plates (60 mm diameter, 0.3 mm thickness) using a hot press (Model PW 40 EH, Paul Otto Weber GmbH, Germany). The rubber-MWCNT masterbatch containing 7.2 wt.% MWCNT was prepared by melt mixing at 100°C with a rotation speed of 200 rpm and with a mixing time of 5 minutes.

The electrical volume resistivity of the composite materials was determined according to the standards ASTM D 4496 and ASTM D 257. The resistivity measurements on the pressed plates with resistances  $>10^7$  Ohm were performed using a Keithley 8009 Resistivity Test Fixture (open symbols in the graphs). For resistances  $<10^7$  Ohm, strips (5 mm x 30 mm x 0.3 mm) were cut from the plates and measured using a 4-point test fixture (external source electrodes spacing 16 mm and measuring electrodes spacing 10 mm, filled symbols in the graphs). Both devices were combined with a Keithley electrometer 6517A or a Keithley multimeter DMM2000 (Keithley Instruments Inc., Cleveland, USA).

For transmission electron microscopy (TEM) investigations ultra-thin sections of the samples were cut with a diamond knife at -180 °C and observed using a Zeiss Libra200MS (IPF Dresden) or using a Zeiss Libra120 (at BASF).

X-ray photoelectron spectroscopy (XPS) measurements were performed on pressed powder film samples using an AXIS ULTRA system (Kratos Analytical, UK) combined with a Mono-

Al  $K\alpha_{1,2}$  X-ray-Source (300 W at 20 mA). An analyser having pass energy of 160 eV or 20 eV was used.

### 3. Results

The localization of the MWCNTs (3 wt.%) in immiscible polyamide 6/rubber blends has been investigated. Anon-reactive and a maleic anhydride functionalized rubber were used to study the influence of a reactive group in the blend system on the blend morphology and the MWCNT localisation. Furthermore, different mixing strategies for the blend preparation were used.

The different incorporation strategies were:

- a) The MWCNTs were added in the molten PA6/rubber blend.
- b) The MWCNTs, PA6, and rubber were dry premixed and melt mixed together.
- c) A PA6/MWCNT masterbatch was diluted with polyamide and rubber.
- d) A rubber/MWCNT masterbatch was diluted with PA6.

In the first three cases, a blend composition of rubber/PA6 of 20 wt.% / 80 wt.% was chosen.

Due to the low MWCNT content in the rubber masterbatch of 7 wt.% a PA6/rubber blend with 40 wt.% rubber was prepared to reach a MWCNT content of 3 wt.% in the blend. A higher MWCNT content in the rubber/MWCNT masterbatch could not be prepared due to the strong increase of the shear force during melt mixing with increasing MWCNT concentration. The MWCNT concentration of 3 wt.% was used due to an electrical percolation threshold around 3 wt.% for PA6/MWCNT composites was found (see Figure 1). At the percolation threshold, the electrical resistivity values of the polymer change very sensitively depending on the conductive network formation of the MWCNT in the polymer.

