13-Defects, Microstructures and Textures

MS-13.02.04 'REAL TIME' SYNCHROTRON RADIATION TOPOGRAPHY : RECENT INVESTIGATIONS AND PROSPECTS. J. Baruchel, ESRF, 38043 Grenoble, France.

Many 'real time' topographic investigations were recently performed on synchrotron radiation facilities (LURE, Daresbury, NSLS). They include the initial stages of lattice relaxation in strained layer semiconductors (Barnett et al), a stroboscopic investigation of vibration modes in quartz (Capelle et al), the nucleation of the incomensurate phase of quartz (Dolino et al), a study of phase transitions in perovskite like crystals (Dudley et al), the transmission of dislocations through a grain boundary in Si (Jacques et al), the formation of cellular microstructure during directional solidification of AlCu binary alloys (Grange et al), the deformation of AlCuZn shape memory alloys (Jourdan et al), the electrical field related distortion in the weak ionic conductor α-$LiIO_3$ (Rejmankova et al), and the behaviour of the interfaces during a magnetic phase transition and nucleation of the phases in the neighbourhood of a triple point in MnP (Sandonis et al), as well as the dislocation motion and multiplication in ice (Whitworth).

This type of study will greatly benefit from the availability of third generation synchrotron radiation sources. We have recently shown that the typical exposure time for a topograph reduces to about 5 .10^{-3} s when working at the ID11 Materials Science beamline of the ESRF. This is also the typical exposure time expected at the ESRF ID19, 140 meters long, topography and high resolution diffraction beamline. On the other hand the high energy characteristics of this radiation allows the investigation of bulky ($\approx$1mm thick iron crystals, for instance) or heavy materials (heavy semiconductors, rare-earth compounds). The new fields of investigation which are opened will be discussed.

MS-13.02.05 TRIPLE CRYSTAL X-RAY CHARACTERIZATION OF NEARLY PERFECT SINGLE CRYSTALS by M. Servidori, CNR-Istituto LAMEL, Via Castagnoli 1, I-40126 Bologna (Italy)

Triple crystal X-ray diffractometry is a high resolution characterization technique for investigating the detailed distribution of intensity in the vicinity of the reciprocal lattice points. The use of highly perfect monochromator/collimator and analyzer crystals enables the separation of the diffracted intensity into its dynamical and kinematical components. In this way, information on lattice strain and static atomic disorder can be obtained from computer simulation of the dynamically diffracted intensity, whereas the analysis of the diffuse (Huang) scattering distribution in the reciprocal space gives information on the type, size, density and, in some cases, depth of the lattice defects. Moreover, dilation effects can be separated from orientation effects (mosaic spread), with a sensitivity depending on the number of reflections in the collimator and analyzer crystals. Examples of application are reported on the analysis of silicon crystals subjected to processes of the electronic device industry, which show the advantages of the triple crystal technique on the conventional double crystal one. Emphasis is also given on the extension from three to four crystal geometry which allows an easy procedure of rocking curve simulation of thick surface deformed layers, such as those produced by high energy ion implantation.

MS-13.02.06 X-RAY TOPOGRAPHY AND DIFFRACTOMETRY OF MULTIPLE LAYER STRUCTURES. By R. Koehler, MPG-AG Roentgenbeugung, Berlin, Germany.

Topography and diffractometry are complementary techniques for the characterization of single crystalline layer structures.

Early stages of strain relaxation and lateral strain gradients with dimensions larger than a few micrometer are the domain of topography. Quasi plane wave techniques as e.g. the double crystal topography are highly sensitive to minor strain differences of the order of 10^{-7}. The double crystal technique is applicable even to curved samples with radii down to some meters if a specially bent collimator is used (Jenichen, Köhler and Möhling, J.Phys.E, 1988, 21, 1062). Monochromator-collimator combinations provide a collimated beam with a reduced wavelength band, so decreasing the dispersion effects in (n,-m) sample settings (DuMond, Phys.Rev., 1937, 52, 872-883; Bartels, J.Vac.Sci.Technol., 1983, B1, 338-345). An additional analyzer crystal restricts the detected signal to a small region in the reciprocal space of the sample. This triple crystal technique can be used as well for diffractometry as for topography (Fewster, J.Appl.Cryst., 1992, 25, 714-723). In general, however, the optimum apparatus layout is different for topography and for diffractometry.

Topographs can be taken also in layer reflections even with laboratory sources. Misfit dislocations were shown to generate very different contrasts in substrate and satellite reflections of an about 3 µm thick AlGaAs Bragg reflector. As in this case, the contrast of defects is usually understood only by doing contrast simulations. Still most widely used is the halfstep algorithm (Authier, Malgrange, and Tournarie, Acta Cryst. A, 1968, 24, 126) based on the Takagi-Taupin theory (Takagi, Acta Cryst. A, 1962, 15, 1311-1312; Taupin, Bull.Soc.Fr.Mineral.Crystallogr., 1964, 57, 469-511). The penetration depth of the radiation is small in the Bragg case. Therefore the topographic contrast can be evaluated in many cases taking into account orientation contrast only. This provides a simple means to calculate lateral strain inhomogeneities and to characterize threading dislocations.

Simulation of diffraction curves is the basis of extracting data from experimental high resolution diffractometer curves. For nearly periodic structures this can be done automatically.

PS-13.02.07 EFFECT OF STRESSES ON SILICON SINGLE-CRYSTALS. By A.Kohno, N.Aomine, Y.Soejima, H.Sakashita† & A.Okazaki*, Department of Physics, Kyushu University, Fukuoka 812, Japan; †Centre of Advanced Instrumental Analysis, Kyushu University, Kasuga 816, Japan.

By the method of high-angle double-crystal X-ray diffractometry (HADOX), high resolution measurements have been made on silicon crystals at low temperatures. To the original experimental arrangement in HADOX, two slits have been added: one for limiting the area of the specimen crystal to be examined, and the other for defining the resolution of 2θ. Thus we can couple the original ω scanning of the specimen with the 2θ scanning of the detector, and determine two-dimensional intensity distribution on a reciprocal lattice plane. This technique, named the 2θ-resolved HADOX, enables us to determine a change in the lattice constant and that in the crystal orientation, if any, separately and precisely. With specimen crystals in a special form, diffraction from a region with or without stresses has been examined; the design of the specimen is based on the information obtained from a γ-ray diffraction experiment. It is found that the silicon crystal shows normal behaviour on both cooling and heating when it is stress-free. With stresses, on the contrary, behaviour is anomalous: The lattice constant, the Bragg peak width in ω and 2θ, the crystal orientation and the diffraction intensity are discontinuous, as functions of temperature, around 210 K on cooling, while they are normal on heating. The results are compared with those of previous experiments of HADOX of the original version, neutron diffraction, ultrasonic attenuation and thermal expansion; a discussion is given in terms of stresses and relaxation of stresses.

