TEM SAMPLE PREPARATION OF POLYMER BASED NANOCOMPOSITES USING FOCUSED ION BEAM TECHNIQUE

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Recently, several studies have been conducted to investigate the behavior of polymer based composites reinforced with clay particles, which can remarkably improve the properties of the polymers.[1] Studies using transmission electron microscopy (TEM) are necessary to understand the role of clay minerals/particles in the reinforcing effect in the polymer based materials. TEM sample preparation of polymer/clay nanocomposites using conventional techniques has been difficult and tedious.[2] Nevertheless, the focused ion beam (FIB) technique[3,4] for preparing metal and ceramic samples provides another method for preparing polymer nanocomposite samples. This paper presents a new approach for preparing TEM specimens of the polymer nanocomposites using the FIB technique.

Two types of epoxy (Dow Chemical Company, DER 331) nanocomposite samples were investigated: one containing 7.5 wt.% organomontmorillonite clay (Southern Clay Products Inc., Cloisite 30B) and the other (carbon fiber reinforced plastics; CFRP) containing carbon fibers (Hexel Fibers, AS4) in addition to 5 wt. % clay. Details of preparing the epoxy based clay nanocomposites will be published elsewhere. Procedures for preparing TEM thin sections using FIB were based on techniques developed by Ramirez de Arellano et al.[4] A small piece was cut from each sample, and then carefully ground and polished to a thickness about 50-100 ?m. The polished samples were attached on a special Mo grid for FIB milling. The samples were then milled by Ga ions (accelerated at 30 kV) with different probe sizes using a Hitachi 2000A FIB system. To prevent the thin section from bending, the milling areas were gradually reduced (from about 20 ?m to 10 ?m in width). The milling process was terminated when electron transparent areas (~200nm in thickness) were obtained (arrows showed in Fig. 1). The TEM observation was carried out using a Hitachi H8100 TEM.

A low magnification TEM micrograph (Fig. 2) shows a general view of the dispersed clay particles in the epoxy resin. As expected, the concentration of the clay particles was low. Figure 3 presents a close-up view of the clay fabric in the epoxy resin. Due to the nature of the clay minerals, layered aluminum silicate with platy morphology, the clay particles viewed along c-axis tended to be more transparent (white area pointed by an arrow). Particle size of clays ranged from 0.6 to 0.8 ?m on the a-b plane. The view of perpendicular to the c-axis (i.e., along a-b plane) shows typical elongated fiber-like feature. A high magnification TEM micrograph (Fig. 4) clearly reveals the basal spacing of clay particles in the epoxy resin. The d-spacing of the montmorillonite clays was 1.85 nm before sonicating in acetone. The measured large d-spacing (3.4-3.8 nm) indicated that large organic molecules (polymer network of the epoxy resin) have been well-intercalated in between clay basal layers. Figure 5 shows a TEM micrograph of CFRP. The clay particles were scarcely dispersed in the epoxy matrix between the carbon fibers. A higher magnification TEM view depicts a few clay particles aligned along the carbon fiber/epoxy interface as shown in Fig. 6. Although only at a low concentration, these clay particles were found to play an important role in improving the transverse mechanical properties (strength and ultimate strain) of CFRP. In summary, FIB technique is a valuable means for preparing the composite specimens in a relatively short time that would be difficult to prepare through the conventional techniques. Research on correlating the clay fabric and the composite material properties is in progress.

References:

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5. This research made use of MRC Facilities supported by the NSF (DMR-9632742) at NU.

