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Report:

Main objective of the proposal was to use White Beam Synchrotron Radiation Source X-Ray Diffraction Topography (WB-SRS-XRDT), transmission geometry, for the study of lamellar structures in micas and twin lamellae in spinels. The results suggest to leave over the studies on micas, whereas the analysis of spinel twin lamellae merits to be continued and deepened.

MICAS

The purpose was to obtain a better understanding of the nature of misoriented “macroscopic layers”, observed in some natural samples by XRDT with conventional source (MoKα_1 radiation). Such layers often showed complex stacking sequences, which could be related to different polytypes coexisting in the same sample. Therefore micas were investigated by WB-SRS-XRDT to determine the mutual orientation of the “macroscopic layers” and their stacking sequences; besides informations on the growth mechanism responsible for the “layered” structures were expected.

Screw dislocations parallel to the c axis are widely considered as playing a major role in the mica growth and in the polytypes formation: such dislocations were not found. Unfortunately this observation, that would be important, can’t be utilised because of the high strain concentration of the samples that determines strong unrisolved contrasts, that could mask screw dislocations contrasts. Furthermore, the defects observed on the topographs don’t play significant role in the growth, and consequently, they don’t allow to specify the operating growth mechanism.

Finally, WB-SRS-XRDT didn’t give the expected additional information on the orientation of the “macroscopic layers” and on stacking sequences; therefore the micas analyses will be temporarily left over.

SPINELS

The aim of the experiment was to apply WB-SRS-XRDT on natural samples from different localities, in order to investigate the presence, within them, of twin lamellae (TL) – thin twinned individuals – and to determine their crystallographic orientation. TL in natural spinel were previously observed and characterized, in samples from Burma that macroscopically appeared as single crystals, by means of WB-SRS-XRDT

(Fregola, 2001), thus arising interest in checking their presence in other natural spinel samples from different provenances, in order to find a relationship between their growth mechanism and the genetic environmental conditions.

The data collected at ESRF show further evidences of thin TL (their thickness ranging from about 100 μm to 300 μm), in spinel samples coming from Burma and Ceylon. The twin law has been found and the mutual orientation between TL and the matrix has been determined.

Twinning of TL has been found to be always according to the spinel twin law: {111} as both twin and composition plane, or equivalently, <111> as twin axis.

The morphology of some samples is flattened in such a way that, the slice plane is parallel to the two {111} twin planes bounding the TL inside the bulk crystal, or in other words, the slice normal coincides with the twin axis. For such an orientation of TL with respect to the slice plane (and, consequently, to the incident beam), all the reflections with diffraction vectors $\mathbf{g}_{hkl}$ parallel to the slice plane, corresponding to lattice planes normal to it, produce the superposition on the same topograph of the diffraction contrasts from the TL and from the matrix, making it difficult to recognize the presence of TL, to determine its orientation and, mainly, to distinguish whether the observed structural defects are inside the TL or in the upper or lower part of the surrounding matrix. However, TL parallel to the slice plane give a few Laue topographs of only the TL, with well resolved diffraction contrasts from the defects inside them, and a few others when mounting the sample on inclined morphological faces. This is the case of two samples from Burma: each of them contain a thin TL parallel to the slice plane, with an anomalous incomplete morphology, and with central inclusions from which originate channels running parallel to the twin plane towards the rim of the TL

The Laue topographs from some other samples show complex cross-twinning patterns of different TL intersecting each other. For example, a spinel coming from Ceylon contains two distinct TL (labelled B and D) intersecting but not penetrating each other, within a matrix consisting of two larger and equally orientated individuals (A and C). This sample was cut transversally with respect to both B and D, thus reducing their thickness so that the diffraction contrasts from the defects within them are too much short and superimposed on most of the topographs to allow their characterization. However, with this kind of orientation of the TL to the slice plane and to the incident beam, the determination of the reciprocal orientation of the individuals from the Laue topographs has been easier than the case of TL parallel to the slice plane, because there was a larger number of spots of the TL alone. It has been found that: 1) the slice plane cuts A and C parallel to their $(\bar{1}\bar{1}1)$ lattice planes, B parallel to its $(\bar{1}\bar{5}1)$ lattice planes and D parallel to its $(\bar{1}\bar{1}5)$ lattice planes; 2) the twin planes are $(1\bar{1}\bar{1})_{A/B}$ between A and B, $(\bar{1}11)_{B/C}$ between B and C, and $(\bar{1}\bar{1}\bar{1})_{AC/D}$ between A and D and also between C and D; 3) for indexing the topographs superimposed on the same Laue spot, the transformation of the Miller indices from the reference system of A and C to that of B, and the one from A and C to D, are respectively represented by the following relations:

$$h_B = 1/3\ (h_{AC} + 2k_{AC} + 2l_{AC}),\ k_B = 1/3\ (2h_{AC} + k_{AC} - 2l_{AC}),\ l_B = 1/3\ (2h_{AC} - 2k_{AC} + l_{AC});$$

$$h_D = 1/3\ (h_{AC} - 2k_{AC} - 2l_{AC}),\ k_D = 1/3\ (-2h_{AC} + k_{AC} - 2l_{AC}),\ l_D = 1/3\ (-2h_{AC} - 2k_{AC} + l_{AC});$$

4) the two twin lamellae B and D are not in twinning orientation the one to the other (Fregola *et al.*, 2003).

An interesting result is that TL were found in samples coming from the same kind of mother rocks (impure dolomitic marbles) of two different localities (Burma and Ceylon), thus suggesting a possible relationship between the nucleation and growth of TL and the genetic environmental conditions. The characterization of defects inside the TL, in particular of dislocations, would be very useful for the understanding of the origin of TL in spinels and for evaluating their role in promoting the growth of the whole samples, but at the moment data are not enough. To obtain the lacking data, in the next experiment each sample will be investigated with several different orientations of TL to the incident beam.

Therefore, the results obtained till now confirm the interest in going on studying TL in natural spinels. In particular, the next objective will be the WB-SRS-XRDT characterization of defects inside the TL, in order to clarify the growth mechanism, promoting the growth of the whole sample, and the environmental genetic conditions under which TL can nucleate and grow.

References

Fregola, R.A.: Growth defects and growth marks of spinels from Pegu (Burma). *PhD Thesis* (2001).

Fregola, R.A., Scandale, E., Tempesta, G. and Agrosì, G. (2003): XRDT study of twin lamellae and dislocations in natural spinels. *FIST GEOITALIA 2003, Bellaria (Italia) 16-18 sept. 2003.*