doi:10.1017/S1431927617009746
Microsc. Microanal. 23 (Suppl 1), 2017
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Polymer imaging in SEM – charge, damage and coating free.

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Typical information provided by the SEM is sample morphology and composition. Acquiring an image is an easy task if samples are conductive but it becomes difficult in case of soft matter insulators, such as polymers. Two main challenges are sample charging and radiation damage. Traditional approach is to coat the polymer by thin conductive layer. However, coating has severe disadvantages, such as masking tiny surface details or impossibility of using the sample for further analysis. This article summarizes the best practices for imaging and analysis of polymers in the SEM without coating to deliver information about their morphology and composition.

Low primary electron beam energy and dose (low beam current and fast beam scanning) are crucial for charge- and damage-free imaging of sensitive samples. Both are enabled by field emission SEM providing high resolution at beam energies below 1 keV [1]. Moreover, advanced scanning strategies, such as scan interlacing and drift corrected frame integration [2] reduces sample charging to minimum not noticeable in the secondary electron image. In order to minimize the radiation damage, the beam energy and the dose have to be adjusted even more carefully. The most beam sensitive specimens require beam energy below 500 eV and dose as low as units of $pC/\mu m^2$ or even less. Capabilities of the low energy and dose imaging of polymer morphology and composition are demonstrated by two examples.

The first example (Figure 1) shows a highly beam-sensitive polymer specimen, polylactic acid (PLA) – biodegradable thermoplastic aliphatic polyester, whose surface was treated by plasma [3]. While beam energy of 1 keV and dose of 8.8 $pC/\mu m^2$ are sufficient for charge mitigation, the energy has to be decreased down to 300 eV to reduce the radiation damage. Boundary between the irradiated and virgin area is clearly visible while detailed surface structure is melted away in the 1 keV image (bottom right part of Figure 1a). These effects don't occur at beam energy of 300 eV and the irradiated area isn't noticeable (bottom right part of Figure 1b).

The second example (Figure 2) is a polymer blend with very similar components: high-density polyethylene (HDPE) and cycloolefin copolymer (COC), which is a copolymer of polyethylene and norbornene [4]. While the examples in Figure 1 shows surface morphology delivered by secondary electrons (SE), in the case of HDPE/COC blends the sample composition is of the particular interest and backscattered electrons (BSE) must be used as the imaging signal. Design of the BSE detector has to reflect requirements for high signal to noise ratio at low beam energy, dose and fast scanning speeds. Naturally, good compositional contrast is fundamental. All these requirements are fulfilled by sensitive T1 in-lens BSE detector collecting large solid angle and enabling energy filtering to strengthen the contrast or filter the charge [5]. Ability of the T1 BSE detector in polymer research is demonstrated by resolving the chemically almost identical phases in the HDPE/COC blends in Figure 2a. The results were acquired at beam energy of 500 eV and dose of 8.8 $pC/\mu m^2$. No sample coating or other traditional preparation, such as selective COC etching was applied prior imaging. Complementary morphological image was taken simultaneously to minimize the radiation damage (Figure 2b).

https://doi.org/10.1017/S1431927617009746 Published online by Cambridge University Press

In conclusion, charge and damage free imaging of polymers requires SEM that provides high resolution imaging at beam energies below 1 keV, electron dose of units of pC/μm^2 together with advanced scanning strategies and sensitive SE and BSE detectors with high yield.

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[6] The authors acknowledge funding from the Technology Agency of the Czech Republic, TE01020118

