

Vibrational spectroscopy meets electron microscopy

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A broad range of new and conventional materials, namely polymers, blends, composites etc., have been characterized by light and electron microscopy. The properties of these often very complex materials are strongly influenced by the spatial distribution of their chemical compounds [1]. Analytical information has mainly been obtained by X-ray spectroscopy (EDX, WDX), providing elemental composition of the investigated samples. Vibrational spectroscopic imaging methods, such as Fourier transform infrared (FTIR) spectroscopy and Raman spectroscopy are particularly attractive analytical tools, as they provide both chemical and spatial information simultaneously. That spectrally and spatially resolved chemical information can be rapidly collected, analyzed and visualized, even for systems that do not have much inherent visible contrast in conventional light or electron microscopy [2].

We obtained comparative images from polymer blends by IR-ATR (FPA detector), Raman and ESEM (environmental scanning electron microscopy). Results will be discussed with regard to different lateral resolution and depth penetration, both depending on excitation wave length. We were able to show that the chemometric processing of imaging data can be a better approach than the classical data evaluation. The resulting images are characterized with more details and a better separation of the small objects.

Global Raman imaging can be a fast and simple technique, providing high lateral spatial resolution (down to the diffraction limit corresponding to the excitation laser wavelength) images of the sample of interest. Images of a PA/PTFE sample were obtained by Raman imaging and in an environmental SEM (ESEM). We imaged the same area first in ESEM, then in the Raman microscope, and afterwards in the ESEM again. In both methods the penetration depth is several micrometers (depending on exciting beam energy and interaction of photons or electrons with matter). During the second observation in the ESEM (Fig. 1) some additional PTFE clusters (obviously somewhat below the surface) became visible, and clusters at the surface appear very bright (charging by electrons). A subsequent Raman image revealed that the clusters originally at the surface were destroyed by the ESEM electron beam, and new clusters became visible, obviously due to a certain destruction (and melting) of the PA surface [3]. Briefly, this means that multiple analyses on the same sample should be well considered with respect to the eventual sample damaging during the analyses, thus leaving the most “destructive” method to be the final one.

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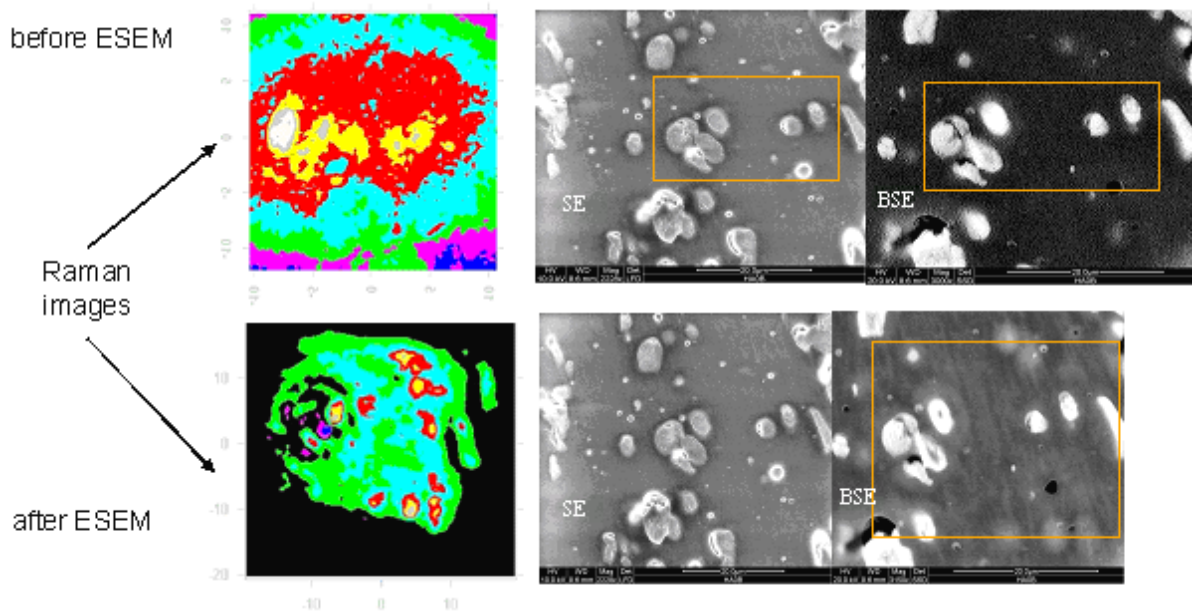


Figure 1. Raman images (left) and ESEM images (right, SE and BSE images).