doi:10.1017/S143192761006085X
Microsc. Microanal. 16 (Suppl 2), 2010

Study on Nanoclay Dispersion in a Polymer Matrix by High-Resolution Electron Microscopy Combined with Electron Tomography

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In this study, we choose poly[(butylene succinate)-co-adipate] (PBSA) as our model polymer. PBSA is a random copolymer of poly(butylene succinate) (PBS); which shows a variety of interesting physical properties, including biodegradability. The nanocomposite of PBSA with methyl tallow bis(2-hydroxylethyl) quaternary ammonium modified montmorillonite (MMT) (commercially known as C30B) were prepared by melt-mixing in a batch mixer. The nature of the silicate layers' dispersion in the PBSA/C30B (4 wt.%) composite was characterized by X-ray diffraction, high-annular-angle-dark-field scanning transmission electron microscopy (HAADF-STEM), and finally, by 3-D electron tomography.

Fig. 1 shows the HAADF-STEM images of the nanocomposite sample; in which, white entities (lines/sheets) are the silicate layers and the black background is the PBSA matrix. From STEM images, we see that most of the silicate layers are homogeneously dispersed in the PBSA matrix; however, the stacking of some silicate layers is still observable and this is more discernible at high magnification STEM image. Another interesting observation is that some of the silicate layers are bended. It has already been reported that individual MMT sheets are not rigid and because of the very high aspect-ratio they can be highly flexible and show localized bending, as observed in high magnification STEM images [1].

To have clearer picture on the degree of dispersion of silicate layers in the PBSA matrix, electron tomography technique was also used. Using the automatic observation system combined with a Fourier reconstruction method based on the electron tomography technique, 3-D images of the PBSA nanocomposite containing 4 wt.-% of C30B were reconstructed by using a simultaneous iterative reconstruction technique (SIRT). Fig. 2 shows the TEM tilt series and z-slice through the corresponding reconstruction of PBSA nanocomposite containing 4 wt.-% of C30B. From the 3-D images of nanocomposite from various directions, many aggregates of silicate layers (black entities) are clearly seen. This observation again supports our previous experimental observations that it is very difficult to get true exfoliation of silicate layers in the polymer matrix [2].

In conclusion, this study shows the homogeneous dispersion of silicate layers in the PBSA matrix; however, the true exfoliation of silicate layers in the polymer matrix is quite difficult to achieve, although there are strong favourable interactions between the polymer matrix and the clay surface [3].

[1] L. F. Drummy, H. Koerner, K. Farmer, A. Tan, B. L. Farmer, R. A. Vaia, *J. Phys. Chem. B* 109 (2005), 17868.
[2] S. Sinha Ray, *Macromol. Mater. Eng.* 294 (2009) 281.
[3] This work was financially supported by the CSIR and the DST, SA.

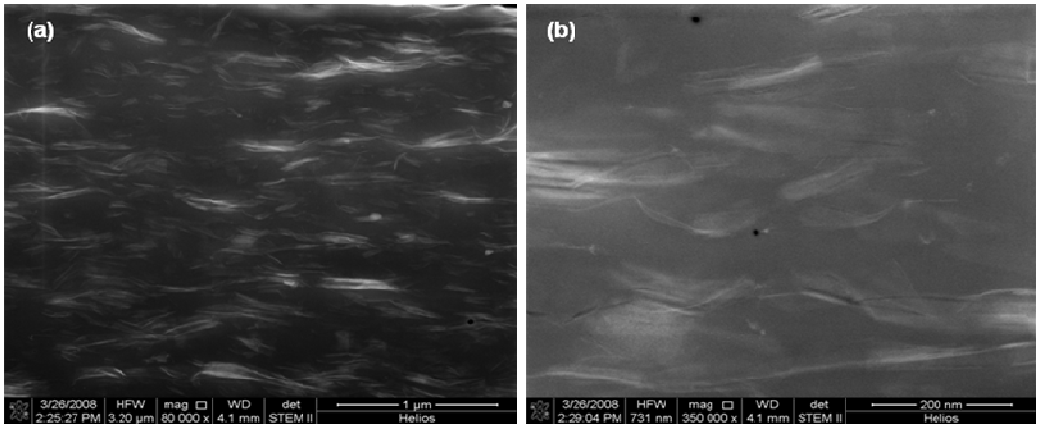

FIG 1. High-annular-angle-dark-field scanning transmission electron microscopy (HAADF-STEM) images of PBSA/C30B nanocomposite (containing 4 wt.-% C30B) at two different magnifications: (a) 80k and (b) 350k.

