

Applications of Low and Ultra-low Energy Scanning Electron Microscopy

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Presented are recent applications of the Scanning Low Energy Electron Microscope (SLEEM) [1]. The device employs the electron signal reflected from the specimen as well as that transmitted through the specimen (Scanning Transmission Electron Microscopy – STEM) for observation throughout the full energy scale down to units of electron volts. We can observe a strong transmitted signal below the primary beam energies below 100 eV, where the inelastic mean free path increases again for the majority of the material.

Figure 1 shows SLEEM images of a polished surface of dual-phase steel obtained at ultra-low landing energies of the primary beam. Organic impurity situated on the surface becomes visible only at units of eV. Thus, the imaging at units of eV in Backscattered Electrons (BSE) is an extraordinarily useful tool for the inspection of organic contamination on steel surfaces.

The material contrast and surface sensitivity increase thanks to the remarkable decreases of the interaction volume at low energies, see Figure 2. The interaction volume decreases from 1 300 nm at 10 keV of the primary beam energy to 15 nm at 0.6 keV, so details of the size below 10 nm can be visible. Monte Carlo simulations in software Casino were used for this calculation [2].

Low energy electrons are very useful for the study of modern 2D materials. Commercial graphene from the firm Ted Pella, prepared by Chemical Vapor Deposition (CVD), was used to understand the contrast formation in the reflected (BSE) as well as the transmitted signal. The specimen was tested in the ultrahigh vacuum SLEEM [1]. Furthermore, was it heated up to 100^o C and then cleaned in situ by low energy electron (40 eV) for 10 hours. The cleaning procedure was described by [3]. Figure 3 shows the series of micrographs in the BSE contrast peaking at an energy of around 10 eV, which achieves an optimum balance between the penetration and escape depths of the signal species. On the other hand, the thickness sensitivity of the transmitted signal grows with the decreasing energy of electrons.

Figure 4 shows the comparison of micrographs taken at two different energies; it demonstrates the higher thickness sensitivity of the transmitted electrons at 3 eV and higher topography contrast of the backscattered electrons at 12 eV. At 3 eV, the corrugation of the near-surface equipotential above the biased sample starts deforming the image around the adhered particle, thus illustrating the demands of this imaging mode regarding the smoothness of the sample. [4].

References:

- [1] I Müllerová and L Frank, *Advances in Imaging and Electron Physics* **128** (2003), p. 309.
- [2] D Drouin et al., *Scanning* **29** (2007), p. 92.
- [3] L Frank, E Mikmekova and M Lejeune, *Applied Surface Science* **407** (2017), p. 105.
- [4] The authors acknowledge funding from the Technology Agency of the Czech Republic (Competence center Electron microscopy, no: TE01020118).

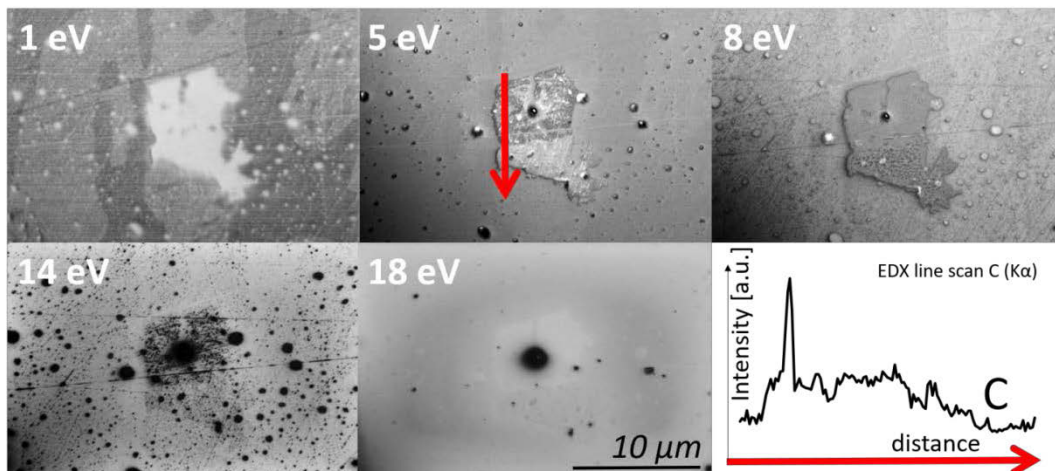


Figure 1. SLEEM images of the dual-phase steel surface obtained at various electron impact energies, together with the results of the EDX line analysis of Carbon distribution along the line marked in the 5 eV image.

