(1) M. Gailhanou, T.Baumbach, U.Marti, P.Silva, F.K. Reinhart, M.Ilegems; Appl.Phys.Lett. (1993) in press

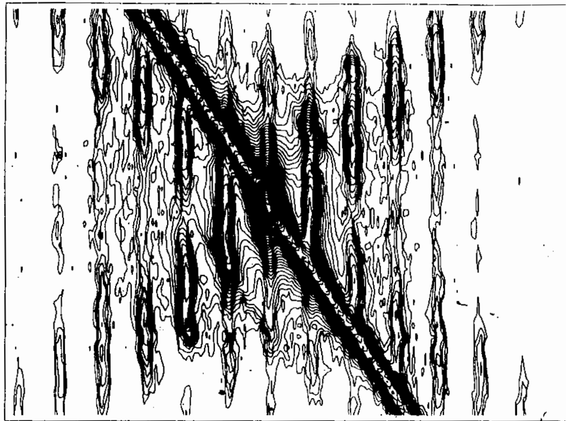

Fig.1 Contour plot of the reciprocal space mapping of a GaAs surface grating in the vicinity of (004) GaAs

PS-13.03.09 SKELETON STRUCTURES IN POROUS SILICON.

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The micro structure of luminescent and non-luminescent porous silicon formed by electrochemical etching has been characterized by X-ray and electron diffraction. Experimental and theoretical research has been recently focused on light emission properties of porous silicon. The mechanism resulting in the visible luminescence at room temperature is still a controversial question but it seems to be related to the structural features of the porous layer. The structure of porous silicon has been studied previously (Barla et al.,J.Cryst. Growth, 1984, **68**, 727).and several authors confirm the single-crystalline nature of the layers. Diffuse scattering around Bragg reflections has also been analyzed recently to obtain information on the pore structure of the films (Bensaid et al., Solid State Comm., 1992, **79**, 923). This communication deals with the comparison of X-ray and electron diffraction patterns of etched silicon wafers with two different crystallographic orientations. Transmission electron microscopy and double crystal diffractometry were used to characterize the samples. Photoluminescence measurements were performed to correlate the results with the structural features of the layers. Results of this study reveal several differences in the crystallinity of the skeleton structure for luminescent and non-luminescent samples.

Sponsors: CNPq, FAEP, FAPESP

PS-13.03.10 TEXTURE EFFECT IN ANALYSIS OF RETAINED AUSTENITE IN STEEL.

By Matti Järvinen, Lappeenranta University of Technology, P.O.Box 20, SF-53851, Lappeenranta, Finland

X-ray diffraction is a standard tool for determining austenite concentration in polycrystalline steel. If the grains of austenite and ferrite are randomly orientated in the sample, the work can be done rapidly and accurately. To resolve the problem it is sufficient to measure carefully the integrated intensity of only one reflection from each phase.

However, very often the metallurgical samples have preferred orientation or texture, especially if the material is made by mechanical working or if there has been recrystallization by heat treatment or due to welding. The texture distorts the true intensity ratios in the experimental data and gives an erroneous value to the austenite content. In these cases more reflections are usually measured and the final outcome is calculated as an average of different pair values.

For improving this calculation procedure I have developed a method that takes the special features of texture effect into account (Järvinen,M. 1985. Lecture notes 2/85. Lappeenranta Univ. of Technology, Finland.). The method is based on symmetrized harmonics expansion for the representation of the orientation distribution of the crystallites in the sample. The method presumes the use of specimen spinner in data collection.

In retained austenite analysis the orientation distribution of each phases is represented separately by cubic harmonics expansion. This introduces adjustable parameters into the formula of theoretical intensities. The parameters of the model, including consentration parameters of ferrite and austenite phases, are determined by fitting theoretical integrated intensities with the experimental data.

For demonstrating the use of the method, integrated intensities of six reflections (*200, 220, 311* for austenite and *200, 211, 220* for ferrite) from several samples were measured using $CuK\alpha$ radiation. It was found that this information was sufficient for determing the parameters, but the result was much more accurate when more reflections was measured.

PS-13.03.11 TEXTURE STUDY OF $YBa_2Cu_3O_{7-\delta}$ THIN FILMS USING X-RAY DIFFRACTION.