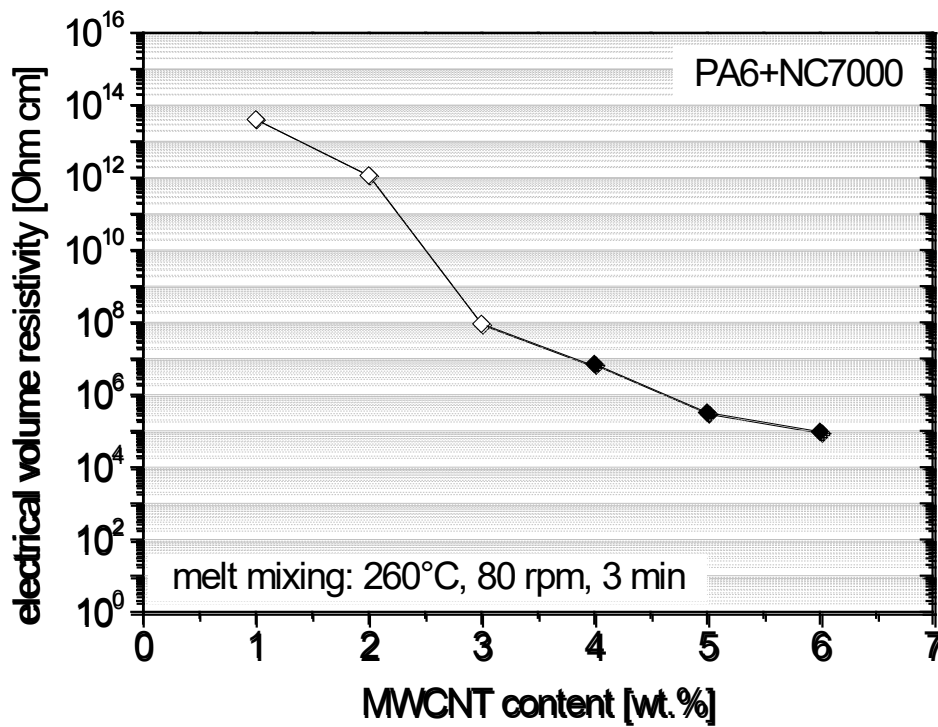


Fig. 1: Electrical volume resistivity as a function of MWCNT content for melt mixed PA6/MWCNT composites

*Blends with non-reactive rubber*

The electrical properties of the blends containing 3 wt.% MWCNT are summarised in table 1. It was observed that the blends containing the non-reactive rubber were always electrical conductive with values between  $10^4$  and  $10^7$  Ohm cm. These values are lower in comparison to the value of 3 wt.% MWCNT in PA6 at  $10^8$  Ohm cm (Figure 1). If all the MWCNTs are located in the polyamide phase of the blend, this is equivalent to a MWCNT concentration of 3.75 wt.% in the pure polyamide, which of a volume resistivity is expected around  $10^7$  Ohm cm (see Figure 1). From that it can be concluded that for the PA6 blend with the non-reactive rubber, all MWCNTs are located in the polyamide phase. Interestingly, when the rubber/MWCNT masterbatch was used or when all three components were mixed together in

one step, the values of electrical resistivity were quite low at  $2-8 \times 10^4$  Ohm cm. In both cases the nanotubes were first wetted by the rubber.

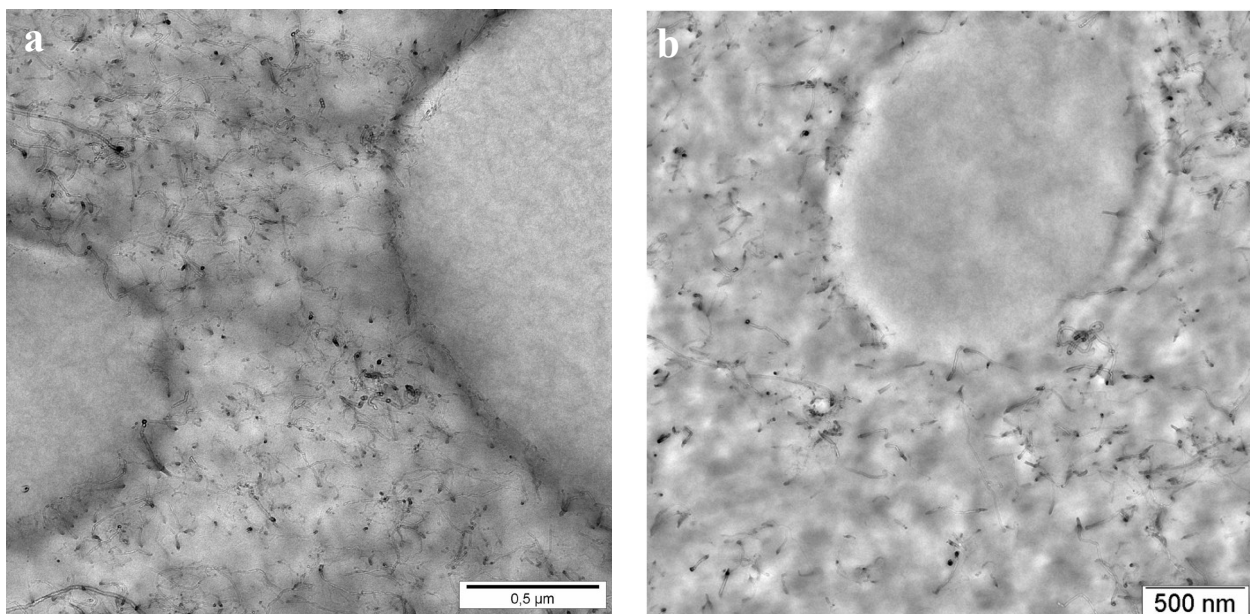
Table 1: Electrical properties of reactive and non-reactive rubber/PA6/MWCNT blends

