

Structure determination by single-crystal X-ray diffraction (SXD) at megabar pressures

Przemyslaw Dera,^{a*} Charles T. Prewitt^b and Steven D. Jacobsen^a

^aGeophysical Laboratory, Carnegie Institution of Washington, 5251 Broad Branch Road NW, Washington, DC 20015, USA, and

^bDepartment of Geosciences, University of Arizona, Tucson, AZ 85721-0077, USA

This issue contains papers based on presentations at an international workshop on *Structure Determination by Single-Crystal X-ray Diffraction (SXD) at Megabar Pressures*, held on 13–14 November 2004 at the Argonne National Laboratory in Chicago, USA, and sponsored by the Consortium for Materials Properties Research in Earth Sciences (COMPRES). The main objective of this workshop was to define and bring together a community of researchers from different scientific disciplines, such as mineral physics, petrology, solid-state chemistry, condensed matter physics and materials science, interested in the development of new experimental methods and state-of-the-art facilities that would allow the study of crystal structures using single-crystal samples at ultrahigh pressures. Discussions at the workshop were aimed at answering the following questions: (i) is it scientifically important/justifiable to develop megabar SXD techniques? (ii) is there enough interest and demand in the community for such development? (iii) is there a consensus among the experts on how such a task should be approached? (iv) how can the effort be coordinated and managed most efficiently?

The meeting was attended by over 60 participants, 14 of whom came from outside the USA, representing five different countries. The large attendance of this relatively specialized workshop alone makes a strong case that there is indeed sufficient interest and demand in the community to develop novel high-pressure single-crystal techniques at the various synchrotron radiation sources. The workshop was timed at a critical juncture in the early development of synchrotron-based megabar single-crystal techniques, which has been, to date, led mostly by our Japanese and European colleagues at SPring-8, the ESRF and the SRS Daresbury facility. The workshop brought together nearly all the active experts in the field and a wide range of supporters representing complementary specialties. An important highlight of the workshop was the participation of experts from non-high-pressure disciplines, such as synchrotron microdiffraction, detector technology and time-resolved protein crystallography, who were willing to help us implement their novel methods and solutions into our experimental setups, and were looking forward to applying solutions developed in the high-pressure field to their own problems.

The workshop was divided into four sessions representing different branches of high-pressure crystallography, and was focused on guiding the participants through the logical path leading from the motivation for the development, through existing solutions (and problems), and toward technologies of the future. The papers are organized into five categories: (i) from powders to single crystals, (ii) minerals and inorganic solids, (iii) molecular solids, (iv) techniques and experimental methodology, and (v) facilities and instrumentation.

Among the techniques of high-pressure crystallography discussed in our special issue are both the well established methods such as monochromatic single-crystal and powder diffraction, as well as novel and revolutionary state-of-the-art approaches including absorption foil spectroscopy, energy scan techniques and X-ray emission spectroscopy. The objects of investigation vary from oxide and silicate minerals to organic molecular crystals, but share the same underlying thermodynamic principles and mechanisms of the pressure-induced phenomena.

It is important to emphasize that although reaching megabar pressures with SXD techniques was the primary objective of our discussions, it soon became clear from the workshop presentations that the applications of the methods developed for high-pressure research will have wider-reaching impact on other communities. The ability to sample submicrometre-sized grains with depth resolution, to solve crystal structures from XRD data collected within milliseconds (possibly during laser heating to several thousand K), and to map spatially the microstructure of deformation, have so far been in the area of

dreams and wishes of geoscientists, materials scientists and engineers. These applications will soon benefit from the development of ultrahigh-pressure SXD techniques.

One of the very important outcomes of the discussions at the workshop was the formation of a collaborative group of high-pressure crystallography experts that submitted a joint proposal to the National Science Foundation Major Research Instrumentation Program for a coordinated nation-wide development of dedicated synchrotron infrastructure and methodology for structure determination of solids from single-crystal micrograins. Just days before closing this volume we learned that the project has been funded and that resources will become available to create the much-needed tools of the future.

We wish to thank the Board of Editors at the *Journal of Synchrotron Radiation* for publishing this special issue emanating from the workshop on megabar single-crystal diffraction, and we hope that synchrotron users outside the high-pressure community will enjoy reading the state-of-the-art from our field. We also express our thanks to COMPRES for financial support and for cultivating specialized workshops such as this one, and to Dr David Lambert and the Instrumentation and Facilities Program at the Division of Earth Sciences of NSF for support through grant 0217389, which enabled us to start the development of novel methods for structure determination at ultrahigh pressure and, in turn, has led to this workshop.

The success of the workshop is attributed to the participants and speakers; and we thank them for their insight and enthusiasm. We truly hope the impulse of knowledge and technique stemming from this initiative will bring forth unforeseen breakthroughs and exciting discoveries in high-pressure science well into the future.