By D. Chateigner, P. Germi and M. Pernet*, Laboratoire de Cristallographie CNRS, BP 166, 38042 Grenoble Cedex 09 France.

Recently there has been growing interest in texture analysis of high temperature superconductors because preferred orientation is closely linked to electrical properties. The superconducting currents are stongly anisotropic and flow in the CuO_2 planes, so the orientation of the ab-planes of individual domains markedly influences the properties of thin films. In the case of $YBa_2Cu_3O_{7-\delta}$ (YBCO) thin films on single-crystal substrates, textures are extremely strong and routine procedures developped for texture analysis of metals are generally inappropriate. This has led us to develop special procedures (Chateigner et al., J. Appl. Cryst., 1992, 25, 766-769).

YBCO films grown on MgO substrates are generally found to consist of domains oriented with either the a-axis or c-axis perpendicular to the substrate surface. The in-plane texture were carefully studied by X-ray pole figure measurements. That texture is revealed by the complex (103/013) pole due to the (110) twinning in the samples. The pole figure data were analysed from χ and ϕ scans (χ and ϕ being the classical angles in the four-circle diffractometer geometry). It was found that there were two in-plane epitaxial state in which the YBCO a-axis (or b-axis) was parallel to either [100] direction or [110] direction of the substrate. For the description of texture components the general formulation : $c_{\perp}\alpha$ is used when the c-axis direction of the film is aligned with the substrate normal, α being the angle between the in-plane a or b direction of the film and the [100] of the substrate.

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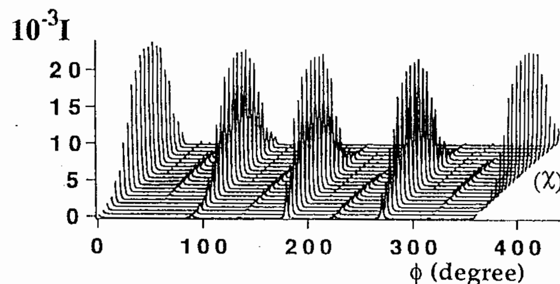

X-ray χ-ϕ scans of the (103/013) reflexion of YBCO film deposited on (001)MgO. The thickness of the film was 150 nm.

The studied films had a relatively constant critical temperature Tc, however a drastic reduction in the critical current density Jc was observed (from 5.10^5 A/cm^2 to 3.10^4 A/cm^2) as the mixture ratio $c_{\perp 45}/c_{\perp 0}$ increased (from 1% to 6%). This fact confirmed the drastic decreasing effect of highly misoriented grains on Jc (Dimos et al., Phys. Rev. Lett., 1988, 61, 219-222)

In conclusion the X-ray pole figure analysis is not only an effective way to study the texture of YBCO films, but also an excellent way to quantify the lattice alignement between the film and the substrate.

The authors wish to thank Alcatel-Alsthom Research for providing the samples and for critical current measurements.

PS-13.03.12 A STUDY OF THE SURFACE DAMAGE ON A GaAs WAFER BY X-RAY DOUBLE CRYSTAL DIFFRACTION.

By Ge Zhongjiu*, Cao Wanghe, Li Mei and Liu Weina, Changchun Institute of Physics, Academia Sinica, China.

It is known from X-ray dynamical diffraction theory that the full width at half maximum (FWHM) of the rocking curve of a reflection from a perfect crystal is very narow, about a few seconds of arc. But the surface damage and the defects in a crystal lead to a lower crystal perfection. The effects of dynamical and kinematical diffraction overlap, and the rocking curve FWHM increases. Patel suggested (J.R. Patel *et al.*, *Acta Met.*, 1962, **10**, 759) that the broadening of the FWHM from the surface damage of the wafer is larger than that due to dislocations. Therefore, the depth of the surface damage layer can be detected by measurement of the FWHM at different depth from the surface of a wafer.

