Reinforcement of bio-based polymers – a study of the distribution and exfoliation of nanofillers by a set of complementary methods

M. Pinnow , J, Ganster, A. Bohn., G. Engelmann

Fraunhofer–Institute for Applied Polymer Research
Geiselbergstraße 69, 14476 Potsdam-Golm, Germany

manfred.pinnow@iap.fraunhofer.de
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In recent years bio-based polymers have been arousing interest as matrix materials in a wide variety of composites. The reinforcement of these materials by nanoparticles is very promising due to a large aspect ratio. Further, the properties of nanocomposites are essentially determined by the interaction between additives and matrix material, as well as by the spatial distribution and the orientation of added particles. Electron microscopy is a useful tool to investigate these characteristics, however, additional methods have to be applied for a comprehensive study of the correlation between structure and mechanical properties of thermoplastic nanocomposites. Complemental methods will be introduced and exemplified with different matrix materials and fillers. Polybutylene terephthalate (PBT), polytrimethylene terephthalate (PTT) and Polyetherimide were extruded with commercially available and organically modified clays (Nanofil2 and Nanofil8, Süd-Chemie AG) with a filler content mostly of 3 w%. SEM-micrographs show that these fillers consist of nanoplatelets with about 500 nm in diameter and 10 to 20 nm in thickness.

The spatial distribution of nanoplatelets was studied preferably by transmission electron microscopy (TEM) with a CM 200 (Philps, Netherlands) at an acceleration voltage of 120 kV. However, during preparation of ultra-thin cuts (thickness about 60 nm) by a microtome at cryogenic conditions (-100 °C), the position and orientation of the Nanofil-platelets may be changed or they might be even pulled out by the diamond knife (Fig. 1). This effect is caused by the hardness of the nanopletelets and is affected by the adhesion to the matrix material, as will be exemplified by the investigation of different polymer matrices.

Alternatively, to avoid the appearance of such preparation artifacts, the polymer matrix was etched in a low pressure plasma (about 2 mbar) in oxygen atmosphere and finally the Nanofil-scaffold was investigated by scanning electron microscopy (SEM, JSM 6330F, Jeol, Japan) at an acceleration voltage of 5 kV. The SEM micrographs of plasma-etched surfaces and cryo-fractured cross sections of nanocomposites showed that in a surface layer the Nanofil-platelets are oriented parallel to the outer sample surface (Fig. 2a), while accordingly in the SEM cross section micrograph they appear perpendicularly (Fig. 2b). in contrast, in the middle of the sample the Nanofil plates exhibit no preferential orientation. This behavior is caused by the flow conditions of the polymer melt during mould filling, i.e. high shear gradients in the outer regions and elongational flow in the centre of the sample.

Investigations using wide angle X-ray scattering supplement the TEM/SEM results. Analyzing the strong layer diffraction peaks (001) of the silicate layers distributed in the composite, changes in peak position and intensity were used to identify the intercalation or exfoliation of the nanoplatelets.

It will be demonstrated that the combination of the applied methods is very helpful in studying the effects of various processing parameters and different nanofillers on the structure formation and induced changes of nanocomposite´s mechanical properties.

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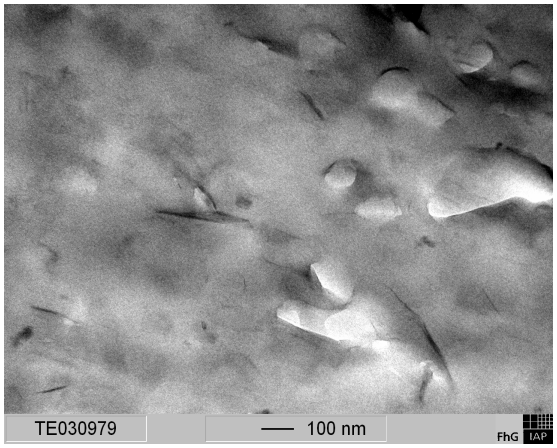
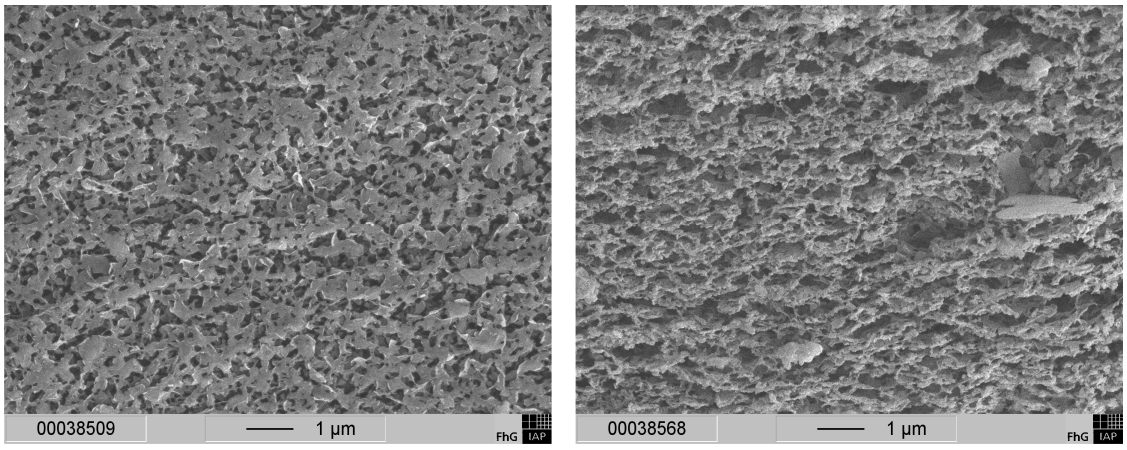


Figure 1. TEM-micrograph of ultra-thin cross section of a PTT-composite performed with nanofiller (3 w% Nanofil2) showing places of pulled out nanoplatelets



a) b)

Figure 2. SEM-micrographs of the surface (a) and the fractured cross section (b) of plasma etched PTT-composite performed with nanofiller (3 w% Nanofil2) showing flat and perpendicular oriented nanoplatelets, respectively

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