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Advanced 2D and 3D Electron Microscopy Analysis of Clay/PP Nanocomposites

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Introduction

Clay/polypropylene (PP) nanocomposites with engineered properties are attractive in developing novel components of interest in a plurality of application fields [1]. An understanding of clay dispersion and intercalation in the polymeric matrix is of great importance to explain the improved macroscopic properties of nanocomposites. In this work, a clay/PP nanocomposite is studied by 2D bright field transmission electron microscopy (TEM) and 3D focussed ion beam – field emission gun scanning electron microscopy (FIB/FEG SEM).

Materials and Methods

A clay/polymer nanocomposite consisting of 3 wt% modified clay in a PP matrix was studied. Prior to microscopy analyses, SEM or TEM samples were cryo-microtomed to a flat surface or thin sections (70 nm), respectively. An FEI Titan T20 TEM microscope operating at 200 kV was used for 2D imaging. An FEI Helios focussed ion beam (FIB) equipped with field emission gun (FEG) and through lens detector (TLD) was used for high resolution 3D imaging of the material via slice-and-view technique [2]. Image analysis was performed using Matlab.

Results and Discussion

Figure 1 (a) shows a TEM micrograph of a clay/PP nanocomposite, where the clay particles are dark and the polymeric matrix is bright. The nanoparticles are rather well dispersed and distributed. Individual clay layers and agglomerates consisting of three or more stacked layers are observed. Figure 1 (b) shows a TEM micrograph of stacked nanoclay layers. The layers are uniformly spaced with an observed average interlayer distance of 2 nm. This indicates that intercalation of the clay within the polymer is occurring since the average interlayer distance of a neat clay is ca 1.2 nm [3].

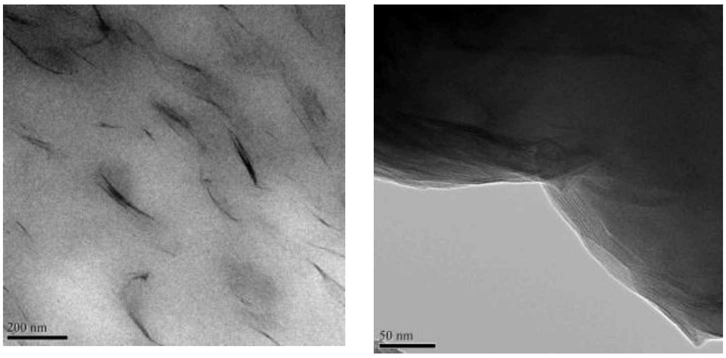
Figure 2 (a) shows a FIB/FEG SEM micrograph of the first cut volume of the polymer nanocomposite. From that volume, thin (20 nm) slices were cut and imaged with the TLD, as shown in Figure 2 (b). The clay nanoparticles are bright and the polymeric matrix is dark. As illustrated in Table 1, the observed individual layers, stacked layers, and larger agglomerates have sizes ranging from tens to hundreds of nanometres and are rather uniformly oriented, distributed, and spaced from each other. Image analysis from these micrographs is very useful since a rather large field view is analysed as compared to TEM micrographs, which are traditionally used to study such properties.

Figure 3 (a) shows a high resolution SEM micrograph obtained with the TLD detector. High magnification and resolution comparable with that of TEM (see Figure 3 (b)) are achieved.

Conclusions

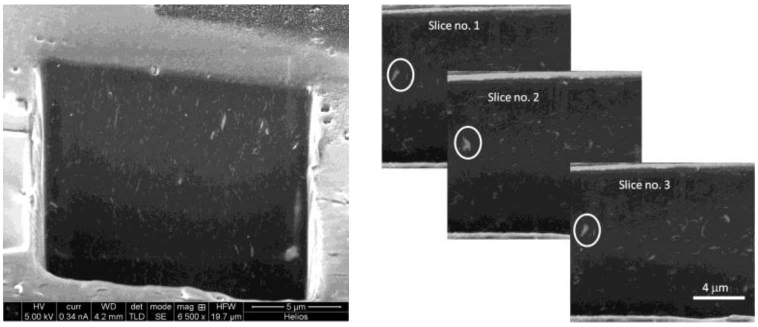
To the best of our knowledge, this is the first work showing the great advantages of FIB/FEG SEM imaging with TLD (namely larger field of view and high resolution) as compared to TEM in the study of polymer nanocomposites. Both microscopy techniques are powerful tools to study these materials and provide a clear, quantitative measurement of the morphology, size distributions, and dispersion of the clay nanoparticles.

1. Tjong, S.C. *Mater. Sci. Eng. R* 53 (2006) 73
2. Benawra J. et al. *J. Microsc.* 234 (2009) 89
3. Ogawa, M. et al, *Langmuir* 9 (1993) 1529



(a) **(b)**

Figure 1. TEM micrographs with different magnifications of the clay/PP nanocomposite studied in this work.



(a) **(b)**

Figure 2. SEM micrographs of the first cut volume (a) and a series of three consecutive slices (b) of the clay/PP nanocomposite studied in this work.

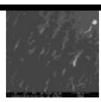
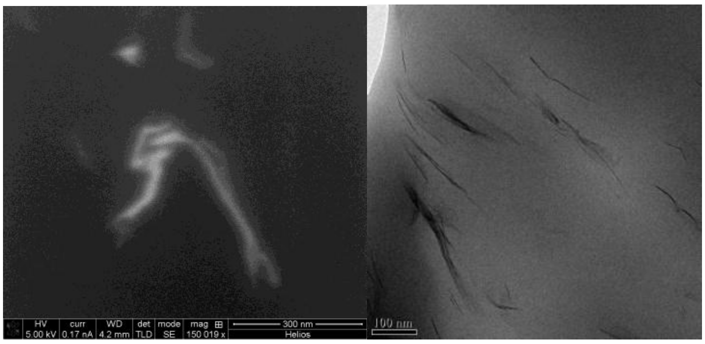
Polymer nanocomposites (no. particles: 101) 				
Area [nm^2]	Av. 4071	min. 19	Max. 23378	STD 4456
Direction Angle [°]	71	31	138	21
Shortest distance Distance between center of particles [nm]	150	22	308	66

Table 1. Geometrical properties of the clay/PP nanocomposite.



(a) **(b)**

Figure 3. High resolution SEM (a) and TEM (b) micrographs recorded at comparable magnifications of the clay/PP nanocomposite studied in this work.