	electrical volume resistivity [Ohm cm]			
PA6 containing 3% CNT and ....	a) MWCNTs were added to the molten PA6-rubber-Blend (rubber/PA6 20 wt.% /80 wt.%)	b) MWCNTs, rubber, and PA6 were melt mixed together (rubber/PA6 20 wt.% /80 wt.%)	c) PA6-MWCNT-masterbatch were diluted with PA6 and rubber (rubber/PA6 20 wt.% /80 wt.%)	d) Rubber-MWCNT-masterbatch were diluted with PA6 (rubber/PA6 40 wt.% /60 wt.%)
Reactive rubber (Exxelor)	2.0E+15	8.3E+14	3.9E+06	4.5E+13
Non-reactive rubber (Affinity)	9.3E+06	7.8E+04	5.6E+05	2.3E+04

When all three components were mixed together, the rubber had the lowest melting temperature whereby in the beginning of the mixing process only the rubber is able to wet the nanotubes. In the second part of the mixing if the polyamide is molten or during the dilution of the rubber/MWCNT masterbatch, the nanotubes move into the thermodynamically favoured polyamide phase. The CNT wetting by a low viscous component like the rubber-MWCNT masterbatch seemed to work as an advantage regarding on the formation of a conductive network in the composite and lower values of volume resistivity could be



achieved. Müller et al. [18] described for LLDPE/MWCNT composites the use of a low viscous poly(ethylene glycol) (PEG) as an additive to achieve lower resistivity values and an improvement of the CNT macro dispersion. It is expected that the poly(ethylene glycol) wets and infiltrates the loosely packed primary MWCNT agglomerates in early states of the melt mixing process followed by reducing the agglomerate strength. Thereby lower shear stress is needed to disperse the nanotube- agglomerates in comparison to neat MWCNT agglomerates. It can be interpreted that the non-reactive rubber in the polyamide 6 induced the similar dispersing effect like the PEG in LLDPE which is also an immiscible blend system. The morphology of the non-reactive rubber/PA6 blends was investigated using TEM and is shown in Figure 2. It was found that the nanotubes are always located in the polyamide 6 phase independent on the mixing strategy. This finding correlated well with the measured values of volume resistivity. The domain phase of the non-reactive rubber is visible. The nanotubes are well dispersed in the polyamide phase. The localisation of the MWCNT at the interface between polyamide and the rubber is not observable.



