Newsletter TECMAT - 2

In this newsletter

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Recap

In TECMAT, the possibility of developing compounds that combine high conductivity with good processability is being investigated. At Centexbel, we focus on compounds for filament extrusion. We try to increase the processability of conductive formulations by using blends consisting of two incompatible polymers (Fig. 1). Here, the conductive additives are added to the phase with the lowest mass percentage in the blend. This would give rise to a double percolation, where a certain level of conductivity can still be achieved despite using a lower concentration of conductive additive.

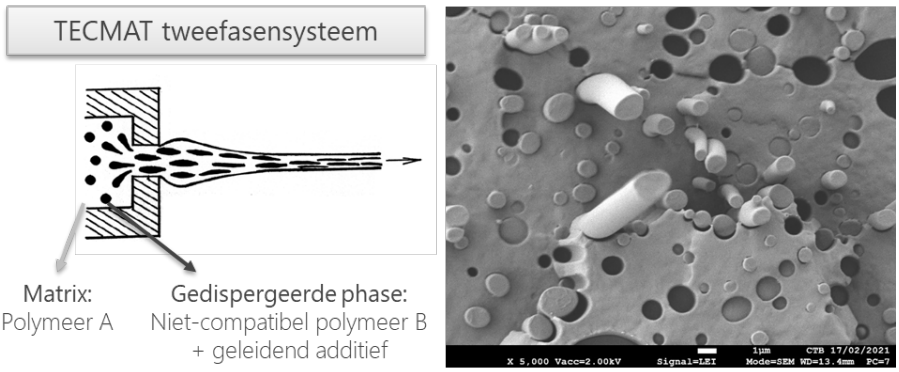

Fig. 1 Presentation of the TECMAT two-phase system for improved processability of conductive materials.

A first set of experiments with dry blends of PP with PA6-CB (carbon black) masterbatches were already explained in previous newsletter. The results of these, as well as of the subsequent study with PA6-CNT (carbon nanotubes) masterbatches (MB) blended with PP with varying MFI, were presented at the previous guidance group meeting. The latter are also briefly summarised in the present newsletter. Furthermore, a brief introduction of the follow-up experiments, in which the influence of various factors, especially dilution, compatibilisation and compounding, were studied, is already given. The results will be presented in detail at the next meeting.

Influence of viscosity

To study the influence of matrix viscosity as well as processing parameters on filament properties, 3 PP grades were selected with MFI 3, 15 and 25, respectively. These were hand-mixed with a PA6-CNT masterbatch (15% CNT) in 90/10 ratio, resulting in a total concentration of 1.5% CNT.

Fig. 2 Pictures of 90/10 PP/PA6-CNT MB filaments produced with a regular (left) and Maddock (right) screw.

Visually, the very inhomogeneous distribution and dispersion of the PA6-CNT phase in PP could already be observed. Following this, it was decided to switch from a standard screw to a Maddock mixing screw, which already brought about a marked improvement (see Fig. 1). In addition, we experienced a steady increase in extruder pressure with eventual leakage at the spin head due to clogging of the filter. The results are very different from the initial tests with CB-filled and unfilled blends.

The best result in terms of processability and filament quality was achieved with the PP grade with MFI 3. Provided sufficient stretching (DR 8x), decent tensile properties were also achieved. However, the filaments showed very low conductivity (resistivity >109 Ω.cm), although a factor 10 improvement was achieved with the Maddock screw. Other processing parameters had no obvious influence on the properties of the filaments

In summary, mainly filaments with rough surface and low conductivity were obtained when using CNT as conductive filler. Via electron microscopy (SEM), the poor distribution and dispersion of the filled PA6 phase in the PP matrix, even after using the mixing screw, could be visualised (Fig. 3).

