Advantage of the use of Focused Ion Beam technique to specify mantle fluid inclusions

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Introduction

It is known that within the fluid inclusions solids (referred to as daughter phase) may crystallize as a result of cooling and/or reaction with the host mineral. If the volume proportions of the daughter phases are not known, the fluid composition determined can be misinterpreted. However, in situ measurement of the daughter phases can be complicated and ambiguous with conventional techniques because of their size and/or composition. In this study we report our results using FIB (focused ion beam) - SEM (scanning electron microscopy) technique on investigation of daughter phases in orthopyroxene-hosted fluid inclusions in mantle xenoliths from the Pannonian Basin.

Samples

Double-polished 100 microns thick orthopyroxenes (enstatite) were prepared from mantle xenoliths that were collected from two different alkali basaltic edifices (Szentbékkálla and Tihany) of the Bakony-Balaton Highland Volcanic Field, Central Pannonian Basin, Hungary. The mantle xenoliths from this region have been a subject of detailed petrologic, geochemical and deformation studies over the last decade (e.g. Szabó et al., 2004). Migrating fluid was trapped in orthopyroxenes at lithospheric mantle condition (Berkesi et al., 2009) forming negative crystal shaped inclusions that occur mostly along healed fractures. Their size varies between 2 and 30 microns, and it was found that the smaller the inclusion the more representative their fluid composition.

Previously, the fluid phases were studied revealing C-O-H-S fluid system (Berkesi et al., 2009). Although there are evidences on the presence of different daughter phases detected by Raman microspectroscopy (magnesite and quartz in orthopyroxene-hosted fluid inclusions), there was no information of their volume percentages because of their poor visibility.

Fluid inclusion exposing procedure by FIB technique

FIB-SEM measurements were carried out in a FEI QUANTA 3D FIB-SEM apparatus having both secondary and backscattered electron detector together with silicon drift X-ray energy dispersive spectrometer (EDS), operating at the Eötvös University, Budapest. Identification of daughter phases were mostly based on their morphology on the secondary electron images and the brightness on the backscattered electron images together with study of EDS spectra. Depending on the size of fluid inclusion, an optimal extent was removed by the ion beam and therefore, we could look at the exposed part of the inclusion by SEM and analyze with EDS. For example, we found that ~100-200 nm large steps are sufficient for 5 microns large orthopyroxene-hosted fluid inclusions to have accurate volume proportions of daughter phases. The actual progress of the inclusion exposing process is monitored acquiring secondary electron images of the sample.

Results

In accord with previous Raman analysis, magnesite and quartz have been found within the fluid inclusions. These daughter phases have sizes between 200 and 2000 nm occurring as cluster on some parts of the inclusion walls. Either the magnesite or quartz is mainly euhedral or subhedral. In addition, subhedral S-bearing solid phase (Fe-sulphide) has also been identified, sizing in a range between 400 and 1000 nm (Fig. 1). The presence of sulphide allows us to understand the role of H_2S fluid molecule found by

Raman microspectroscopy in the mantle fluid inclusions (Frezzotti and Peccerillo, 2007; Berkesi et al., 2009; Hidas et al., 2010). The vol% of the magnesite is ranging from 3.3 to 8.0, quartz from 2.6. to 6.2 vol.%, whereas the sulphide remains between 1.3 and 1.9 vol%.

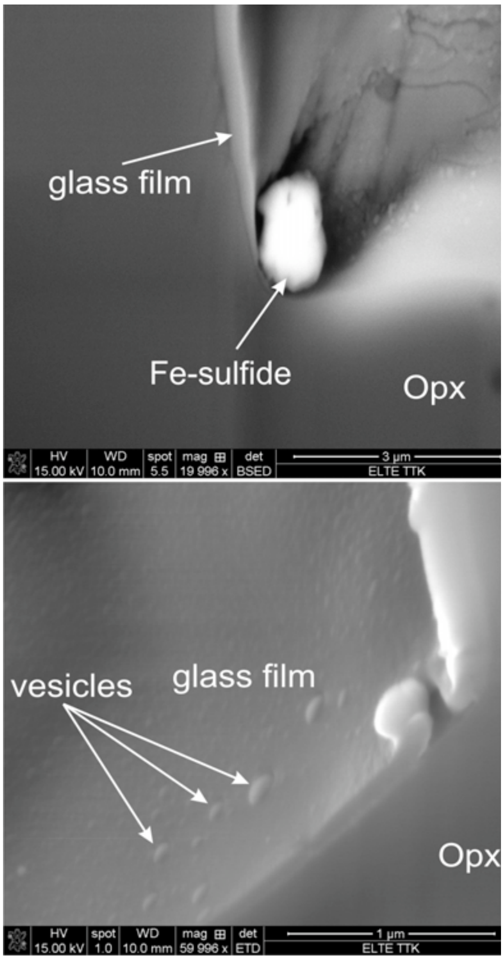

Fig. 1. Daughter phases identified by FIB-SEM in the mantle fluid inclusions. Upper image-backscattered electron image of Fe-sulphide daughter phase. Lower image-secondary electron image of the glass film having numerous spherical-shaped vesicles on its surface. Opx-orthopyroxene

One of the most interesting feature observed was a thin film covering the whole wall of the fluid inclusions in less than 1 vol%. They have a feature that is typical also for the volcanic glasses, showing numerous spherical-shaped holes (vesicles) on the surface as a result of the exsolution of volatiles (Fig. 1). The EDS analysis revealed that the glass has higher Si/Mg and richer in Fe, Ca and in some cases in Al than the host orthopyroxene. The thickness of the thin glass layer is around 100-200 nm, therefore remains invisible by using any other analytical method (optical microscopy, heating-freezing stage and Raman microspectroscopy).

Concluding remarks

Volume proportions of daughter phases, which have not been detected by using any other routinely used analytical technique for fluid inclusion studies, were identified by using the FIB-SEM technique on mantle fluid inclusions.

The acquired results of this study contribute to 1) precise quantification of the bulk fluid composition and 2) better understanding the mechanisms of the post-entrapment processes.

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