PRELIMINARY RESULTS ON THE DETERMINATION OF PHASE VOLUME FRACTIONS IN INCLUSIONS BY SYNCHROTRON-X-RAY MICROTOMOGRAPHY

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Introduction

The composition of individual inclusions can be presently determined with accuracy using recent developments of spot analysis techniques. However, volumetric properties are still very difficult to evaluate. Volumetric properties include the density of each phase, generally estimated at room temperature, and the vapour filling-ratio (φ) defined as the vapour volume relative to the total volume of the inclusion.

After estimating each phase density using microthermometric data and an appropriate equation(s) of state, the bulk density can be determined if we can precisely estimate φ.

Present methods of φ determination include: 1) rough visual estimation (see graphical charts in Roedder, 1984; Shepherd et al., 1985), 2) comparison of inclusion shape to simple geometrical figures (ellipse, sphere, cube), 3) image contouring and analysis on 2D sections and 4) application of the spindle stage (Anderson and Bodnar, 1993; Bakker and Diamond, 2006). In addition, in simple chemical systems, bulk density can be calculated using the microthermometric data only if an appropriate equation of state is available to evaluate accurately the volumetric properties (thermodynamic approach).

Uncertainty in the phase volume-fractions is usually the greatest source of error in geothermobarometric estimations based on fluid inclusions.

In this study, the X-ray microtomography method was applied to evaluate the application of such a technique to the determination of the 3D geometric features of inclusions.

Experimental conditions

Experiments were carried out using 3D X-ray microtomography at the synchrotron beamline TOMCAT (SLS, Villigen, Switzerland). The optimal setup consists in using both the 0.37 x 0.37 µm and the 0.74 x 0.74 µm optical equipment with an energy of about 20 keV to determine the most appropriate setup. The acquisition was achieved using the phase contrast enhance tomography technique, which produces diffraction contrast at the boundary between phases in addition to the present contrast in the transmitted beam. It seems to be the most adequate technique that allows distinction of the liquid (sometimes two liquids), vapour and solid phases on the greyscale histogram, and permits accurate segmentation.

Sample selection

Several natural samples were chosen for the experiments. They include different inclusion configurations:

- two-phase inclusions with a rather small vapour bubble. These inclusions are mainly high salinity water, with no gas component, and occur in fluorite
- three-phase inclusions, including two liquids (aqueous and carbonic, respectively L_1 and L_2) and a carbonic vapour bubble. The vapour bubble and liquid L_2 was analysed by Raman and results indicate that CO_2 is the only gas component. These inclusions therefore belong to the H_2O-NaCl-CO_2 system.

First results and further developements

Inclusions were analyzed by microthermometry and were carefully photographied. When possible, polished sections were made in two perpendicular directions (Fig. 1).

Except in special limited cases described below, imaging results using X-ray micro-tomography are of good quality and three-phase inclusions, both liquids and vapour could be discriminated (Fig. 2).

The application of X-ray tomography is subject to the following limitations:

- inclusions where the vapour bubble is large enough to be at contact with the inclusion

walls; the precise determination of the boundaries is subject to caution because diffraction patterns are complex at the three-phase contact point.

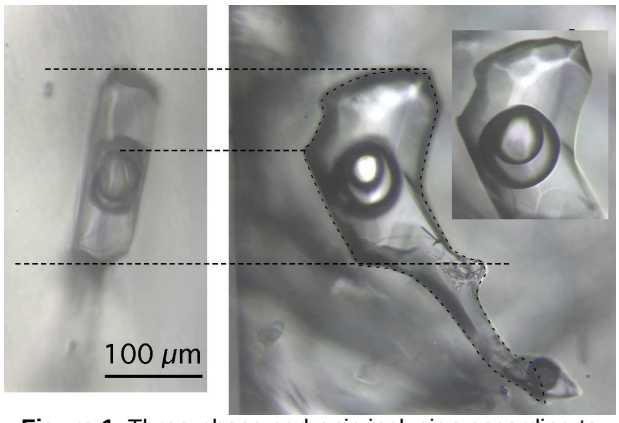

Figure 1. Three-phase carbonic inclusion according to two perpendicular sections

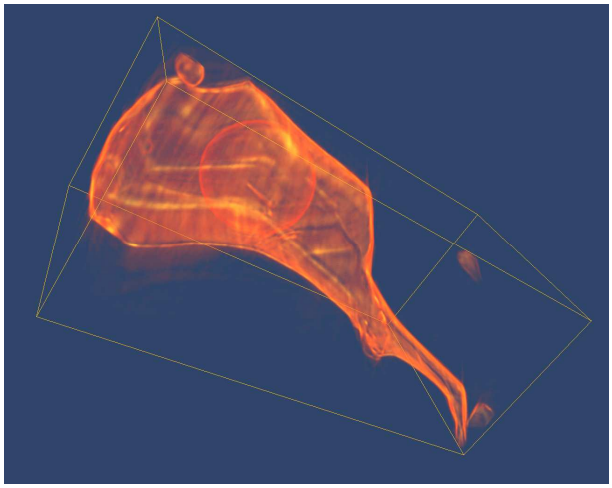

Figure 2. Image of the inclusion shown in Fig. 2 obtained using 3D X-ray microtomography

- inclusions of small size compared to the resolution (pixel size) of the image: The larger the inclusion in the camera field, the smaller the relative error.

- inclusions in which the different phases have similar densities.

Image processing was done using a combination of different filters to first reduce noise on the images, and localise boundaries between the different phases (Fig. 3).

Image processing includes an anisotropic diffusion filter, isoline drawing, and erosion-dilatation filters depending on the properties of the initial 3D image.

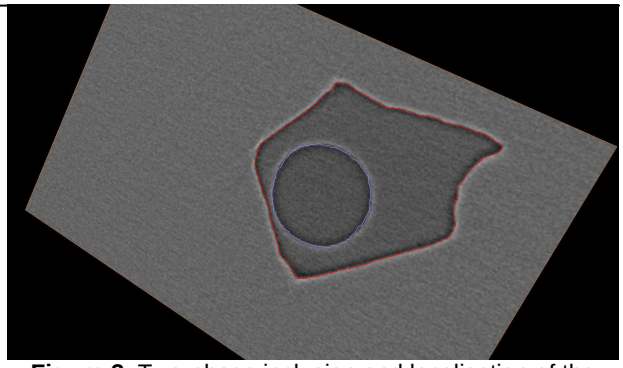

Figure 3. Two-phase inclusion and localisation of the vapour-liquid boundary

Data processing permits estimation of the volume of both the bubble and the bulk inclusion. Then, values of φ were compared with estimations based on the optical and thermodynamic approaches.

Vapour volumes determined on the microscope assuming a spherical shape give very consistent results with 3D X-ray microtomography. The thermodynamic approach is still not conclusive. As the chosen inclusions were relatively large, they were particularly sensitive to decrepitation during inclusion analysis by microthermometry and bulk homogenisation temperatures could not be measured. Moreover, as the chosen samples are natural, the exact composition is not known and so the thermodynamic treatment is subject to errors. Whereas φ calculated by both approaches are of the same order of magnitude, significant differences were noted.

Further developments of this approach would require the production of large synthetic fluid inclusions of known composition in order to evaluate the accuracy.

Determination of volumetric properties is of particular interest to determine the P-V-T-x of natural fluid mixtures when using synthetic fluid inclusions as micro-reactors.

References

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