



ESRF
GILDA
CRG

Experiment title:

XRD STUDY OF METAL NANOCRYSTALS IN
CU, AG IMPLANTED SILICATE GLASSES.

Experiment

number:

HS 175

Beamline:

GILDA

Date of experiment:

from: 31/10/97 to: 2/11/97

Date of report:

26/02/98

Shifts:

9

Local contact(s): K.KNUDSEN

Received at ESRF:

02 MAR. 1998

Names and affiliations of applicants (* indicates experimentalists):

F.d'Acapito* CNR- Uff Sincrotroni Ple A.Moro 7 00195 Roma (Italia)

E.Cattaruzza*, F.Gonella* Univ. di Padova - Dip. di Fisica Via Marzolo 8 35131 Pdova (Italia)

J.R.Regnard*, CEA-Grenoble 17, Av. des Martyrs F-38054 Grenoble (France)

The aim of this experiment was to perform precise determination of the lattice parameter of clusters of Cu and Ag (better than previously performed EXAFS investigations). The samples proposed were Silica and Soda-lime glasses implanted with the cited ions at a dose ranging between $1 \cdot 10^{16}$ at/cm² and $5 \cdot 10^{16}$ at/cm².

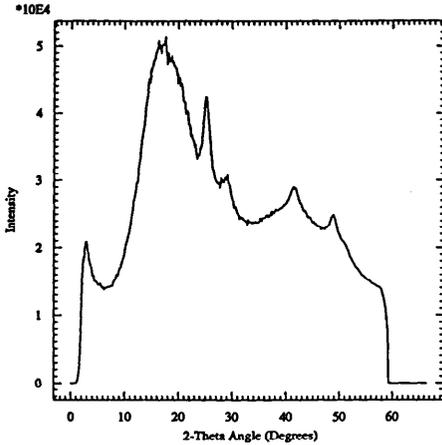
A previous proposal (HS126) was put in reserve time on ID09 and one day of test on that beamline was used to demonstrate the feasibility of the experiment. During that test it was realized that a careful alignment of the sample on the falling edge of the reflectivity curve was necessary in order to maximize the low cluster signal over the huge background contribution. The precise mechanics for sample positioning used on ID09 resulted to be perfectly suitable for this kind of experiments.

This experiment was performed on BM01 due to the lack of beamtime on the requested beamline ID09. Unfortunately this beamline, although disposing of a good MAR detector for 2-D diffraction spectra, is not equipped with precise mechanics for sample handling on the MAR table. Samples were aligned in an extremely rough method using an optical level and absolutely no signal was extracted from implanted glasses after a whole day of trying. Under this point of view the experiment has not been performed at all.

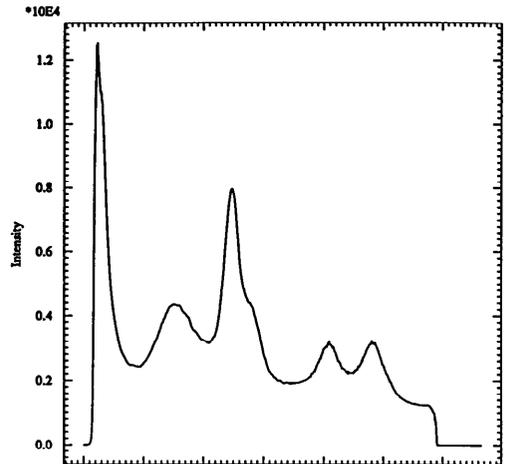
However we disposed also of samples doped with Cu and Ag by binary ion-exchange and subsequently implanted with He ions or exposed to Hz, atmosphere to promote the cluster

formation. In these samples the cluster rich zone extends down to several μm under the glass surface (in contrast with 1500 \AA in the case of implanted samples) and the quantity of metal in the glass is as high as **10-100** times that in the implanted samples. This means that we do not need the near-total reflection condition to evidence the cluster signal and a low incidence angle ($1-2^\circ$) is sufficient. Thus we spent the following two days measuring this kind of samples and some of the spectra collected are shown below.

Y7_1_013.image: 2-theta Scan



W1_1_003.image: 2-theta Scan



The noise is higher than that obtained on ID09 but the quality is sufficient to make a reliable data analysis. We are performing at the moment the data analysis.

We will submit again this proposal on the implanted samples asking this time explicitly the use of ID09 line that revealed to be the only one permitting this kind of measurements.