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Behavior of Semi-volatile Particles under a Laser and Electron Beam – Influence on the Quality of Analytical Results

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INTRODUCTION

The combination of scanning electron microscopy (SEM) equipped with the energy dispersive X-ray detection (EDX) and micro-Raman spectrometry (MRS) for environmental research can give detailed information about individual atmospheric particles and is an extremely useful analytical tool [1,2,3]. Despite the undoubted advantages that combined analytical techniques can potentially offer, it is very important to not only examine the fundamental analytical requirements of the individual techniques, but also the characteristic behaviour during measurement. One of the main aspects, which needs be taken into account during the analysis of individual particles by combined SEM-EDX/MRS, is particle damage and any subsequent molecular changes that might occur as a result of exposure to a laser or electron beam. It is vital that modifications induced by the analytical method employed, be properly understood and controlled. Failure to do so could lead to unreliable data and a misinterpretation of the results obtained. While damage to particles by the electron beam can be minimized by employing a cold stage [4], the physical and chemical processes that affect damage caused to small particles by lasers still remains somewhat unclear. It can certainly be expected that the atmosphere (vacuum/air) and the beam intensity could have very significant influences, but the effects on the result and spectra have to date not been fully investigated and exploited.

EXPERIMENTAL RESULTS

Synthetic semi-volatile, single particles of different chemical compositions, i.e. $(NH_4)_2SO_4$, NH_4NO_3 and $NaNO_3$, were examined under different measuring conditions, e.g. vacuum or ambient atmosphere pressure and at room and liquid nitrogen temperature. Standard particles were prepared from the saturated suspensions using an airbrush (BADGER AIR-BRUSH, model 200). The samples were examined with a Micro-Raman (InVia micro-Raman spectrometer, Renishaw, UK,) interfaced with a FEG-SEM (SUPRA 40VP, Zeiss, Germany), equipped with liquid Nitrogen Cryo-stage. A laser with an excitation wavelength of 514.5 nm was used. Particles were selected using secondary electron images (SEI), after which the Raman spectra were collected. Thereafter particles were exposed for 10-20 s to the electron beam with an accelerating voltage of 10 keV and a current of 1 nA, after which a second Raman acquisition was performed. Reference spectra from the bulk standard materials were collected under the same conditions as those used for the single particles investigations.

It was observed that the electron beam sensitive particles remained stable under the laser beam in both ambient air and under vacuum conditions, and Raman spectra of sufficient quality could be recorded (red line in Figure 1 for $(NH_4)_2SO_4$). Spectra collected from the different particles showed good repeatability. The Raman spectrum collected after exposure to the electron beam (blue line in Figure 1 for $(NH_4)_2SO_4$) was of significant lower quality and clearly showed a higher background. Some of the Raman shifts could not be recognized, and in general Raman bands shift and broadening was observed.

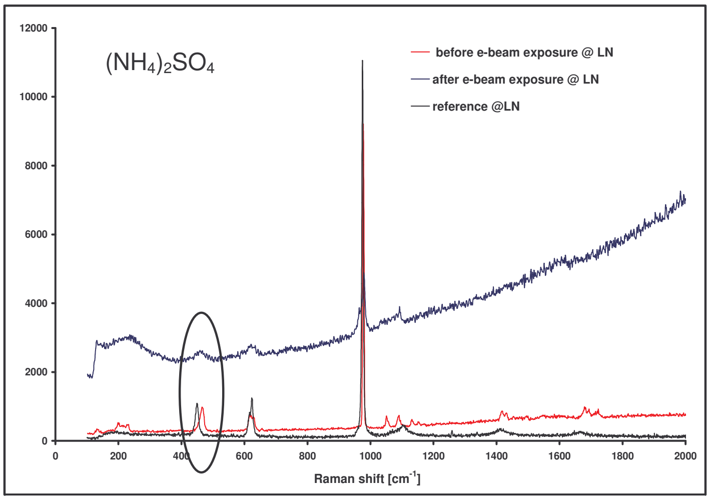

FIGURE 1: Raman spectra collected from an artificial $(NH_4)_2SO_4$ particle before and after the exposure to the electron beam

The deterioration under a laser beam can also be seen in Figure 2C. Particles no longer appear to be stable under the laser beam as it was before the electron beam exposure. This phenomenon has an impact on the analytical sequence in cases where the two spectra (Raman and X-rays) of the same particle are recorded and / or required.

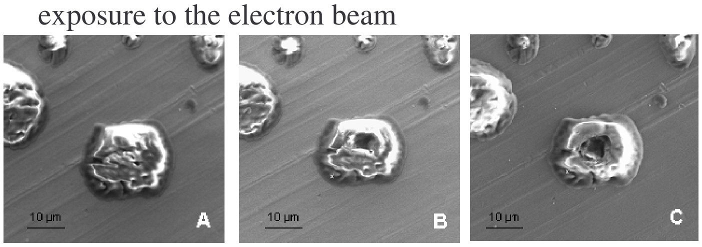

FIGURE 2. Artificial $(NH_4)_2SO_4$ particle (A) before the exposure to the laser and electron beam; (B) after exposure to e-beam; (C) after exposure to the laser and e-beam.

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