16. APPARATUS AND TECHNIQUES

16.6-8 MOUNTING A CLOSED CYCLE TWO STAGE COOLING DEVICE ON A FOUR-CIRCLE DIFFRACTOMETER. E. Krabbe Larsen, Z. Hanzlikova and S.B. Rasmussen. Department of Chemistry, Aarhus University, DK-8000 Aarhus C, Denmark.

Instrumentation for collection of three dimensional diffraction data at temperatures below the boiling point of liquid nitrogen has been developed. A closed cycle two stage refrigerator has been mounted on the 4-shaft of a four-circle goniometer. The sample crystal is attached to the nominal 100 mm via a copper block. The refrigerator mount is used in principle as a goniometer head without arcs. The x-y-z centering device has a range of ±15 mm for the z-direction along the 0-axis and ±22 mm along x and y. Dose tails and eometrics with narrow divergence allows a precise, firmly lockable centering of the crystal.

The whole refrigerator unit undergoes all the normal φ-, ω- and 2θ-movements during data collection without any rotating vacuum seals. These are usually integral, vulnerable parts of existing liquid helium temperature cryocoolers. When refrigeration is not required, the cryostat can be driven at a peak voltage of ~5k~ with a repetition rate of ~10Hz ~ and a peak current of ±2×10^6mA. The whole refrigerator unit undergoes all the normal φ-, ω- and 2θ-movements during data collection without any rotating vacuum seals. These are usually integral, vulnerable parts of existing liquid helium temperature cryocoolers.

16.7-1 TIME-RESOLVED STRUCTURE ANALYSIS BY A REPEATED PULSE X-RAY DIFFRACTION TECHNIQUE. H. Terashita, T. Minato, A. Hichijo, K. Sakaue and S. Iida. Department of Physics, Kwansei-Gakuin University, Nishinomiya 663, Japan.

X-ray diffraction technique has so far been applied mainly to static measurements, where the specimen is kept in thermodynamic equilibrium. There, however, an increasing interest in the time-dependent structure developed after the system is brought into a non-equilibrium state by a sudden change of the electrical mode. The use of this apparatus in a ferroelectric crystal after a sudden change of the electric field will be discussed. The preliminary results of the above experiment appeared in Jpn. J. Appl. Phys. 22 (1983) 144.

16.7-2 SURFACE ANALYSIS BY X-RAY MICROBEAM DIFFRACTION*. By M. Rappaz, M. Kaspar and E. Blank. Materials Department, Swiss Federal Institute of Technology (EPFL).

Microstructural features in the size range from approximately 20 µm to 1 mm are frequently encountered in materials science (e.g. phases distribution in cast alloys, plastic zones at crack tips, etc.). Existing diffraction techniques do not match very well with this size range: while electron diffraction in TEM applies to areas smaller than 10 µm, standard X-ray techniques, apart from topography, normally average over microstructural inhomogeneities.

In order to characterize as-cast or deformed dendritic specimens, a Scanning X-ray Microbeam Diffractometer (SXMD) has been developed. In this technique the specimen is attached to a four-circle goniometer and analyzed in back-reflection. With a special goniometer setting, the specimen surface can be translated with respect to the X-ray beam without changing the diffraction conditions. The microbeam (diameter > 100 µm) allows selection of a small area while decreasing the beam divergence (> 3° of arc). The irradiated zone can be directly viewed with a laser beam directed through the X-ray pinhole system using a set of adjustable mirrors. Lattice orientation and diffraction line profiles within individual dendrites were measured by SXMD over areas of approximately 2 mm². Such mappings were directly correlated with optical micrographs and Berg-Barrett topographs, the latter being recorded on the same apparatus.

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