High resolution polymer imaging using scanning electron microscopy with back scattered electron detector

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Semi-crystalline polymers based on olefin chemistry are in particular interesting due to their low cost of manufacture and the ability to tailor properties by controlling polymer morphology. High resolution polymer morphology was traditionally visualized by transmission electron microscopy (TEM), where lamellar structure was revealed by heavymetal selective staining to enhance contrast [1]. Despite that TEM delivers excellent results,m it requires highly skilled and laborious sample preparation of ultrathin sections.

Recent developments in new generation of field emission gun scanning electron microscopes (SEM), that incorporate a variety of improvements including high current, low beam energy electron optics and new detector systems offer new opportunities for polymer morphology analysis that enables SEM to become an alternative characterization method to TEM. The approach is based on imaging of stained polished block surfaces at low accelerating voltages by means of backscattered electron (BSE) detector. BSEs consist of high-energy electrons, originating from the electron beam, that are reflected or back-scattered out of the specimen interaction volume. The signal comes from a depth of up to half the penetration depth that is a function of the beam energy (accelerating voltage) and sample composition. The efficiency of production of BSEs is proportional to the sample material's mean atomic number, the higher the average atomic number the more the primary electrons are backscattered providing compositional information about the sample. Since the BSE contrast is inverted as compared to TEM contrast, often negatives of the BSE images are collected to be consistent with the traditional TEM gray scale. The use of low beam energy results in small beam-sample interaction volume that provides good spatial resolution of BSE signal allowing visualization of detailed information on polymer crystalline structure. Under optimum conditions a spatial resolution of 1 nm can be attained. With these advances the SEM can now be used to image both gross morphology at low magnifications, and high resolution imaging that was until recently only possible in the TEM. Moreover, the main advantage of the SEM-based technique is significantly less demanding and easier sample preparation than thin sections for TEM. For example for samples that contain high filler content, particle pull out and obtaining good sections were always a challenge, whereas block surface preparation is not problematic at all.

In the presentation sample preparation, staining contrast formation, and SEM-BSE imaging will be discussed. Several examples of high resolution BSE SEM images of olefin based polymers will be presented, including polypropylene impact copolymer (Figure 1), high density polyethylene (Figure 2), ethylene octene, and linear low density polyethylene. The demonstrated capability of BSE imaging of stained, block faced samples opens new possibilities for automation and automated image acquisition, which will be shown in a separate presentation.

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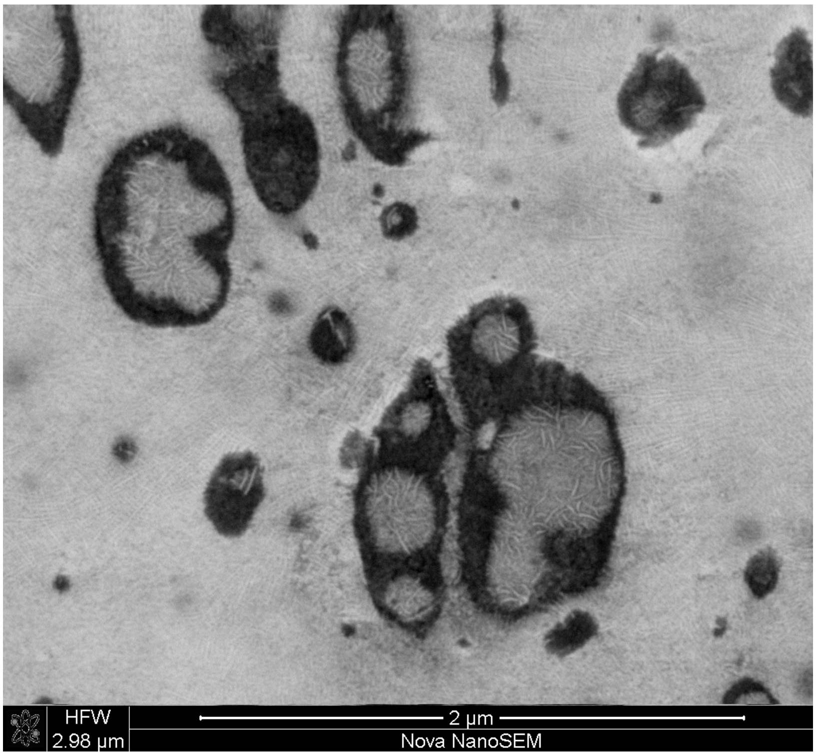

Figure 1. High magnification SEM-BSE images of impact polypropylene copolymer. Horizontal field of view of 2.98 µm.

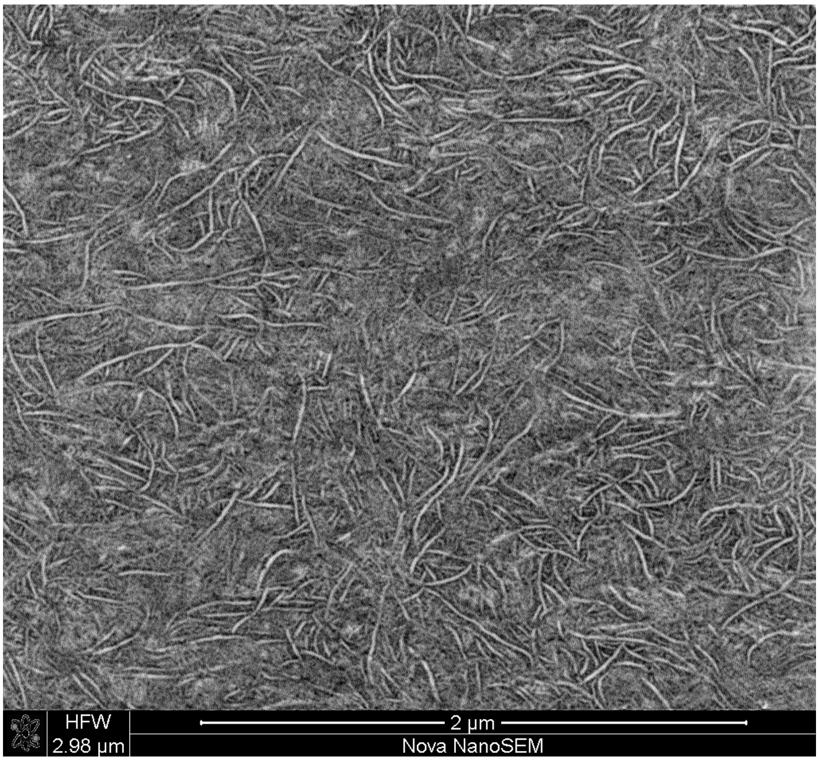

Figure 2. High magnification SEM-BSE images of high density polyethylene. Horizontal field of view of 2.98 µm.

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