

16.X-14 HIGH PRESSURE AND VARIABLE TEMPERATURE RESEARCH WITH SYNCHROTRON RADIATION IN THE UNITED STATES. By Earl F. Skelton, Condensed Matter and Radiation Sciences Division, U.S. Naval Research Laboratory, Washington, D.C. 20375, U.S.A.

By combining existing diamond-anvil cell high pressure techniques with extremely brilliant synchrotron radiation (SR), high pressure, variable temperature structural information can be obtained in subsecond time frames. Programs designed to exploit the advantages of SR have been underway for several years on the two hard x-ray SR rings in the U.S., on SPEAR at Stanford University and on CHESS at Cornell University. These efforts involve several groups and span a wide range of materials problems at elevated pressures, including, inter alia, critical points in a first-order/second-order low temperature phase transition, pressure induced chemical disproportionation, high temperature transition mechanisms in geologically important materials, cross-calibration, and transformation kinetics studies. This work will be reviewed and future trends in this field, which may be anticipated with the increased availability of wiggler beam-lines and more sophisticated photon-detection systems, e.g. areal detectors and charge coupled devices, will be discussed.

16.X-15 HIGH PRESSURE AND HIGH TEMPERATURE CRYSTALLOGRAPHIC STUDIES WITH SYNCHROTRON RADIATION IN JAPAN. By H. Iwasaki, The Research Institute for Iron, Steel and Other Metals, Tohoku University, Sendai, Japan.

In the Photon Factory in Tsukuba where synchrotron radiation is available for general use, a multi-anvil apparatus has been constructed. It has been designed to make possible crystallographic studies under combined high-pressure and high temperature conditions using larger volume of sample. The essential part of the apparatus consists of six sets of square-faced tungsten-carbide anvils and a 500 ton hydraulic press. The sample is placed in the center of a solid cube-shaped pressure-transmitting medium and irradiated by a polychromatic, horizontally polarized beam. Diffraction pattern is recorded in the energy-dispersive mode within a few minutes using an SSD mounted on the 2 θ arm. The press and goniometer system (5.5 ton in total weight) is fixed on a movable stand which can adjust precisely the sample position with respect to the incident beam. Taking advantage of brightness and small divergence of synchrotron radiation, an X-ray optical system is designed so that unwanted component of intensity from pressure transmitting medium is kept as small as possible. The intensity data stored in an MCA are transferred to a microcomputer system and promptly analysed.

Examples of the studies performed are a determination of the phase relation in phosphorus in the range of 1 bar-60 kbar and 25°C-1100°C and a precise measurement of the compressibility of gold at 600°C.

16.X-16 SINGLE CRYSTAL DIFFRACTION UNDER HIGH PRESSURE WITH SYNCHROTRON RADIATION. By Heinz Schulz, MPI Festkörperforschung, D-7 Stuttgart 80, Fed.Rep.Germany

Synchrotron radiation offers an excellent tool for diffraction experiments with single crystals under high pressure. The narrow natural collimation of the synchrotron radiation and the high intensity gives a much higher signal/background ratio compared to a normal X-ray tube. It allows therefore to reduce considerably the size of the crystal and in this way the size of the high pressure chamber. In a pilot experiment it has been demonstrated that Bragg intensities could be measured on a CaF₂ crystal with only 6 μ m edge length (Bachmann, Kohler, Schulz, Weber, Kupcik, Wendschuh-Josties, Wolf, Wulf, Angew. Chemie 95 (1983), 1013-1014). This shows the way to carry out structure investigations in a pressure range much higher than about 100 kbar, which seems to be the upper limit for such experiments with a normal X-ray tube. The tunable wave length of the synchrotron radiation will make large progress in the orientation procedure of small crystals on the one hand and on the reduction of absorption effects on the other hand. The crystal scattering power is proportional to λ^3 . It follows, longer wave lengths increase the scattered intensity and make it easier to orient a crystal. In addition a reduced resolution can be applied. Smaller wave lengths reduce absorption effects. However, at the moment only very few single crystal diffractometers are available at synchrotron radiation sources, which may be used for such studies. The talk will be focussed on very recent results on high pressure research with these instruments and on corresponding research proposals. It is based on a world wide general inquiry among high pressure and synchrotron radiation scientists on their activities on single crystal diffraction under high pressure with synchrotron radiation.

16.1-1 THE SPACE GROUP DETERMINATIONS FOR 3 SINGLE-CRYSTAL MEMBERS OF THE UO₃-NH₃-H₂O SYSTEM ON A RECIPROCAL LATTICE EXPLORER. By S.D. Le Roux and A. van Tets, Nuclear Development Corporation of South Africa (Pty) Ltd, Private Bag X256, Pretoria, Republic of South Africa.

A main feature of a single-crystal reciprocal lattice explorer is the undistorted reciprocal lattice photographs produced in the De Jong-Bouman and Buerger Modes. Exposures for 3 different single-crystal members of the UO₃-NH₃-H₂O system are presented and the corresponding lattice parameters and space groups determined. The proposed chemical formulae (Debets and Loopstra, J. Inorg. Nucl. Chem. (1963) 25, 945) of the 3 members are (i) UO₃.2H₂O (ii) 3UO₃.NH₃.5H₂O and (iii) 2UO₃.NH₃.3H₂O. The results of our single-crystal study differ from those of previous authors, who mostly used powder samples. Compound (ii) is traditionally better known as ADU or Yellow Cake but the above-mentioned formulae remain to be verified.