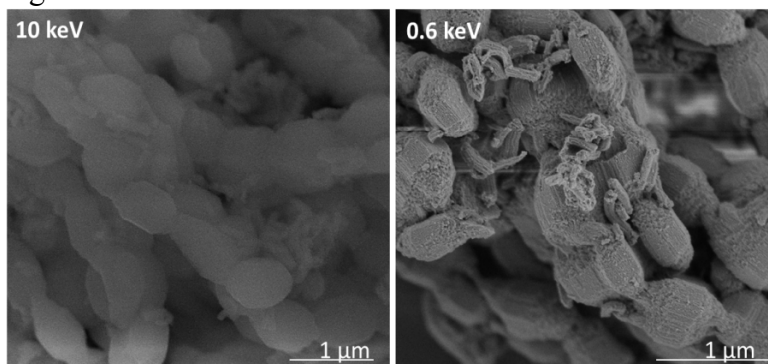


Figure 2. Ordered mesoporous silica-based nanocomposite materials for catalytic applications imaged in SEM at marked electron impact energies in BSE signal.

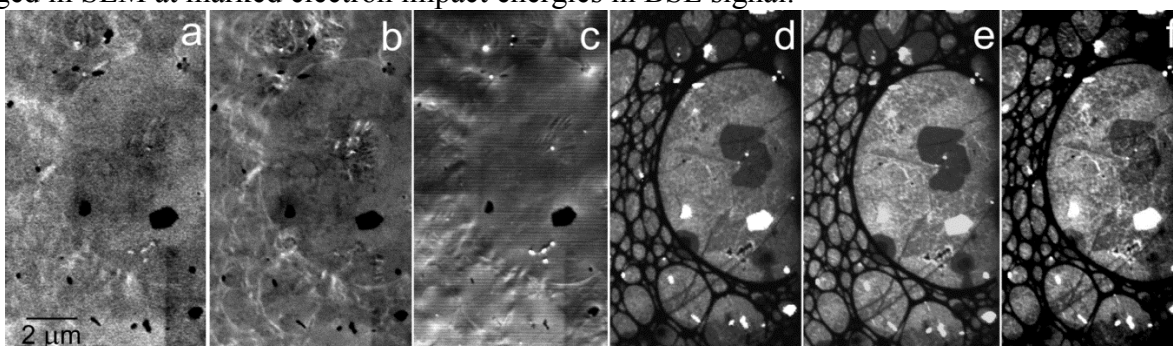


Figure 3. CVD grown graphene on lacy carbon and copper mesh, nominally 2 LG: electron energies 40 eV (a, d), 10 eV (b, e), and 0.9 eV (c, f), STEM images (a, b, c), SEM images in the “dark field” BSE signal (d, e, f).

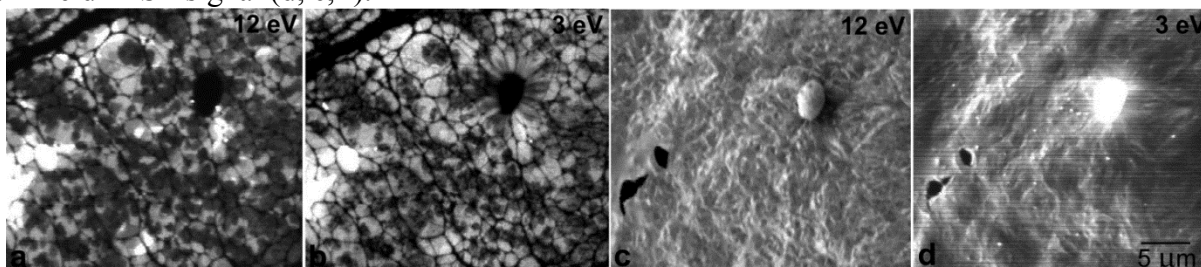


Figure 4. CVD-grown graphene on lacy carbon and copper mesh, nominally 3 to 5 LG, electron energies as labeled: STEM images (a, b), SEM images in the “dark field” BSE signal (c, d).