Imaging Single Crystals in Powder Samples

J. P. Wright¹

¹European Synchrotron Radiation Facility, 71 Avenue des Martyrs, Grenoble, 38000, France.
wright@esrf.eu

What would be the ultimate instrument for a crystallographer to do X-ray diffraction experiments? How close are the instruments we have now compared to the ones we could imagine? I first visited the ESRF to study magnetite at the powder diffraction beamline, and much later, we could find some tiny single crystals in the same sample. We needed to wait for some advances in instrumentation to refine that structure. Since then, synchrotron instrumentation has not stopped improving. The approach I want to tell you about is using tomographic methods together with diffraction from polycrystalline samples to create images of the crystals that are inside.

A very small beam size is the key to getting X-ray data from tiny crystals. In 2020, the ESRF source was upgraded to a “fourth generation” lattice that offers even higher X-ray brilliance than before. Now there is enough flux in the monochromatic X-ray beam to recrystallize a copper wire via the beam heating effect [1]. Various “nanoprobe” endstations have been developed, and many offer facilities for have beam size well below a micron in size. We have optimised the instrument at ID11 [2] for a beam size of about 150 nm and high-energy X-rays (40-70 keV). The limitation for beam size is determined by the mechanics of the diffractometer, which need to be good enough to align a crystal in the beam and do a monochromatic rotation experiment while keeping a tiny crystal aligned in the beam. The station has a photon-counting CdTe Eiger2 4M pixel detector and this allows fast (500 fps) data collection and excellent counting efficiency.

Historically, we would collect powder diffraction data for Rietveld refinement, or PDF measurements from powder samples; or select one single crystal to get a dataset for structure refinement. Now that the X-ray beam size is so small, and the detector is so fast, we can systematically scan across a small powder sample and then process the data according to whatever is in the sample. My first experience with this XRDCT method for single crystal style data was looking at impurity precipitation in some metal particles [3]. Within a metallic glass, the same kind of data collection could reveal the signature of local mechanical strain fields [4] following deformation. This combination of X-ray diffraction and tomography can reveal the atomic structures together with the microstructure of a sample. As well as producing beautiful images of samples (figure 1), these data may open up new avenues for looking at structure-property relationships.

Figure 1. A cross section of a K-type thermocouple. Colours represent orientation and brightness is the reconstructed density.


I would like to thank all of my colleagues at ESRF, as well as the user groups and collaborators who have worked with us at the beamline, as well as during data processing when developing and testing software.