"Methods of high-pressure single-crystal x-ray diffraction"

A satellite workshop of the 26th European Crystallographic Meeting

Darmstadt (Germany), September 3-4 (2010)

Single-crystal diffraction provides the most accurate structural data on the compression of crystalline solids. The methods for high-pressure diffraction developed over the last 3 decades are mature and easy to use in both the laboratory and at synchrotron sources, but are not well known throughout the crystallographic community. In summer 2008 a group of high-pressure crystallographers from Europe and the USA met in Padova (Italy) to discuss how to promote high-pressure single-crystal methods. On the occasion of a second meeting a year later in Copenhagen (Denmark) we agreed to meet on a regular basis and decided to offer a first workshop open to everybody in association with the 26th European Crystallography Meeting held in Darmstadt, Germany (September 3-4, 2010).

The workshop, hosted on the campus of TU Darmstadt and organised by R. Miletich, A. Grzechnik and H. Ehrenberg, attracted 50 participants from all over the world for a very full 2 days of lectures and demonstrations. Following a lecture to provide a general overview of the entire workflow for a high-pressure experiment, and the additional challenges to be overcome compared to ambient-pressure measurements by R. Angel, the first morning featured presentations by R. Miletich, C. Hejny and B. Periotto on diamond-cells, how to load them, and then perform the data collections on the diffractometer. In the afternoon M. Alvaro reviewed software for data integration before the workshop split in to separate sections in which R. Angel, D. Gatta, T. Balic-Zunic and A. Grzechnik showed how to integrate high-pressure data with several commercial and freeware software packages. The second day was devoted to the separate subjects of equations of state (T. Boffa-Ballaran) and intensity data reduction and refinement (K. Friese, R. Angel), followed by structure validation and structure analysis (T. Balic-Zunic, K. Friese). The majority of the second afternoon was devoted to discussing detailed data issues with the participants, along with a demonstration by the research group of R. Miletich of preparing and loading diamond-anvil cells. The workshop was concluded with a short presentation by H. Ahsbahs on a new generation of diamond-cell design, and a review of synchrotron beamlines available for single-crystal diffraction.

The workshop participants included researchers from many fields (chemistry, physics, geology, and materials science) at all levels of experience with high-pressure single-crystal x-ray diffraction ranging from beginners to experts. More than one third of the participants were PhD students. The contribution to the

workshop from all participants was immense; comments on the presentations and subsequent discussions enlivened the workshop. With its completion, we hope that all of the participants (including ourselves, the speakers!) are now more confident that they can perform single-crystal high-pressure X-ray diffraction measurements and can complete the data reduction and analysis to obtain the highest-quality data.

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Matteo Alvaro, Ross Angel, Tonci Balic-Zunic, Tiziana Boffa-Ballaran, Karen Friese, Diego Gatta, Andrzej Grzechnik, Clivia Hejny, Ronald Miletich, Fabrizio Nestola, and Benedetta Periotto.