In this work, the surface damage on a GaAs wafer has been studied by measuring the FWHM of an X-ray double crystal diffraction rocking curve in the non-parallel (+,-) setting. Seven polished GaAs wafers have been etched to a depth of about 30 μm. The FWHMs before and after etching are shown in Table 1. It is obvious that the differences in FWHM for samples No 1, 3, 4 , 5, are slight, but the decrease in FWHM after etching is of 2 to 6 seconds for samples 2,6 and 7. From this, we can conclude that there is a damage layer on these three samples. Fig. 1 shows a double crystal reflection topograph of a polished (001) GaAs wafer. In the topograph, there is a group of streaks whose curvature and trend are the same as the trace of the cutting on the back of the wafer. In order to determine the thickness of the surface damage layer of the polished wafer, some steps have been etched on the surface of the tested wafer with the controlled chemical step-etching technique. The height of the steps is of the order of 1 to 2 μm. Fig. 2 shows the relation between the etching depths and the FWHMs for three polished GaAs wafers. The curves 1 and 2 show that the changes in the FWHMs tend to steady after the depths reach 1.4 and 3 μm, respectively, but curve 3 may be considered a straight line. From the above result, we can draw the conclusion that the thicknesses of the damage layer are 1.4 and 3 μm, respectively for wafer 1 and 2, but that there is no damage layer for wafer 3. From more experimental results, it is shown that the surface damage layer on polished GaAs wafers is in general 1 to 4 μm deep. The damage layer may result from cutting, lapping or polishing. It must be etched out before making a device, because it is very detrimental to the quality and the lifetime of a device.

Table 1. The FWHMs of some GaAs wafers before and after etching.

Number of the sample		1	2	3	4	5	6	7
FWHM	before etching	16.5	17.0	17.0	18.0	18.0	23.0	24.0
(second of arc)	after etching	16.5	14.5	16.5	17.5	18.0	17.0	18.0

Fig. 1. X- ray double crystal reflection topograph. 224 reflection Cu $K_{\alpha 1}$ 40KV. 160mA. 1 st crystal: (111) Si 224 reflection

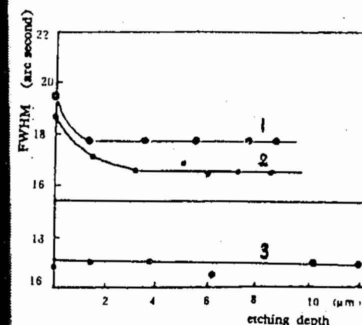

Fig. 2. Relations between etching depths and FWHMs of the (004)rocking curve.

PS-13.03.13 DOUBLE CRYSTAL X-RAY ROCKING CURVE ANALYSIS OF MULTIPLE EPITAXIAL LAYERS.

By Li Mei, Lin Chao Li, Zhang Zhi Shun and Ge Zhong Jiu, Changchun Institute of Physics, Academia Sinica, Changchun, 130021, China.

We have analysed the characteristics of GaAlAs/GaAs hetero-epitaxial structures by the X-ray double crystal rocking curve method. As an example, we have analysed a three-layer-epitaxial structure grown by LPE on a (001) GaAs substrate, where an active layer is sandwiched between two confining layers of GaAlAs (top layer and bottom layer, respectively). The active layer can be GaAs or a GaAlAs/GaAs (5*5) superlattice layer. We have measured the rocking curve of a 400 reflection and observed the interference fringes for the above two kinds of samples. Using kinematical theory, we have calculated the lattice mismatch of the aluminium component. Computer simulation of the experimental curves have been performed with kinematical and dynamical diffraction, respectively. We have discussed the reason for the appearance of the interference fringes, and have calculated the thickness of the different layers. We can come to the following conclusions; 1. For sample No 1, we observed the diffraction peaks of the two confining layers and the substrate. There is a shoulder on the left side of the substrate peak. In the simulated curve, two types of interference fringes can be seen. One of them is located between the diffraction peaks, and the fringe period is 15 second of arc. It is related to the thickness of the top layer. The other type of fringes is superposed on the left side of the substrate diffraction peak. Its period of 7 second of arc corresponds to a thickness of 2.7 μm. This is just the distance between the GaAs active layer and the substrate. This result implies that the interference fringes may be due to the interaction between the beams diffracted by the active layer and the substrate. 2. For sample No 2, the experimental and simulated curves are basically identical. There are fringes of period 26±1 seconds of arc. From the calculated result, it is determined that the interference fringes are not the Pendellösung fringes of the top layer. The interference fringes on both sides of the GaAlAs