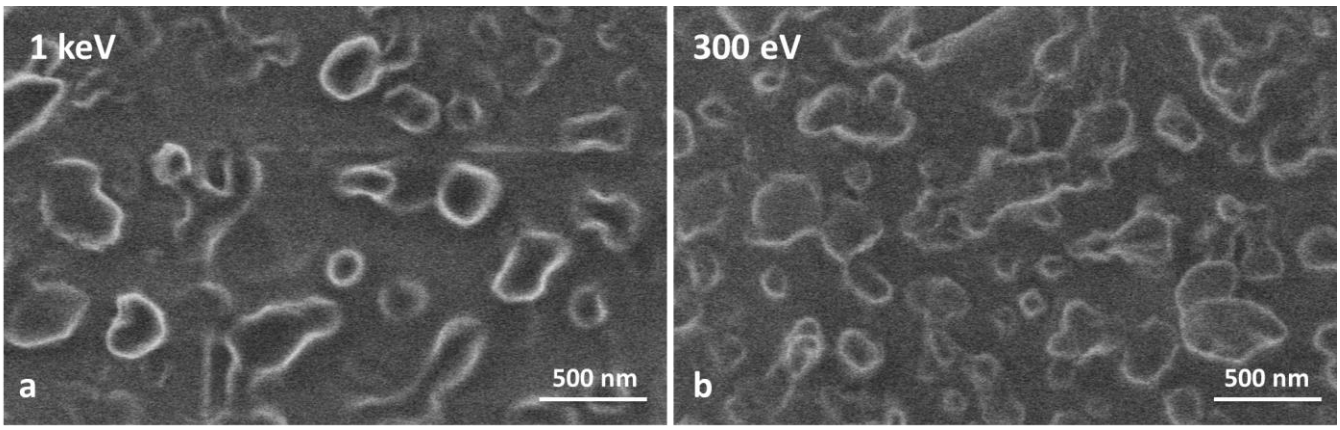

Figure 1. Comparison of the level of the radiation damage of the plasma treated polylactic acid (PLA) imaged at beam energy of 1 keV (a) and 300 eV (b).

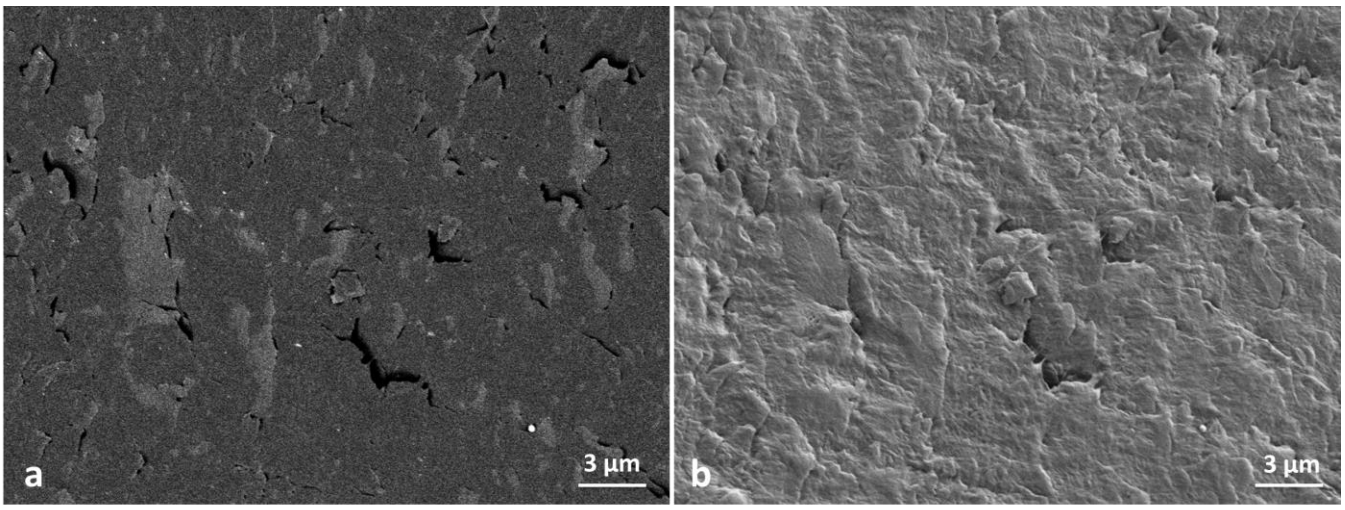

Figure 2. High-density polyethylene and cycloolefin phases in the HDPE/COC blends resolved by BSE compositional image obtained at beam energy of 500 eV (a) and simultaneously acquired morphological information (b).

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