On the potential of volcanic ash and powder sample investigation with FIB/SEM EBSD-EDS

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Keywords: EBSD, FIB/SEM, volcanic ash.
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We present a method for sample preparation of single particles and powder samples for characterization with microscopic techniques, i.e. focused ion beam (FIB), electron backscatter diffraction (EBSD) and energy dispersive X-ray fluorescence (EDS).

In terms of analytical and preparation methods, particulate matter are non-ideal samples. Particles have rough surfaces and may consist of multiple mineral phases and glasses. These phases may have different hardness and densities. Furthermore, charging may occur depending on the conductivity of the material. All of these can potentially impede proper microscopic data acquisitions. In case of EBSD, due to the 60° - 80° tilting which is normally applied, the prepared surface should be as flat as possible, otherwise backscatter electrons are shadowed and do not reach the detector. EDS measurement may also suffer from irregular surfaces due to shadowing as well as deflection of the electron beam or outgoing X-rays. Because of different sputtering rate of different phases, ion milling may be far from optimal for multiphase materials and re-deposition of milled material can be an issue on irregular surfaces.

Determination of the chemical and mineralogical composition of volcanic ash is crucial for estimating their hardness, density and melting points, which are needed to calculate radiative properties (optical properties for detection and recognition, climate effects) their transport trajectories (plume development and atmospheric live time) and harm to aircrafts (sand blasting effect to aircraft windows and engine failure by plugging of engine parts).

To determine the mineral content in single particles or grains, bulk methods such X-ray powder diffraction fail, since large diffracting volume is needed. In contrast, EBSD combined with EDS provides crystallographic and chemical information on a sub-micrometer scale. By simultaneous utilization of the two techniques, phase boundaries and grain boundaries can be confidently determined. Hence, the surface area fraction of mineral grains can be estimated. In addition, by acquisition of maps from various cross-sections, i.e. improving the statistics, a volumetric estimations of mineral and glass content of the bulk can be obtained.

Transported volcanic ash particles are large (2-50 µm e.g. Schumann et al. 2011), they are non-conductive in large parts and the matrix is supposedly glassy (Lieke et al. 2013), they have rough surfaces and their mineral composition may vary. Hence, they are far from ideal for the mentioned microscopic characterization techniques.

To overcome the mentioned shortcomings, samples were prepared by embedding small amounts of powder in an epoxy blended with colloidal graphite and silver particles. The blend is designed for satisfactory conductivity, stability under electron beam and for mechanical milling. Careful evacuation is applied to make the embedding material porosity-free. The dried mixture was cut, grinded and polished under angles of 45 and 54 degrees, to enable mounting the sample in ion milling (52°) and EBSD (70°) positions. Ion milling can optionally be applied for further sample preparation or for extension of analysis in the third dimension. Silver paint was used to glow the samples to a holder. Coating (gold or copper) was applied before the last polishing step (0.05 µm), to remain conductivity over gaps, holes and cavities that may have formed during grinding.

The preparation method was successful for data acquisition in a Helios Nanolab 600 DualBeam instrument (FEI, Eindhoven, The Netherlands) equipped with EDAX (Mahwah, IL, USA) EDS and EBSD detectors.

Identified phases embedded in glassy matrix were Rutile and Plagioclase, whereas Olivine was identified as single grains.

In the present work, the microscopic characterization of particles is shown on the example of volcanic ash particles from the recent Eyjafjalla eruption on Iceland, collected upon deposition in the vicinity of the volcano. That opens future perspectives for the investigation of other challenging particulate samples.

The particle samples were kindly provided by Andreas Vogel, FH Düsseldorf, Germany. We gratefully acknowledge an inspiring working environment at DTU-CEN. Excellent technical assistance, expertise and fruitful discussion were provided by Adam Fuller, Zoltan Imre Balogh and Berit Wenzel.

Schumann, U., et al. (2011). Airborne observations of the Eyjafjalla volcano ash cloud over Europe during air space closure in April and May 2010. Atmospheric Chemistry and Physics 11: 2245-2279.

Lieke, K. L., Kristensen, T. B., Korsholm, U. S., Sørensen, J. H., Kandler, K., Weinbruch, S., Ceburnis, D., Ovadnevaite, J., O'Dowd, C. D. and Bilde, M. (2013). The 2011 Grímsvötn volcano eruption: characterization of long range transported single volcanic ash particles. Submitted to Atmospheric Environment.