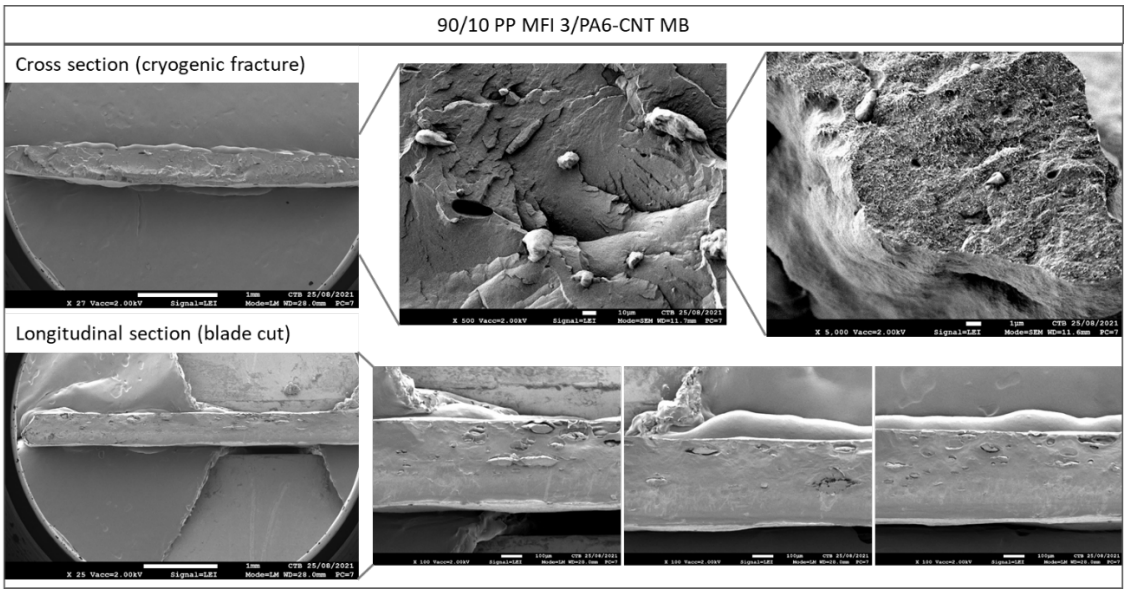

Fig. 3 SEM images of the transverse (top) and longitudinal (bottom) cross-section of a 90/10 PP/PA6-CNT MB filament.

A subsequent series of experiments was set up to investigate different routes to increase the partitioning/dispersion of the PA6-CNT phase in PP to improve filament quality and conductivity.

Influence of dilution & Compatibilisation

Compounding trials by partner Fraunhofer ICT showed that adding virgin PA6 during the compounding of PA6-CNT masterbatches with PP led to an increase in conductivity. Moreover, this was accompanied by a reduction in the roughness of the obtained strands. Triggered by these findings, a similar strategy was applied in monofilament extrusion of the dry blends of PP and the PA6-CNT masterbatch.

It was opted to keep the PP phase constant and compare a dry blend of 70% PP and 30% PA6-CNT masterbatch on the one hand and a dry blend of 70% PP and 30% diluted PA6-CNT masterbatch on the other. The masterbatch was diluted with 2/3 PA6 via compounding. In addition, a small amount of compatibiliser (≤1%) was also added to improve the interaction between the two phases without mixing the phases too well. Both strategies resulted in a visible improvement of the filament, in particular lower roughness and better distribution of the PA6-CNT phase (Fig. 4). After the addition of compatibiliser, the filaments could be provided more, leading to better tensile properties. The presence of a small amount of compatibiliser did not affect the resistivity.

Fig. 4 Pictures of 70/30 PP/PA6-CNT filaments, f.l.t.r. undiluted, diluted and with compatibiliser.

Compounding vs. dry blending

The compounding route was also further investigated. Partner Fraunhofer ICT compounded PP and PA6-CNT MB with PA6 in various ratios (ranging from 90/10 to 50/50 PP/PA6) in 1 or 2 steps. These compounds were processed at Centexbel via monofilament extrusion. The filaments were again extensively characterised, with emphasis on the morphology of the blend studied via SEM. Compounding combined with dilution of the PA6-CNT phase was indeed found to strongly enhance the distribution and dispersion of the latter (Fig. 5).

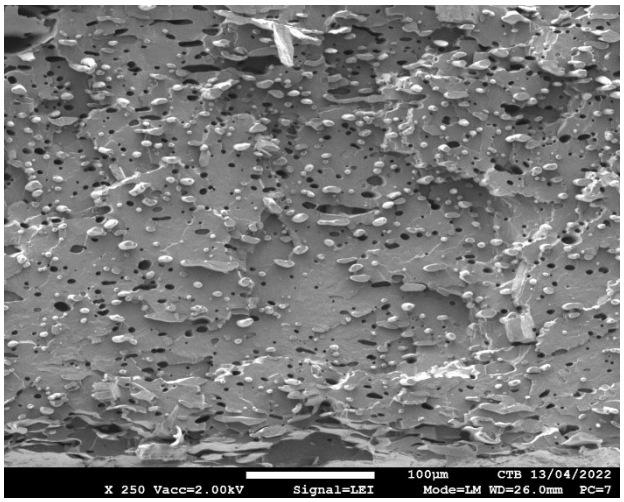

Fig. 5 SEM image of the cross-section of a 70/30 PP/PA6-CNT (diluted) filament the material of which was compounded in 2 steps.

Furthermore, compounding tests were also carried out at Centexbel to determine the percolation concentration of the CNT MB. The results are currently being analysed and will be discussed in detail at the next guidance group meeting.

Prolongment of the project

The TECMAT project was not spared COVID-related delays either. The 30/05/2023 final date was initially postponed to 31/08/2023, but the project was further extended to 30/11/2023 to allow for proper completion of ongoing and planned actions.

Save the date!

The next guidance group meeting will take place on 14 October 2022 (9:00-11:00) at VKC (Etienne Sabbelaan 49, 8500 Kortrijk). During the meeting, the results of the dilution and compounding tests will be presented. Moreover, new and planned actions will also be explained.