

SYNCHROTRON RADIATION GRAZING INCIDENCE X-RAY DIFFRACTION: A NEW TOOL FOR STRUCTURAL INVESTIGATIONS OF ION-IMPLANTED GLASSES

F. D'ACAPITO AND F. ZONTONE

ESRF, EXPERIMENTS DIVISION

Composite systems consisting of metallic nanoparticles embedded in glass have recently attracted a great interest in the field of optoelectronics because of their non-linear optical behavior. The implantation process creates a thin (a few hundreds of Å) cluster-rich layer just about 1000 Å below the glass surface. A novel x-ray technique has been developed to carry out structural investigations of such systems. It is based on the signal enhancement from the surface layer with respect to the substrate contribution when working at the critical angle for total external reflection at the implanted layer-substrate interface. By using the refracted beam as a probe, the diffraction profile of the metallic clusters in very diluted samples (a single implant in the 10^{16} at/cm² range) can be extracted by a simple subtraction procedure. A demonstration of the new method on SiO₂ glasses implanted

with Ag ions is shown in Figure 1. The measurements were performed at the High Pressure station at the ID9 beamline. The samples were illuminated with a 10(h) x 24(v) μm² very intense monochromatic beam in the grazing incidence geometry with grazing angles ranging from 0.05 to 0.2°. The data were collected by using an imaging plate system in the q range 0.1 – 8.5 Å⁻¹ with integration times in the minute timescale. Curves A and B are the 2θ scattering patterns after integration of the 2D data at the grazing angles for total external reflection at the metal-rich layer-substrate and at the air-substrate interfaces (positions A and B in the reflectivity curve respectively). The profile A shows small diffraction peaks from crystalline phases of the metallic clusters emerging from the smooth amorphous SiO₂ background which disappear in the profile B. After the subtraction procedure ten main peaks are clearly visible in the

difference signal coming from the fcc phase of the Ag clusters. The data quality is high enough to enable crystalline phases identification, to retrieve lattice parameters and to determine clusters mean sizes. The results are very useful in complementing information from x-ray spectroscopic (EXAFS) methods and «single particle» investigations with electron techniques (TEM). ■

REFERENCES

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Fig. 1: Extraction of the cluster rich layer signal with the new grazing incidence x-ray method in the case of an Ag implanted SiO₂ glass: the diffraction peaks coming from the cluster are maximized at the total external reflection condition for the glass-clusters layer interface (A case). They are not visible in the profile B. In the difference profile ten main peaks (with indices) are clearly visible related to the fcc phase of the Ag clusters, the other coming from unknown phases. The insert shows the reflectivity curve.