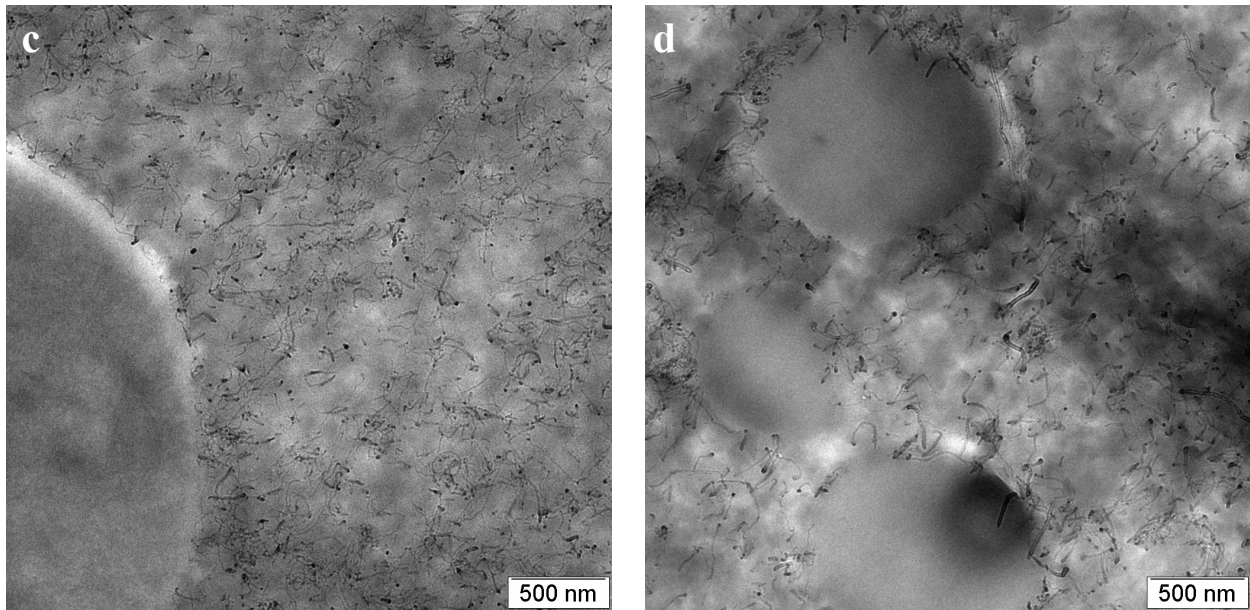


Fig. 2: TEM image of a non-reactive rubber/PA6-blend with 3 wt.% MWCNT: prepared by adding the MWCNTs to the molten PA6-rubber-blend (rubber/PA6 20 wt.% /80 wt.%) (a); prepared by melt mixed together MWCNTs, rubber, and PA6 (rubber/PA6 20 wt.% /80 wt.%) (b); prepared by the dilution of a PA6/MWCNT-masterbatch with PA6 and non-reactive rubber (rubber/PA6 20 wt.% /80 wt.%) (c); prepared by the dilution of a non-reactive rubber MWCNT-masterbatch with PA6 (rubber/PA6 40 wt.% /60 wt.%) (d). The MWCNT are always completely located in the PA6 phase.

#### *Blends with reactive rubber*

The electrical properties of the PA6/reactive rubber/MWCNT blends differ strongly with the applied mixing strategy (table 1). Only in the case that the PA6/MWCNT masterbatch was used for the blend preparation, the blend was electrical conductive. Otherwise, the blends were non-conductive. Based on this observation, it can be concluded that not all nanotubes were located only in the polyamide 6 phase. The only one conductive sample with the volume resistivity of  $4 \times 10^6$  Ohm cm could be assumed that the nanotubes are located mainly in the polyamide phase because this value is in the range of the expected value for the PA6/MWCNT composite (see discussion above).

To investigate the localisation of the nanotubes TEM studies were performed. For two representative blends, a nitrogen mapping was done by EFTEM (Energy Filtered TEM). Figure 3a shows a bright field TEM image of a reactive rubber/PA6 blend prepared by the dilution of a rubber/MWCNT masterbatch with PA6 (rubber/PA6 40 wt.% /60 wt.%). The corresponding N-map is shown in Fig. 3b and demonstrates the localization of MWCNTs in the rubber phase which appears black. For the other non-conductive blends (melt mixed the three components together or added the MWCNTs to the molten blend) TEM images indicate that the localisation of MWCNTs was not selective and the MWCNTs were localized in both phases.

