

16.6-8 MOUNTING A CLOSED CYCLE TWO STAGE COOLING DEVICE ON A FOUR-CIRCLE DIFFRACTOMETER.

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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 ϕ shaft of a four-circle goniometer. The sample crystal is attached to the nominal 10K station 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 ± 5 mm for the z-direction along the ϕ -axis and ± 2 mm along x and y. Dove tails and eccentrics with narrow tolerances allow a precise, firmly lockable centering of the crystal.

The whole refrigerator unit undergoes all the normal ϕ -, χ - and ω -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 cryo unit can be dismantled in a few minutes and the device with a small attachment allows mounting an ordinary goniometer head for room temperature data collection. Thus, the system is simple, robust and easy-to-operate both at room temperature and at cryogenic temperatures. Furthermore, the use of a closed cycle refrigerator makes the system more economical than comparable cryocooler systems which are based on flow cryostats. The centering unit will be on display at the Congress.

16.7-1 TIME-RESOLVED STRUCTURE ANALYSIS BY A REPETITIVE PULSED X-RAY DIFFRACTOMETER SYSTEM.

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X-ray diffraction technique has so far been applied mainly to static measurements, where the specimen is kept in the thermal equilibrium. There is, however, an increasing interest in the time-dependent structure developed after the system is brought into a non-equilibrium state by a sudden change in the external condition. For investigating the time-dependent system we have developed a repetitive pulsed X-ray generator. Our generator utilizes the impact of high-velocity electrons on a target material. The high voltage is generated by a Marx generator coupled to a pulse forming line. The X-ray tube can be driven at a peak voltage of 300kV and a peak current of 5kA with a duration of about 40ns at a repetition rate of up to 10Hz. One X-ray pulse contains about 3×10^6 Cu-K α photons. The peak intensities of the K α radiation of the pulsed X-ray is about 10^6 times as large as those from the sealed-off X-ray tube operated at 30kV and 20mA. Since the maximum duty cycle is 10Hz, the average intensity is comparable to that from the sealed-off X-ray tube. A transmission Laue photograph can be taken with only ten shots. The diffraction patterns are detected using a multichannel photodiode (MCPD) array. Since the dead time of the MCPD detector is negligible, diffraction data can be effectively accumulated in a stroboscopic mode. The use of this apparatus as a time-resolved X-ray diffractometer will be reported at the meeting. Especially, the time-dependent diffraction patterns 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 μ m 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 μ m, 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 μ m) 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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16.7-3 USE OF TEXTURE GONIOMETRY FOR THE AUTOMATIC DETERMINATION OF A LIMITED SET OF ORIENTATIONS. By A. VADON, J.J. HEIZMANN, Centre d'Etude des Textures, Laboratoire de Metallurgie Structurale, Faculté des Sciences, Ile du Saulcy, 57045 METZ (FRANCE).

The determination of the relative orientations between crystals and between non-similar phases is important to understand the mechanisms of the evolution of materials (crystal growth, topotaxy, deformation and transformation of unit cells ...). The crystal orientations are generally found by means of LAUE prints or by electron diffraction, but the procedure soon becomes complicated when several orientations are present.

We propose a method which allows to find the orientations by texture goniometry and computing. In the case of a small number of orientations, the complete or incomplete pole figure shows a limited number of spots. Some of the spots belong to the same orientation.

The automatic treatment allows

- 1- to select the spots belonging to each orientation,
- 2- to determine each orientation,
- 3- to infer the relative orientations.

The method is illustrated by several examples :

- the growth of Al, Al₂ Cu orientated eutectics
- a topotactic reaction showing the relative orientations of Fe₂O₃ and of Fe₃O₄ resulting from the chemical reaction.