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Advantages of a local charge compensation system for FIB/SEM applications on insulating materials

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In recent years the use of FIB/SEM instruments has become a well established standard technique for three dimensional imaging and analysis of both inorganic as well as biological materials [1]. One remaining challenge lies in the investigation of insulating specimens as any charging effects on the sample surface inevitably inhibit clear and stable imaging, analysis and also sample modification such as FIB milling [2].

Latest FIB/SEM instruments rely on the method of local charge compensation (CC) to overcome the problem of surface charging. The CC functionality is based on injecting a gas locally close to the area of interest. The gas molecules are ionized by collisions with charged particles and the thus formed electrons or gas ions are attracted by the opposite charges at the sample surface resulting in the desired compensation effect. Due to the fact that the gas is only applied locally the overall chamber vacuum remains in a regime that still allows employing all standard detectors commonly used in FIB/SEM instruments. In particular no special detection schemes with a typically inferior signal to noise ratio are required. Different insulating materials were used to investigate the advantages of this CC method for SEM imaging, FIB milling as well as in-situ analytics such as EDS, EBSD or SIMS.

Fig. 1 shows SE images of a fibre glue sample taken at an acceleration voltage of 5kV. The picture without CC (left) is characterized by strong jittering and arcing-like artefacts, those with CC (right) shows the fine surface details of the fibre glue. Milling experiments were carried out on different insulating materials such as glass ceramic. In the left cross section of Fig. 2 several discrete cutting lines are visible on the surface denoting a random deflection of the primary beam by simultaneously stimulated surface charges. Using the CC mode as demonstrated in the left cross section of Fig. 2 a proper cross section can be prepared. The EDS spectra of a ZrO_2 sample acquired at 15kV acceleration voltage (see Fig. 3) indicate a significant impact of a charged sample surface. In the left spectrum without CC the cut-off voltage of bremsstrahlung is located at around 6.5kV, with CC switched on (right spectrum) the so called Duane-Hunt-Limit moves back towards the excitation beam voltage of 15kV. Since there is no detection of any characteristic X-rays possible above this cut-off voltage less information for elemental analysis can be gained when doing EDS on charged surfaces.

In this paper we will describe the impact of charging effects in FIB/SEM applications such as imaging, milling as well as the use of various analytical techniques and demonstrate how the use of the local charge compensation technique in the newest generation instruments can help to overcome some of the related issues.

[1] P.R. Munroe, *Materials Characterization*, **60** (2009) 2.
[2] M. Milani and S. Magni, *Imaging & Microscopy*. **8b** (2006) 38.

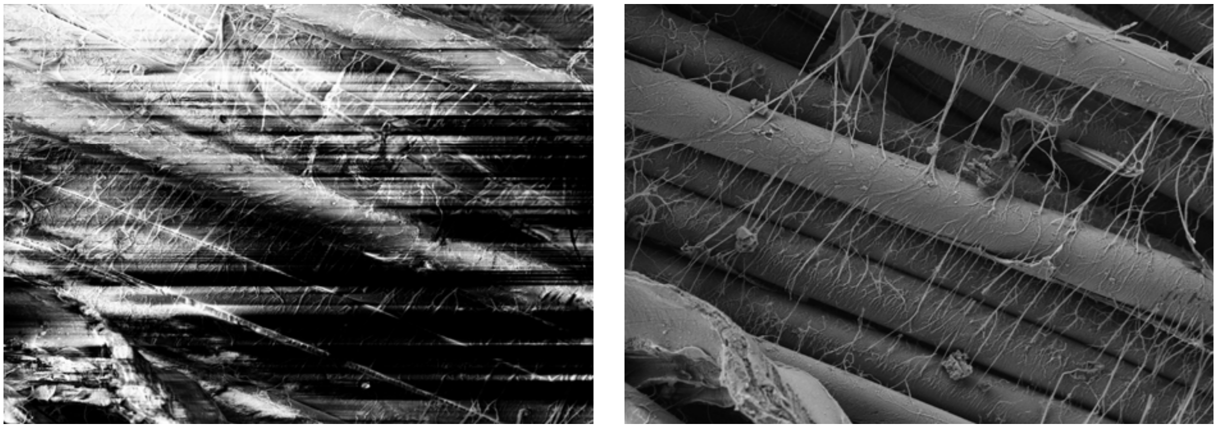

Fig. 1: SE images of a fibre glue sample taken at 5kV SEM beam voltage without charge compensation (left) and with charge compensation (right).

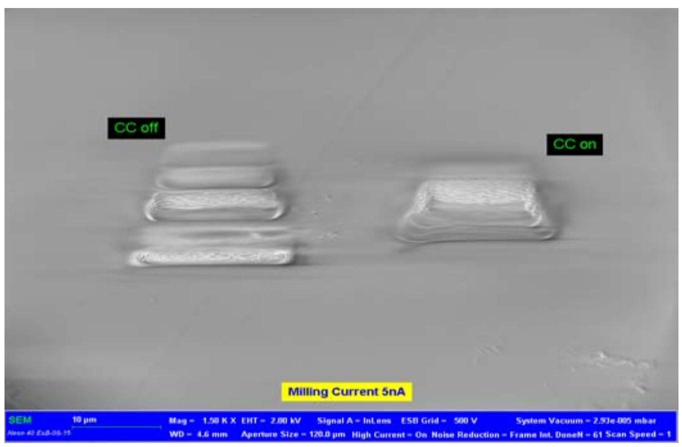

Fig.2: Cross sections on a glass ceramic surface milled with a 30kV Ga^+-beam. The left cross section was prepared without, the right with charge compensation.

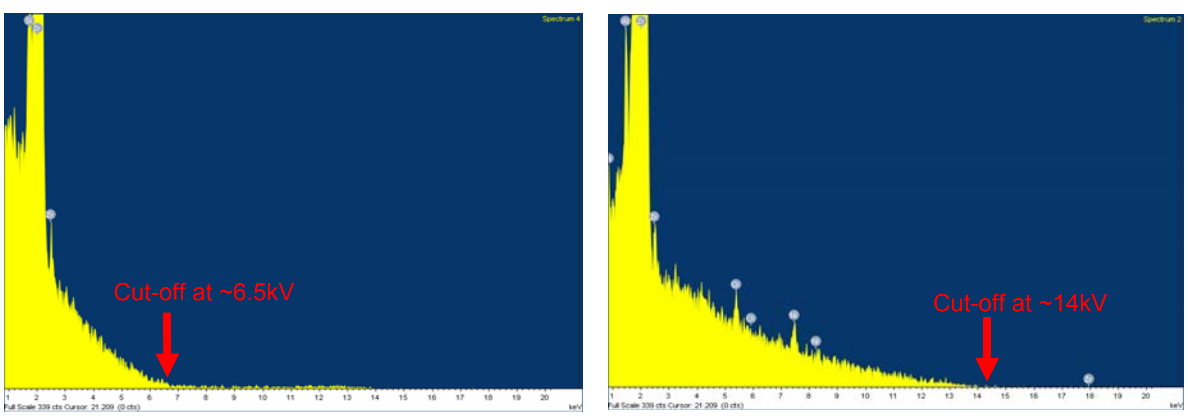

Fig. 3: EDS spectra of a ZrO_2 sample without (left) and with (right) charge compensation. The spectra were acquired using 15kV electron beam voltage.

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