


Contribution ID: **76**

Type: **Oral presentations**

TTS Microdifraction of microcristals embeded in thin polished films

Wednesday, 17 June 2015 09:50 (20 minutes)

One interesting problem in the field of petrology is the use of non-destructive techniques to investigate thin mineral polished films (25-30μm thickness) attached to a glass substrate. In this type of samples commonly show microsized crystal of different minerals formed in close contact during its genesis (see Fig. 1). In order to study such systems synchrotron radiation has two very interesting properties: a high penetration capacity when a short wavelength is used; and the possibility of using microfocused spots. Four years ago, we developed the through-the-substrate microdiffraction (tts-μXRD) technique for studying this type of films using a spot down to ($100\times100\mu m^2$). By this way we obtained powder patterns of aerinite and discovered a new mineral called ilerdite [1].
Microdiffraction station of MSPD beamline is very well suited to further develop this technique. Superconducting wiggler can reach quite high energy (30 KeV and above) and KB-mirrors render a highly focused spot ($15\times15\mu m^2$). With such capabilities, we have extended the μ-tts technique to the study of microcrystals embedded in thin polished films of compact materials [2]. The procedure consists on a series of steps: i. collection of a limited number of 2D diffraction patterns (frames), for every selected microcrystal (randomly oriented, usually between 5 and 10 crystals); ii. refinement of crystal metrics from a 1D diffraction pattern obtaining by radial average of the frames; iii. determination of the orientation of the reciprocal lattice for every microcrystal, this allows assigning hkl miller indices to the spots in every frame and combining the intensities of the different frames for each crystal (frame merging); iv. combination of the different sets of intensities for every crystal (multicrystal merging) to obtain a set of single-crystal-like integrated intensities.
We show the validity of the technique by some representative examples in petrology in which glass substrates have been used and Patterson-function direct methods applied to solve the corresponding crystal structures [3]. This technique can be applied to other fields like material science and cultural heritage.

References

[1] J. Rius, A. Labrador, A. Crespi, C. Frontera, O. Vallcorba, C. Melgarejo, J. Synchrotron Rad., 2011, 18, 891-898.
[2] J. Rius, O. Vallcorba, C. Frontera, I. Peral, A. Crespi, C. Miravitlles, IUCrJ, 2015, 2, (accepted)
[3] J. Rius, IUCrJ, 2014, 1, 291-304.

Caption (s) - Add figures as attached files (2 fig. max)

Figure 1. Thin ofite film with polycrystalline aerinite (AE) veining and diopside (D) microcrystals.

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Session Classification: Session 2 - VII AUSE Congress 2015

Track Classification: VII AUSE Congress