Synchrotron *tts* Microdiffraction: from Data Acquisition to Structure Solution

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The viability of the synchrotron through-the-substrate microdiffraction technique (*tts*-μXRD) to the study of polycrystalline zones in polished thin-sections on glass-substrates was already shown in [1]. In general, data collection for this particular application is relatively simple and the intensities from circularly averaging the Debye rings can be processed with multiple powder diffraction software. However, further extension of the technique to the structural study of microvolumes of randomly oriented crystals embedded in polished thin sections of compact materials is far more complicated. Among the principal difficulties one can mention i. the large background due to the glass-substrate; ii. the limited accessible portion of reciprocal space (no free rotation of the polished thin sections); iii. the diffracting volume reduction if the illuminated embedded microcrystal partially leaves the gauge volume during rotation (e.g. when the microcrystal and beam sizes are of the same order). Another circumstance to consider (especially for space groups prone to twinning) is the presence of intensities with contributions from multiple twinned domains. For this extended application, two data collection modes can be used to obtain the integrated intensities of the diffraction peaks (i.e. sequential and specific *tts* modes). Both are complementary and the respective data (suitably modified in the sequential case) can be processed with the TTS_software [2]. The procedure based on the specific *tts* mode which will be described in detail, *i.* collects a limited number of consecutive 2D patterns (frames) for each randomly-oriented crystal microvolume; *ii*. determines the orientation of each randomly-oriented crystal microvolume from the central frame which allows assigning the *hkl* indices to the spots; *iii*. merges the intensities of the different frames into a single crystal dataset (frame-merging); *iv*. merges the individual crystal datasets to produce a more complete dataset suitable for structure refinement/solution (multicrystal merging). The application of the δ-recycling approach [3] to structure solution will be shown on some representative examples from Petrology. Special attention will be devoted to its more demanding application to low-symmetry phases (large number of missing reflections) only existing in one microvolume of the polished thin-section.

[1] Rius J., Labrador A., Crespi A., Frontera C., Vallcorba O., Melgarejo C. (2011) *J.Synchrotron Rad*. **18** 891-898
[2] Rius J., Vallcorba O., Frontera C., Peral I., Crespi A., Miravitlles C. (2015) *IUCrJ*, **2**, 452–463.
[3] Rius J. (2012) *Acta Cryst.* A**68,** 77-81.