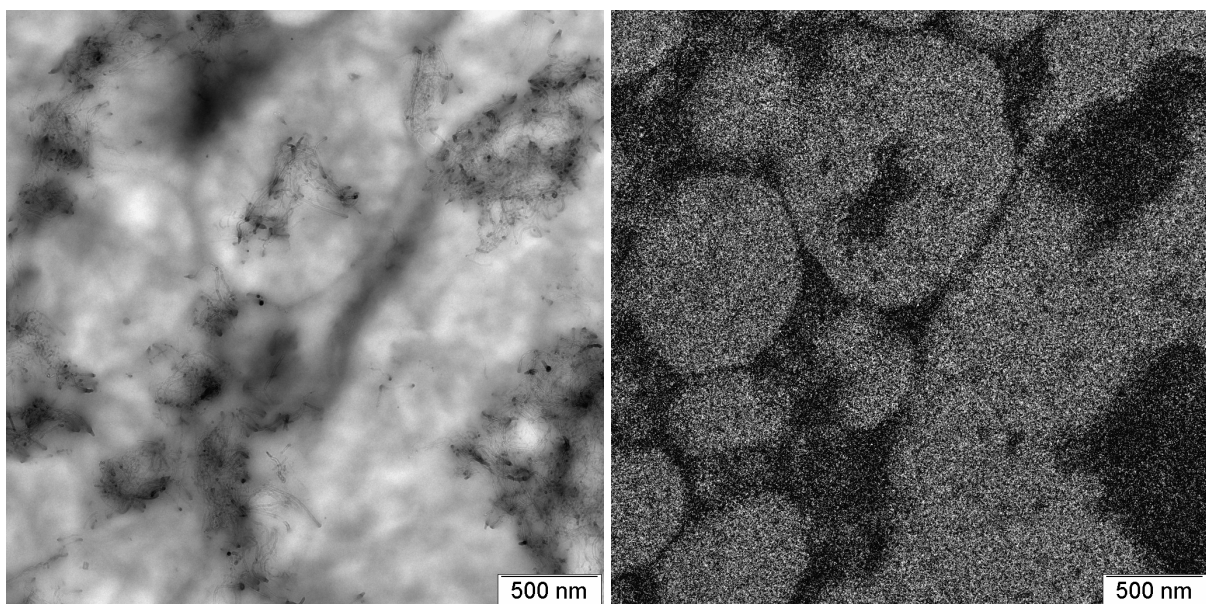


Fig. 3: TEM images of a reactive rubber/PA6-blend prepared by the dilution of a rubber MWCNT-masterbatch with PA6 (rubber/PA6 40 wt.% /60 wt.%): bright field image (left), Nitrogen-Map (right, areas containing nitrogen appear bright). The MWCNT are completely located in the rubber phase.

Furthermore, for the only conductive blend prepared by dilution of a PA6/MWCNT masterbatch with PA6 and reactive rubber, the bright field image and the corresponding N-

map are shown in Figure 4. In this case, the nanotubes are completely located in the phase which appears bright meaning the polyamide phase. This result correlated with the measured electrical properties.

