## Sub-percentage Rietveld phase analysis of nanocrystals in obsidian

Ende, Martin[1] Kloess, Gert[1] Schorr, Susan[3]
[1]Universität Leipzig, Institut für Mineralogie, Kristallographie und Materialwissenschaft, D-04275 Leipzig, Scharnhorststraße 20 [2]Universität Leipzig, Institut für Mineralogie, Kristallographie und Materialwissenschaft, D-04275 Leipzig, Scharnhorststraße 20 [3]FU Berlin, FB Geowissenschaften, D-12249 Berlin, Malteserstraße 74

Quantitative phase analysis by X-ray powder diffraction (XPD) is normally restricted to mineral phases exceeding about one weight percent. The aim of the present investigation is to quantify mineral phases in the sub-percentage range using Rietveld analysis. The main object of the presented investigation is obsidian. This natural volcanic glass contains typically around 1...2 vol% micro- and nanocrystals. The structure and composition of the crystalline phases in obsidians are a basic source of information concerning the pressure and temperature of crystallisation as well as the kinetics of crystallisation and therefore the thermal history. The obsidian samples investigated in the present study are from different occurrences (Turkey, Armenia, Mediterranean Islands). Powder diffraction experiments were performed at the high energy beamline, ID15B, at the ESRF synchrotron radiation facility in Grenoble, France. The experimental set up was composed of a high energy monochromatic beam (87 keV energy), 500 $\mu$m x 500 $\mu$m size, and an on-line 2D detector consisting of a MAR345 image plate. During the measurement the sample was placed in a silica tube (˜4 mm diameter), which was rotated around the tube axis to reach higher statistics. A visual inspection of the obtained 2D images shows besides the signal from the vitreous matrix of the obsidian little spots, which correspond to single crystallites or clusters, which fulfil the Bragg condition. The small amount of the mineral phases, the resulting peak-to-noise ratio and the nanosize caused peak broadening, peak superposition, and the high background of the broad reflections of the vitreous matrix complicate the Rietveld analysis. Usually 1D diffractograms are obtained by azimuthal integration of the 2D images. Our more unconventional method to divide the integration over 360° in 1°-sections results in 360 diffractograms per one measurement. The part of the diffraction pattern arising from the vitreous matrix and the silica tube was fitted as far as possible and eliminated in the further data treatment. After this step the resulting 360 diffractograms were scanned for diffraction peaks of the mineral phases, which were collected into a matrix. Finally an integration of all Bragg peaks within the matrix has to be performed. This data treatment procedure results in one diffraction pattern reflecting only the crystalline part of the sample. The following quantitative phase analysis was done by Rietveld analysis using the FullProf and PowderCell programs.
The results thus obtained for the Ikizdere obsidian (Eastern Pontides, Turkey) should exemplify the efficiency of our procedure. Although the vitreous matrix amounts to 97.90.6 weight%, the rest of 2.10.6% of mineral phases could be separated and quantified with an uncertainty of 0.3 % or less to: albite 1.10.3%, cristobalite 0.40.1%, magnetite 0.240.07%, biotite 0.220.07%, hematite 0.080.02%, and quartz 0.070.22%.

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