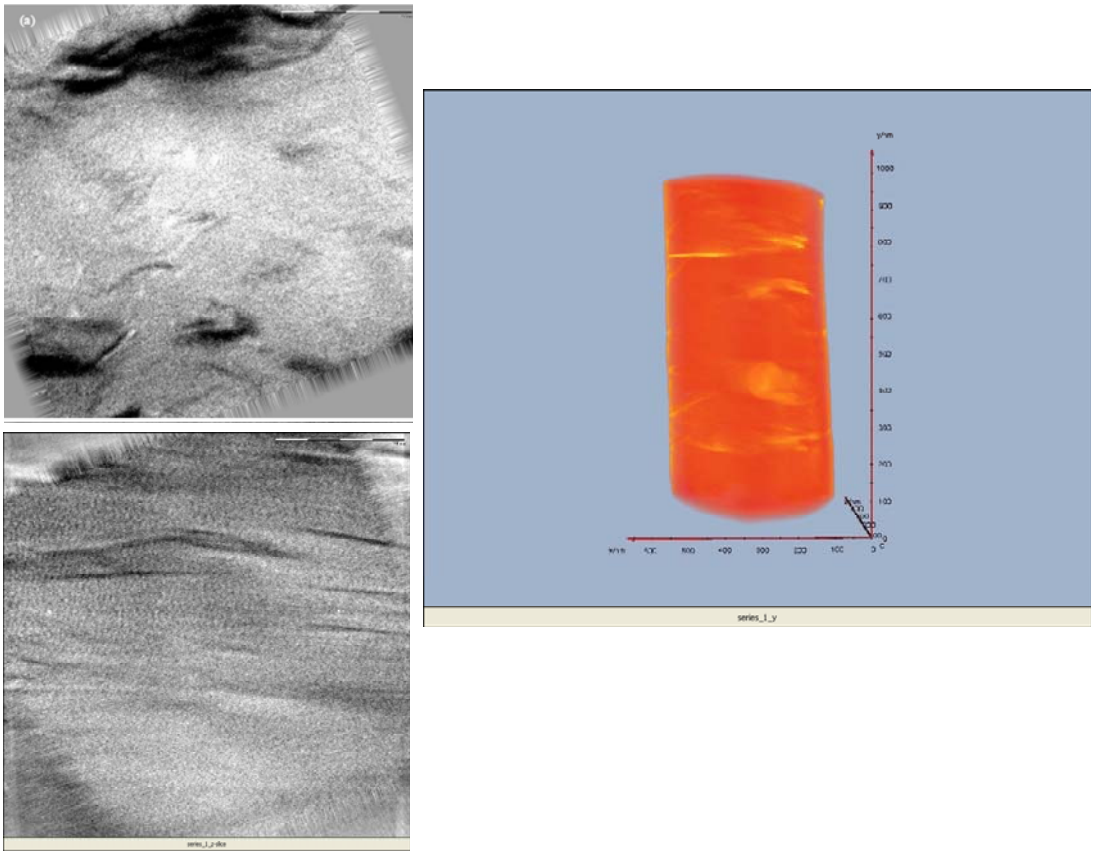

FIG. 2.The bright field TEM tilt series and z-slice through the corresponding reconstruction of PBSA nanocomposite containing 4 wt.-% of C30B. The black entities are the dispersed silicate layers in the PBSA matrix.

https://doi.org/10.1017/S143192761006085X Published online by Cambridge University Press