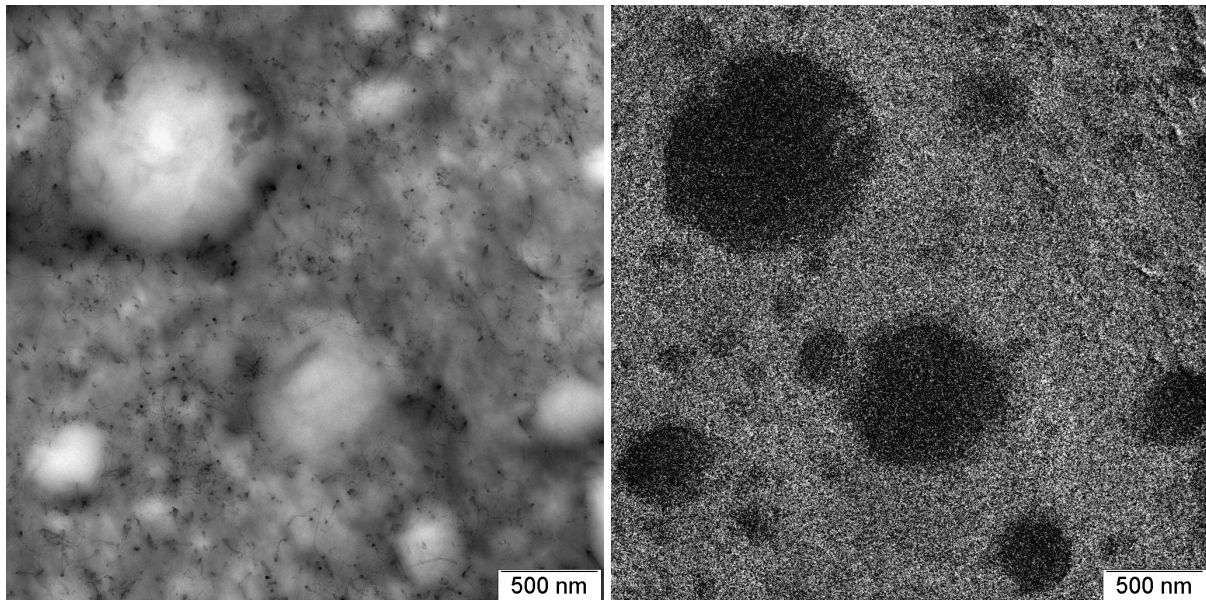


Fig. 4: TEM images of a reactive rubber/PA6-blend prepared by the dilution of a PA6/MWCNT-masterbatch with PA6 and reactive rubber (rubber/PA6 20 wt.% /80 wt.%): bright field image (left), Nitrogen-Mapping (right, areas containing nitrogen appear bright). The MWCNT are completely located in the PA6 phase.

Summarizing, it was found that the different strategies resulted in case of the reactive rubber in different localisation behaviours of the MWCNTs. This finding correlated well with the results from Yan et al. [7] and Dasari et al. [9]. As one reason for this finding the differences in the wettability of MWCNTs by the two polymers can be discussed. Due to the lower melting temperature and the lower melt viscosity of the reactive rubber in comparison to the polyamide a better wettability and infiltration of the MWCNTs is given. On the other side, a chemical reaction or adsorption between the maleic anhydride groups of the reactive rubber and the MWCNT have to be taken in to account as well. Even if these nanotubes are as-

produced non-functionalised tubes, in XPS measurements a  $[O]:[C]_{\text{spec}}$  ratio of 0.008 could be detected from which it can be hydroxyl- or carboxyl-groups located on the surface of MWCNTs. These functional groups of the MWCNTs are able to react with the maleic anhydride groups of the rubber followed by an immobilisation of the MWCNT in the rubber phase. With it this reaction can influence the localisation of MWCNTs. Such behaviour was also described from Gültner et al. [10]. Only in the case of the PA6/MWCNT masterbatch which used for the blend preparation, the MWCNT were completely located in the polyamide phase and their contact with the rubber phase is unlikely. Therefore, the MWCNTs remain in the thermodynamic preferred polyamide phase and a lower volume resistivity could be measured. In all other three mixing strategies it is possible that the dry MWCNT can wet by the rubber during the melt mixing especially if the process method with the rubber/MWCNT masterbatch was chosen. Especially, the comparison to the blends containing the non-reactive rubber shows that a chemical reaction between the rubber and the MWCNTs is a reason for the differences in the localisation behaviour of the MWCNTs.

An additional strategy to prove whether the chemical reaction between the MWCNT and the reactive rubber is the reason for the MWCNT localisation in the rubber phase is the use of really non-functionalised MWCNT. For this purpose the MWCNT were annealed. XPS measurements showed that the annealing leads to a reduction of the oxygen content of the MWCNT to a  $[O]:[C]_{\text{spec}}$  ratio of 0.002. Thereby the preparation of really non-functionalised MWCNT was not achieved but the degree of functionalization was significantly reduced. The PA6/rubber/annealed MWCNT blend was prepared using a rubber/annealed MWCNT masterbatch (strategy d) to maximise the contact between the maleic anhydride groups of the reactive rubber and the residual functionalised groups of the annealed MWCNTs. The electrical volume resistivity was found to be  $10^9$  Ohm cm. In comparison to the blend containing the as-produced MWCNT the volume resistivity is reduced by about 4 decades. However, the volume resistivity of the PA6/rubber/annealed MWCNT blend is 5 decades

higher than the PA6 blend containing non-reactive rubber and as-produced MWCNT.

Therefore, it can be concluded that more but not all annealed MWCNT are in the polyamide phase than if as-produced MWCNT were used in the blend.

