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# Sample preparation condition for SEM-EBSD: An example of quartz minerals in granite

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Electron Backscatter Diffraction (EBSD) is used to determine a crystallographic orientation of the mineral in the Scanning Electron Microscope (SEM). For rock samples, thin section is generally used for EBSD analysis. Mechanical polishing of thin sections with diamond paste cause damages to the lattices of minerals at the specimen surface. Thus, additional processes of vibratory chemical-mechanical polishing with colloidal silica suspension is required to remove the mechanical damage (e.g., Prior et al., 1999; Lloyd, 1987). Although a number of study of EBSD for rocks are published, there is little report of detailed procedure or optimum conditions for thin section preparation with colloidal silica vibratory polishing. The objective of this study is to examine the optimum conditions of rock sample preparation, especially time and normal load of vibratory polishing, for EBSD analysis using quartz crystals in granite.

Vibratory polishing were conducted using VibroMet$^{TM}$ 2 vibratory polisher (BUEHLER Inc.) with colloidal silica suspension (Model No.: 5904-S-64, pH: 9.8, particle size: 40 nm) (PRECISION SURFACES INTERNATIONAL Inc.).

Examined conditions of vibratory polishing with colloidal silica suspension were as follows:

Time: 0, 60, 120, 180 minutes

Normal load: weight (200 g)$\times$1, weight (200 g)$\times$3

The EBSD analyses were performed at the Waseda University with an automated electron back-scattered diffraction system (Channel5, HKL) attached to a SEM (HITACHI S-3400N) with a tungsten filament, an accelerating voltage of 25 kV, a working distance of 30 mm, and with the specimens tilted 70$^o$. Coating the specimen with a conductive material such as carbon removes the problem of charge build-up, but reduces the quality of Electron Backscattering Patterns (EBSPs). To eliminate the problem, EBSD analyses were performed under low-vacuum condition (30Pa) without coating. We selected 20 quartz grains per samples under the BSE image and 70$\times$70 $\mu$m area with 2 $\mu$m step size (total 1225 points) were measured for each grains. To evaluate the effects of the different vibratory polishing steps on EBSD pattern quality, hit rate which is percentage of value of correctly indexed points to total measured points was used.

The results of 0 minute vibratory polished sample (only diamond polishing) shows less than 55% of hit rate. After 60 minutes of colloidal silica polishing, 70% of the points are correctly indexed, while after 180 minutes this value increases to around 80%. Significant difference of effect of weight was not found. This result shows 60 minutes vibratory polishing is enough for EBSD analysis of quartz grain in granite. For another minerals or rock type, further examination might be required.

Keywords: SEM-EBSD, colloidal silica, vibratory polishing, thin section