Morphology Characterization of Particle Filled Polymers by Tomographic Methods

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Abstract

Several polypropylene composites with varying amounts of inorganic particles of different sizes were investigated. In dependence of the particle sizes four different tomographic methods were used to describe the morphology 3-dimensionally. For particle sizes of several micrometers sub-µm X-ray tomography and light optical tomography are suitable whereas for smaller particle sizes scanning electron microscopy based methods, i.e. focused ion beam (FIB) tomography and *in situ* ultramicrotomy have to be used. The presentation will show the advantages and the limitations of every method. A comparison with two-dimensional morphology analysis will be done which shows the problems and errors of two-dimensional methods in the description of orientation and shape especially in the case of low particle content.

1 Introduction

During the last years the application of tomographic methods in the characterization of polymeric composites has been of increasing interest. There are two main reasons for that: the availability of appropriate methods and equipment and the increased informative value especially in the case of complex morphologies.

2 Experimental

Two different composite systems were investigated, a PP/talcum system containing 1, 10 and 20 wt% talcum and a PP/mica system containing 1, 3, 5 and 10 wt% mica.
X-ray tomography was carried out at a GE Phoenix X-ray Nanotom 180NF. Detector size was 2300 x 2300 pxl. The whole cross-section was scanned over a length of 7 mm, i.e. the measured volume was 10 x 4 x 7 mm^3, voxel size was 6 µm. Time for data collection was 140 min.
For the so-called light optical tomography the samples were embedded in araldite resin and than polished manually. The depth was checked by an embedded reference object of pyramidical shape, the alignment was done with the helt of hardness impressions. Note, that in the case of injection moulded parts containing plate-like fillers the polishing should be not done parallel to the main orientation plane if possible.
The FIB/SEM investigations were done at a FEI Quanta 3D 200 Dual Beam device under high vacuum at 10 kV. To prevent the sample it was coated with a thin Pt layer. No further coating was needed to observe the exposed surfaces. After collecting the pictures the post processing was done using software AMIRA.
Insitu ultramicrotomy was done using a prototype devise from Gatan integrated in a FEI Quanta 600 scanning electron microscope at room temperature. Slice thickness was 50 nm.

3 Results

An example of the results of X-ray tomography of PP + 10 % mica is shown in figure 1. The morphology of the whole cross-section is visible. Due to the relative large voxel size of 6 µm the error in the description of the smallest particles is not negligible. Nethertheless the shown pictures give a lot of information. It can be seen that the sample contains a high amount of very big particles. The 3-dimensional orientation of the particles can be seen and quantitatively described.