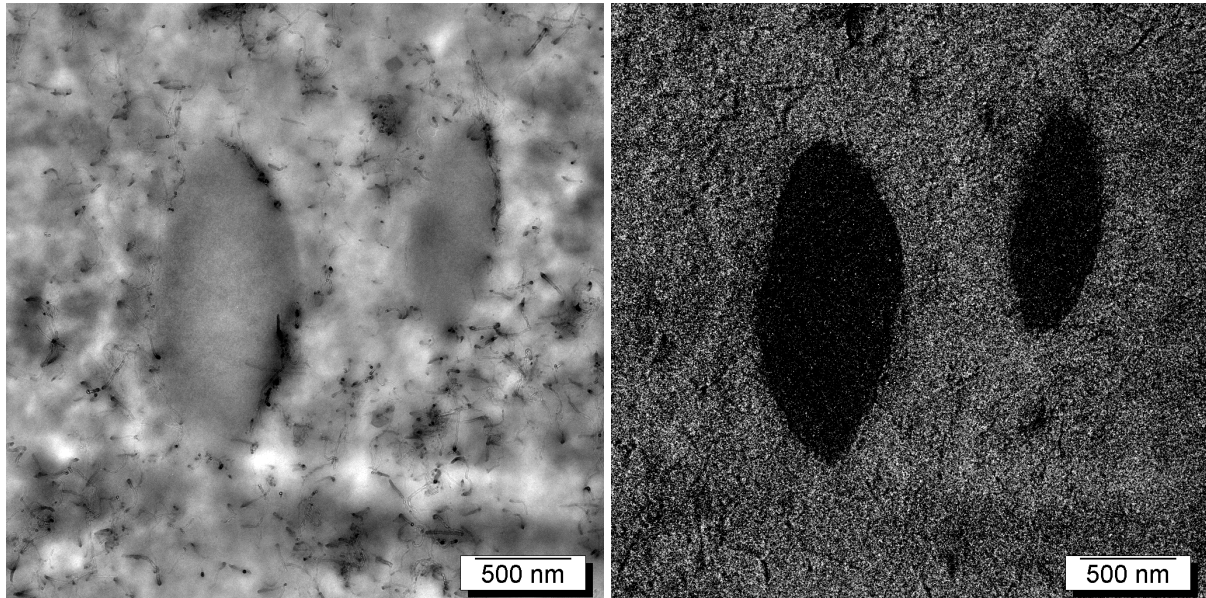


Fig. 5: TEM images of a reactive rubber/PA6-blend prepared by the dilution of a rubber/annealed MWCNT-masterbatch with PA6 (rubber/PA6 40 wt.% /60 wt.%): bright field image (left), Nitrogen-Mapping (right, areas containing nitrogen appear bright). The MWCNT are located in the PA6 phase and in the interface.

To investigate the localisation of the annealed nanotubes a nitrogen mapping was done by EFTEM. Figure 5a shows a bright field TEM image of a reactive rubber/PA6 blend prepared by the dilution of a rubber/annealed MWCNT masterbatch with PA6 (rubber/PA6 40 wt.% /60 wt.%, strategy d). The corresponding N-map is shown in Figure 5b and demonstrates that the main part of the annealed MWCNTs is localised in the polyamide phase which appears bright. A small part of the MWCNTs is also visible at the interface. It can be assumed that these MWCNTs contain some residual oxygen. Summarizing the investigation with the annealed MWCNT in the rubber/PA6 blend, a phase transfer of the nanotubes from the rubber phase to the polyamide phase takes place due to the chemical reaction between MWCNT and

reactive rubber becomes less important. Obviously, the chemical reaction between the carboxylic groups of the surface of the as-produced MWCNT and the maleic anhydride groups of the reactive rubber is the reason for the localisation in the reactive rubber phase which is not the thermodynamic favoured phase in comparison to polyamide 6.

### **Summary and Conclusion**

In this study, it could be shown that in blends containing non-reactive rubber the MWCNTs were always localised selectively in the polyamide phase as visualized by transmission electron microscopy (TEM) images. This seems to be the thermodynamically preferred phase. Independent of the mixing strategy the blends were electrically conductive.

