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Low Voltage Electron Microscopy of Organic Materials

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We have investigated the utility of a DeLong table-top sized (~5 kV) Low Voltage Electron Microscope (LVEM) for examining polymer, organic molecular, and biomaterials [1]. This instrument can operate as a transmission electron microscope (TEM), scanning electron microscope (SEM) (with either secondary or backscattered detectors), or as a scanning transmission electron microscope (STEM). It can also perform selected area electron diffraction (SAED) experiments. Because of the small size of the chamber, it has rapid sample exchange times (~5 minutes or less).

Samples for LVEM are typically prepared by suspending on a thin, amorphous carbon support film over a conventional copper TEM grid. The 5 kV beam has been shown to penetrate up to 50 nm of hydrocarbon materials. The images have high contrast, due to the higher effective cross-section for scattering at these lower voltages. The resolution is somewhat reduced from a conventional TEM (~2 nm), but is still more than sufficient to resolve many interesting nanostructures. Of particular value for polymer and biomaterial microscopy is the potential to reduce or even eliminate the need for staining with heavy metals such as osmium, ruthenium, tungsten, lead, or uranium. These stains can cause imaging artifacts and may be hazardous to use in the laboratory.

Figure 1 shows an example of a STEM image of a polycrystalline pentacene thin film. Pentacene is an organic semiconductor of considerable interest for making flexible electronic devices such as thin-film transistors [2]. The properties of these materials are limited by carrier transport across grain boundaries. The LVEM clearly resolves the grain boundary structure in these thin films. Studies of grain boundary structure as a function of processing condition make it possible to optimize the performance of devices constructed from these polycrystalline substances.

Figure 2 shows a thin crystalline platelet of a montmorillonite clay that is under investigation as a reinforcing agent in various polymer matrices. These nanocomposite materials show dramatic increases in properties at small additive content because of the dispersion of these sheets into individual layers. The LVEM shows high contrast when imaging single clay layers. These single layers are extremely difficult to resolve by conventional high voltage TEM due to the limited contrast. The inset shows a SAED pattern obtained from a ~150 nm region inside one particular platelet confirming its single crystal nature.

Figure 3 shows a TEM image as well as a backscattered SEM image of a droplet of *Bombyx Mori* silk that was deposited onto an amorphous carbon film, supported on a copper TEM grid. LVEM studies have revealed consistent, systematic changes in the polycrystalline structure of these films under different processing conditions.

References

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[2] L. F. Drummy et al., *J. Phys. Chem. B,* (2006), in press.
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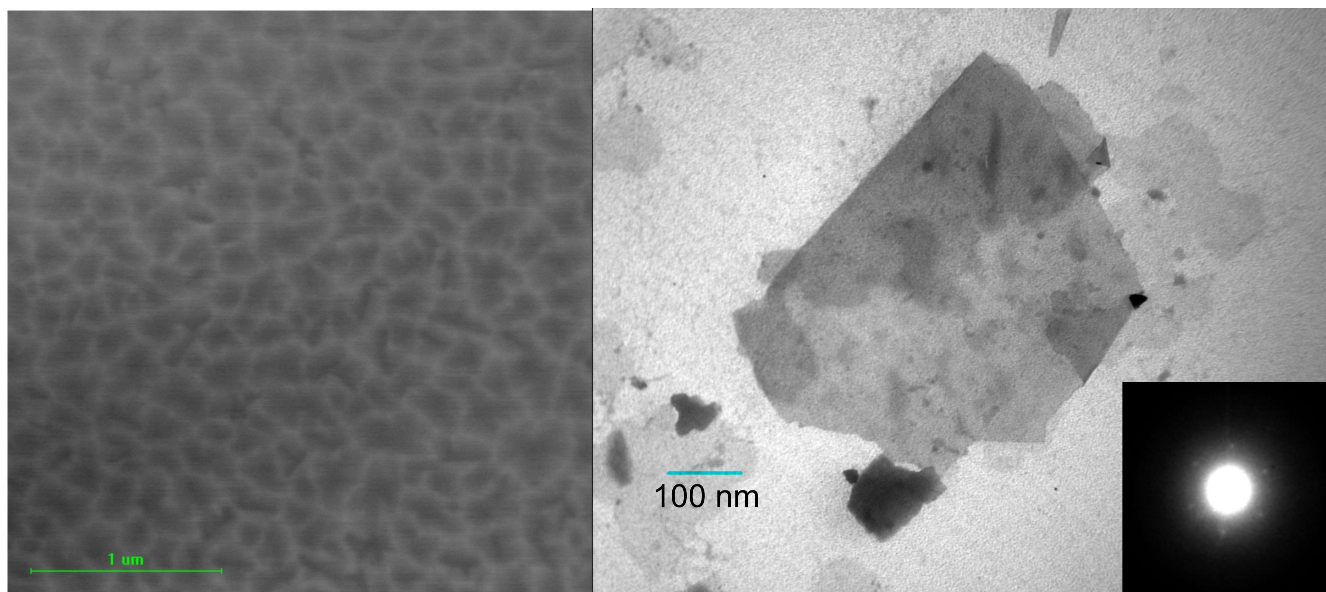