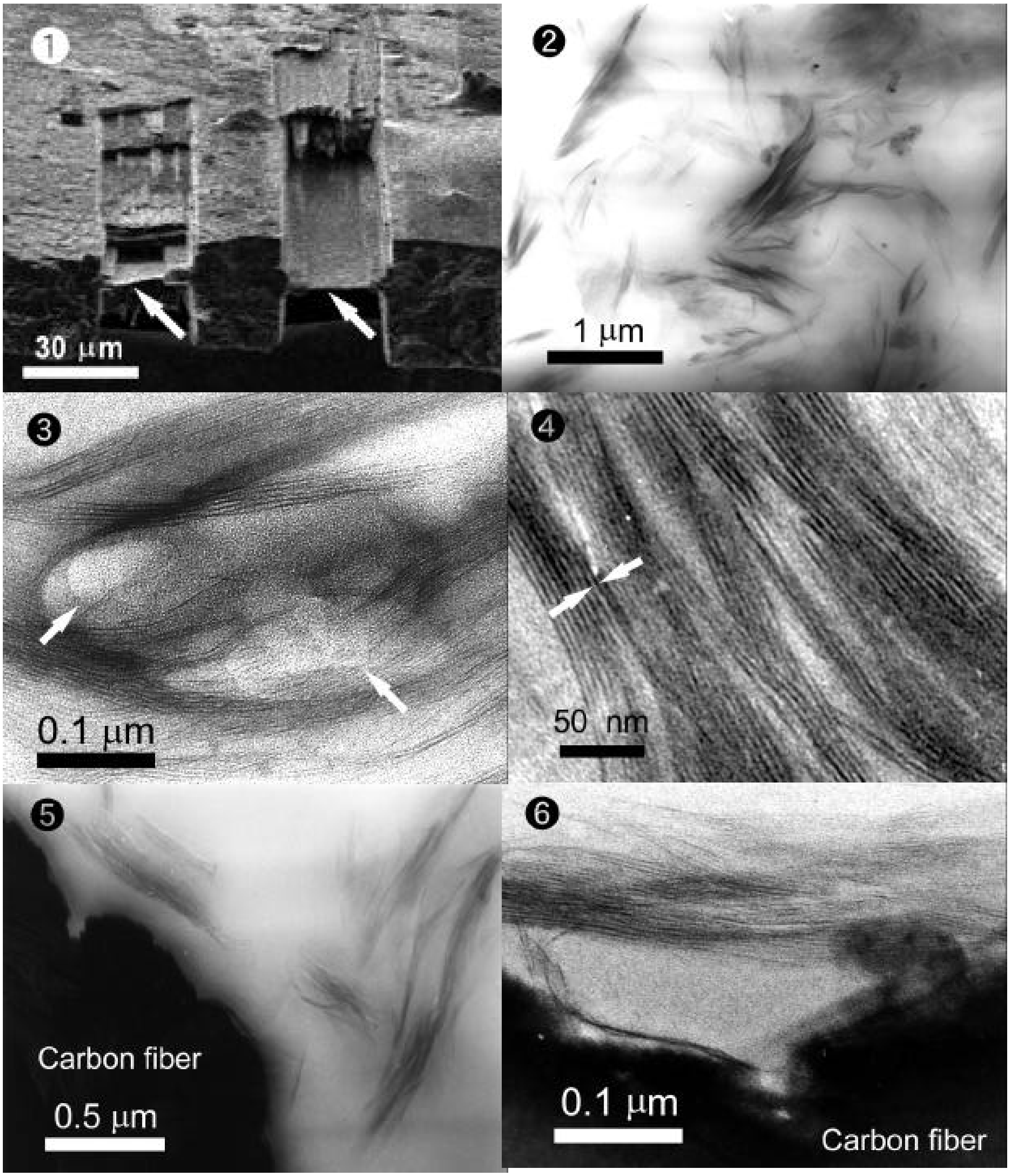

Fig. 1. SEM micrograph showing FIB thinned area.(arrows).
Fig. 2. TEM micrograph showing clay particles in epoxy resin
Fig. 3. TEM micrograph showing clay particles in two different orientations.
Fig. 4. TEM micrograph revealing well-intercalated/expanded clay layers.
Fig. 5. TEM micrograph showing dispersed clay particles in CFRP.
Fig. 6. TEM micrograph showing clay particles aligning along carbon fiber/epoxy interface.