Higher throughput analysis of polymer morphologies by scanning electron microscopy

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Among the microscopy techniques, transmission electron microscopy (TEM) has been traditionally used as the main established approach to visualize polymer morphology [1]. TEM image contrast is due to electron scattering, and polymers have low atomic number and scatter electrons weakly. Therefore, appropriate contrast enhancing heavy metal stains need to be used to allow direct imaging of the morphology as well as of the lamellar structure of semi-crystalline polymers. Despite the fact that TEM imaging has a proven record of delivering superior polymer morphology information, this approach faces several challenges and difficulties: the laborious nature of ultra-thin section preparation, high capital investment, and no obvious routes to high-throughput, automated imaging. Manual sample handling and manual microscopic examination of hundreds of polymeric samples is routinely performed and standard approach in both academia and chemical industry. Particularly industry would benefit from fully automated microscopic imaging in that it would not only alleviate the tremendous workload from professional and result in greater efficiency, but more importantly, accelerate materials research and set user independent work process standards across geographies and locations.

This paper focuses on the approach to cover the major morphology imaging needs on the basis of new generation of high resolution scanning electron microscopes (SEM) that do not rely on the need for ultra-thin section preparation. Key of this approach is that it allows automated image acquisition, therefore resulting in substantial productivity increases and high speed characterization options. It is based on the use of backscattered electron (BSE) imaging of stained and polished block faces at low accelerating voltages, which requiring less demanding sample preparation than producing ultra-thin sections. Even though the concept of using BSE imaging for stained and microtomed surfaces has been explored earlier [2, 3] it became only recently possible to study polymer morphology in detail at the required low voltage conditions due to the improvement of BSE detector technologies and performance. The demonstrated capability of BSE imaging of stained, block faced samples opens new possibilities for automation and automated image acquisition. In the proposed concept, a customized multi-sample holder (Figure 1A) can accommodate up to 20 stained, block faced samples. A custom developed software user interface allows the user to define the number of samples, number of locations per sample, magnifications and number of images per sample. Full flexibility allows to pre-set these parameters for several batches or change any of them on the desired needs. This flexibility is an important feature reflecting the complexity of different samples and imaging needs in polymer research. Once these conditions as well as a number of basic microscope parameters have been selected, the software will locate and advance through all the samples of interest and acquire the defined number of images in a fully automated fashion using a set of auto-focus and auto-contrast routines. It has to be noted that it was critically important to optimize and to control the auto-focus and auto-contrast parameters. Several commonly used auto-focus algorithms [4] were explored for this purpose

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and optimized for challenging polymer samples to be imaged in an automated fashion at high magnifications and low accelerating voltages using SEM-BSE imaging. Figure 1B-1D shows images of various magnifications that were acquired in a fully automated fashion. The program has yielded almost 100 % acceptable images, and the images shown in Figure 5 are representative for one of four sets of BSE images on one of 20 samples imaged (in this case per sample four locations were examined, per location images at three magnifications were acquired, resulting in 3 x 4 x 20 = 240 images in total). As can be seen, even the highly demanding imaging at high magnification to visualize lamellar structures was successfully accomplished in an automated fashion.

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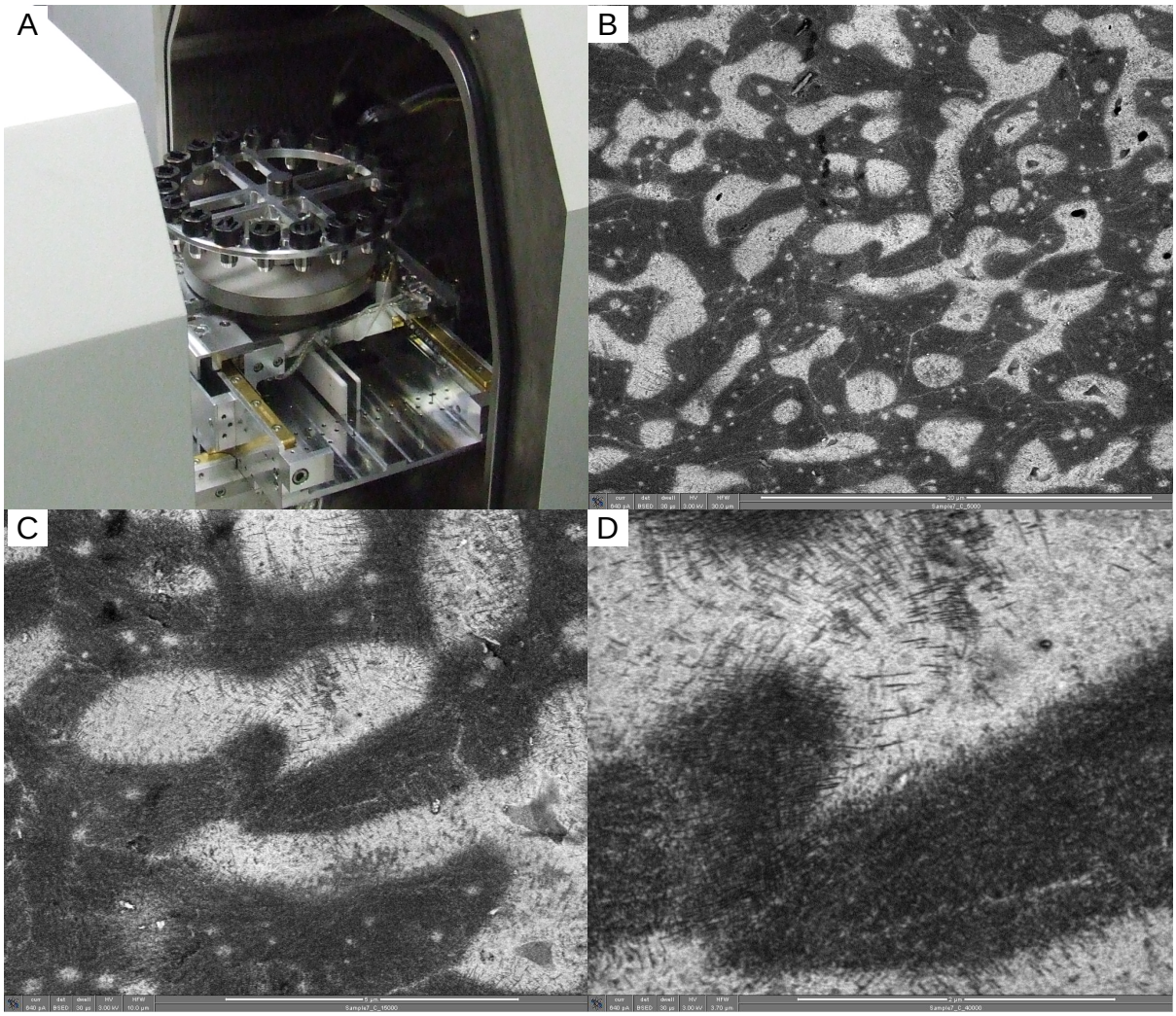

Figure 1. Multi stage holder with 20 stained and microtomed polymer samples (A). One of four sets of BSE images on one of 20 polymer samples imaged with unattended automation software. 31 µm field of view. (B) 10 µm. (C), and 3.7 µm (D).