If the reactive rubber was used for the preparation of the blends, the incorporation strategy influenced significantly the localisation of the nanotubes and the electrical volume conductivity of the blend. Only if a polyamide-MWCNT-masterbatch was used for the blend manufacturing, a conductive blend with electrical volume conductivity of  $4 \times 10^6$  Ohm cm could be obtained. In all other cases the blends were non-conductive. It may be assumed that nanotubes with functional groups first coming in contact with the reactive rubber, either by incorporating them in a rubber-masterbatch or the molten blend or as the rubber melts first during mixing all components together, may be trapped in that phase. TEM images indicate that the localisation of MWCNTs was not selectively; the MWCNTs were localized in both blend phases.

These findings demonstrate the significant influence of maleic anhydride groups of the rubber component on the localisation of MWCNTs in the different blend phases which results in different values of electrical volume conductivity of the blends.

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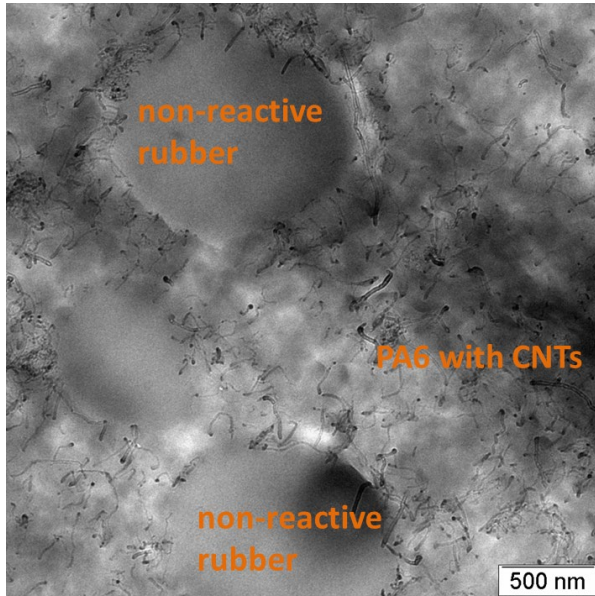
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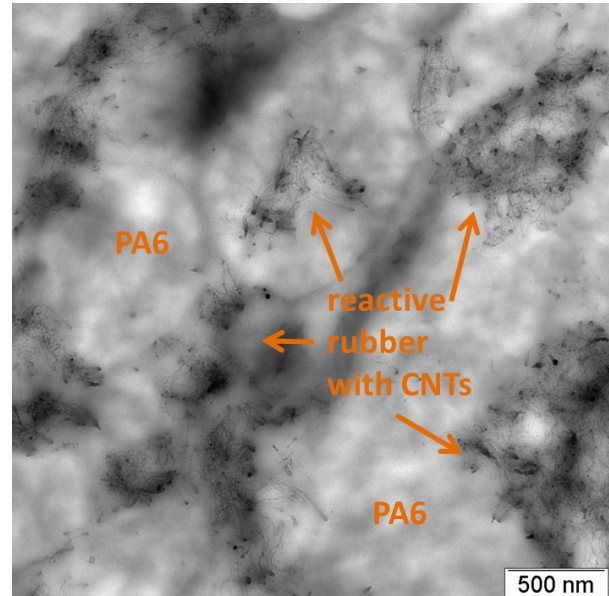
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## Graphical abstract TOC

### Localization of MWCNT in PA6/rubber (60 wt.% /40 wt.%) blends



Selective MWCNT localization in the PA6 component by using a non-reactive rubber



Selective MWCNT localization in the rubber component by using a reactive rubber

## Highlights

- MWCNT localization in PA6/rubber blends is influenced by incorporation strategy.
- For non-reactive rubber the MWCNTs are always localized in the PA6 component.
- As-grown MWCNTs can react with maleic anhydride functionalized rubber.
- Reaction with reactive rubber is depending on oxygen groups at the MWCNT surface.
- MWCNT localization influences the electrical properties of PA6/rubber blends.