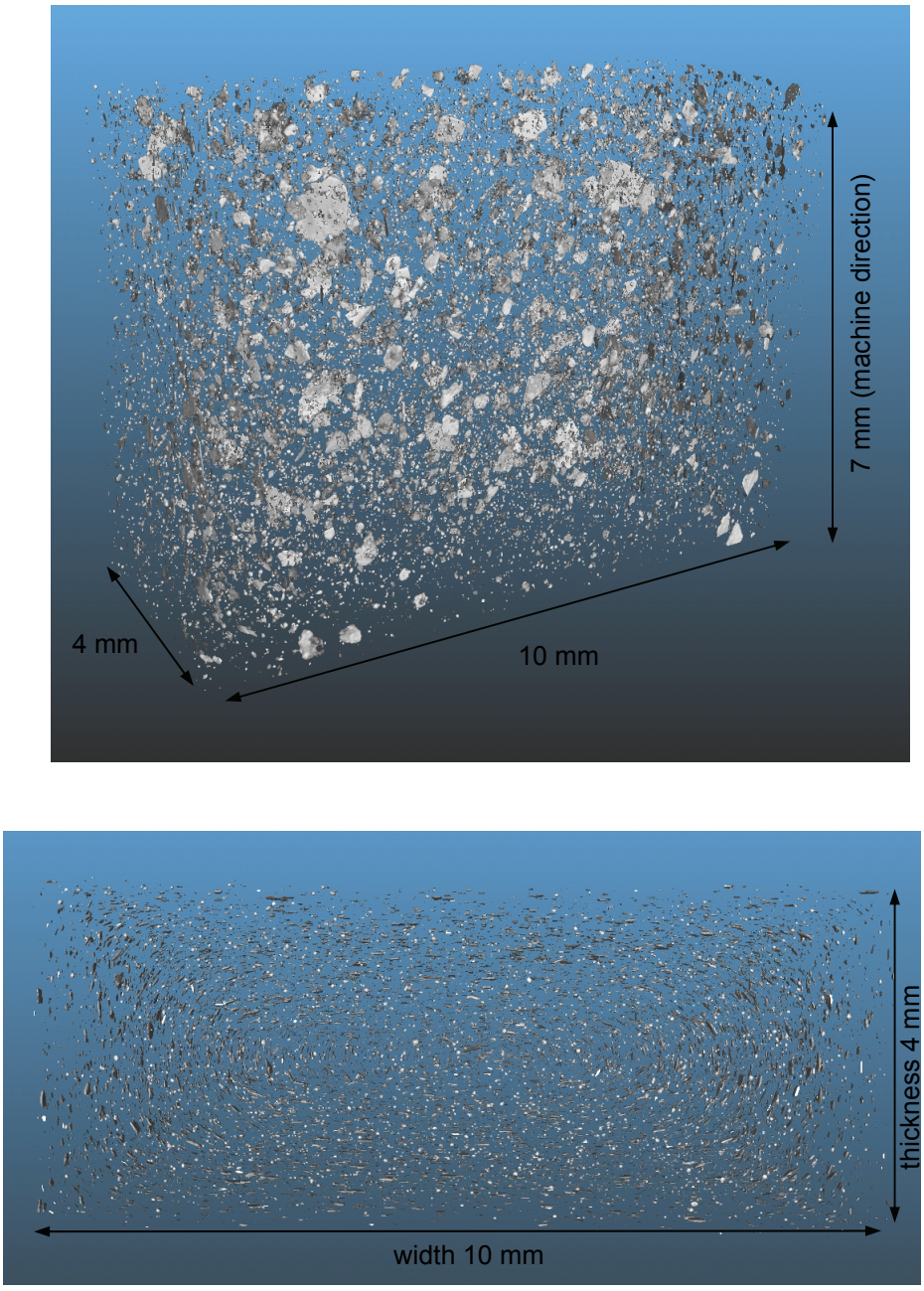

Figure 1: Mica particles in an injection moulded specimen

In principal the same results can be achieved by light optical tomography but to visualize large volumes as shown above too many layers are needed. Assuming a layer thickness of 3 µm this means more than 2000 polishing steps to observe the same volume as shown in figure 1.

At present the smallest voxel size in X-ray tomography is 50 nm reached at synchrotron beamlines with special optics. Besides the not as easy availability of experimental time at synchrotron facilities especially for routine investigations there will be also a problem if the particles have dimensions smaller than 100 nm in at least one direction as it is the case in our PP/talcum systems. The combination of FIB and SEM could be an alternative. Our results show that even at high vacuum and high acceleration voltages the sputtering of platinum to the top and lateral surfaces of the prepared block was satisfactory, i.e. the observed surface did not needed to be sputtered to prevent charging (Fig. 2).

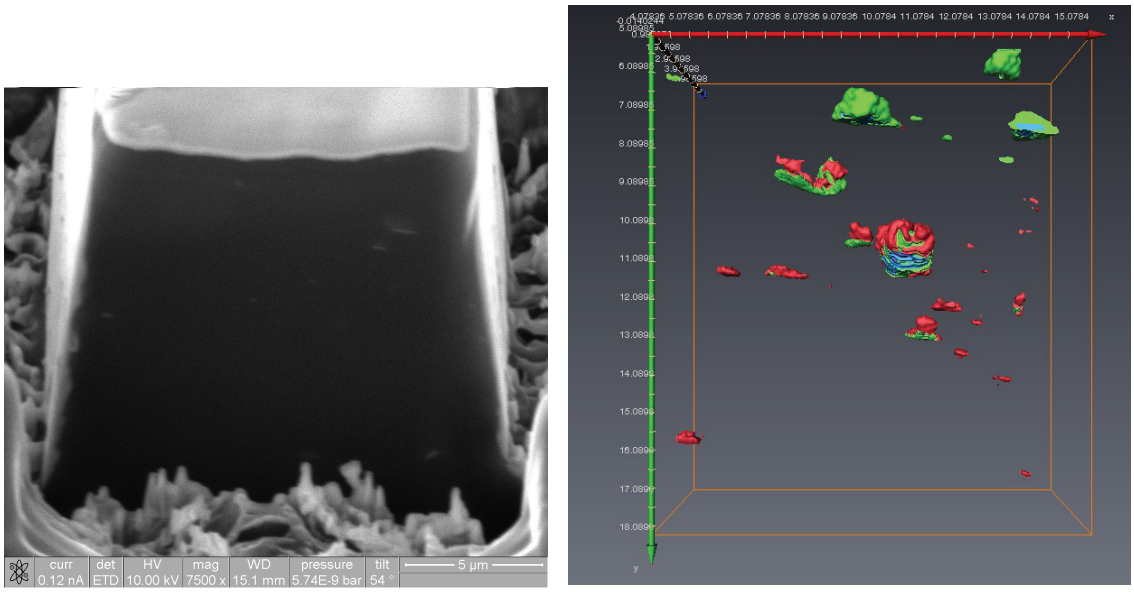

Figure 2: Prepared block for subsequent FIB-tomography (left) and arrangement of talcum particles determined by FIB-Tomography (right)

A typical result of the insitu microtomy is shown in Figure 4. Cutting of the PP/talcum samples at room temperature was not a problem due to the low hardness of talcum. So, there was no movement of the particles in the matrix. To check how influential is a possible smearing in the interphase region tests with stained samples will be done. The stiffness of the matrix will increased by staining which improves the quality of the cut surface. A disadvantage is the relatively small range of optimum stained region.

Figure 3: Arrangement of talcum particles determined by insitu microtomy in the SEM

4 Summary

The applicability of different tomographic methods for the particle visualization in polymer composites was shown. If the well established X-ray tomography reaches its limits destructive cutting methods can be used to describe the morphology. They have the advantage of a higher resolution and in the normal case easier post processing but the disadvantage of a smaller observed volume compared with X-ray methods.

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