FIG. 1 (left). Low voltage STEM image of a polycrystalline film of pentacene showing the individual crystalline domains (dark) and grain boundaries between them (light).
FIG. 2 (right). Low voltage, bright field TEM image of individual platelets of a montmorillonite clay (~1 nm thick). The selected area electron diffraction pattern from the largest platelet using a probe size of ~150 nm is inset.

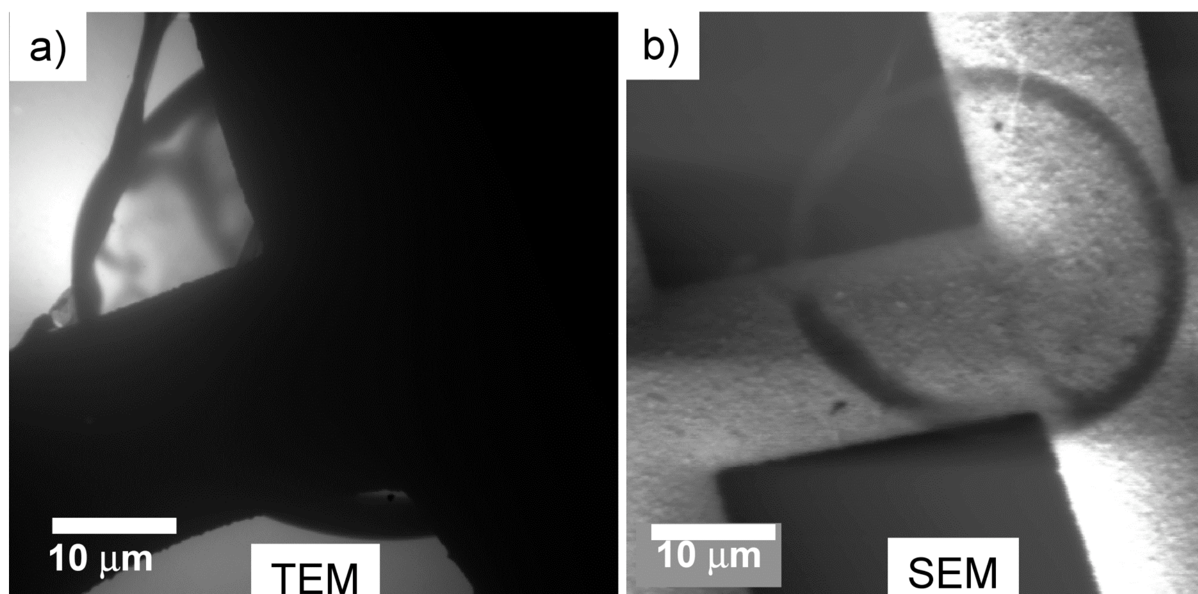

FIG. 3a) LVEM image of a *Bombyx mori* silk thin film droplet atomized from dilute solution in hexafluouroisopropanol onto an amorphous carbon-coated copper grid taken in TEM mode. b) The same droplet imaged in backscattered SEM mode.

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