| $\overline{\mathrm{ESRF}}$ | Experiment title: Reconstruction of the 3d configuration of antiphases in an ordered alloy (AuAgZn2) from the configuration of the speckles at the superstructure peak | Experiment number: 02-02-192 |
|---|--|------------------------------------|
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| Shifts: 8 | Local contact(s): Dr. Frederic LIVET | Received at ESRF: |
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Report:

The D2AM goniometer was used for the first time in a speckle experiment in the vicinity of a Bragg scattering. For this purpose, some experimental conditions had to be fulfilled.

Brilliance: In order to select a coherent beam of a reasonable intensity, the higher brilliance in the vicinity of the sample position has to be obtained. This is achieved if the focusing optics provides the smallest image of the source as possible. In our case, this image can be as small as $30(v) \times 70(h)\mu m^2$ (FWHM). We have selected a small part of our optical setup (about $400\mu m$ size), where the focus spot was $38(v) \times 77(h)\mu m^2$. Its stability was better than $\pm 15\mu m$ within 24 hours.

Beam selection: For the observation of coherence effects, a small part of the beam has to be selected, so that the product of its size ϕ by its angular divergence ϵ is of the order of the wavelength of the X-ray beam ($\lambda = 1.57$ Å). The angular divergence was fixed to $\epsilon = 30\mu$ rad. The size of the beam was fixed by means of high precision slits, with carefully polished Tantalum edges /1/, in order to obtain beams in the micrometer range.

Detection: For the speckle detection, the same conditions have to be fulfilled for the angular acceptance of the detector. We have used a Direct Illumination CCD of $22\mu m$ resolution at a distance of 1.6m from the sample. The angular acceptance was $\epsilon' \simeq 14\mu$ rad.

Slit diffraction: Our setup was tested by means of the measurement of the diffraction of the slits. Figure 1 shows typical results obtained with $1\mu m$ and $2\mu m$ slits.



An excellent agreement with calculated intensities (see Ref /1/) is observed. This is an important test for the future of image reconstruction: the shape and the intensity of scattering corresponds to the propagation of an electronic plane wave through $1\mu m$ asymetric slits (the two opposite slit edges have a 1mm offset in order to avoid collisions).

Observation of sample diffraction: A map of the speckles has been observed in the ordering superstructure peak of the $AuAgZn_2$ monocrystalline sample.



Typical results are shown in figure 2. In this figure, we compare different apertures $(10, 7, 5 \text{ and } 4\mu m)$ for the same sample region. We observe a similar speckle structure, and it is the most peaked for the smaller aperture (better coherence).

Discussion: Figure 2 shows that the observed speckle structure is elongated horizontally. This correponds to the large superficial mosaic of this sample, and the contribution of antiphases appears small, in the vertical direction. Due to sample preparation, a surface stretch was created during quench, leading to relatively large surface mosaic. We have estimated the degree of coherence of our setup from the speckle contast, like in Fig. 2. It appears in all cases too small for carrying out any phase reconstruction. For these two reasons, the experiment cannot provide a map of the sample antiphase configuration. We nevertheless have obtained a stable coherent beam. From wave propagation calculations, we are now able to have a-priori estimates of the degree of coherence, and to modify the characteristics of the beam in order to improve it. A better choice of the sample seems nevertheless necessary.

Reference: D. Le Bolloc'h, F. Livet, F. Bley, T. Schulli, M. Veron and T. H. Metzger, J. Synchr. Rad. (2002), 9, p. 258-265 ESRF Experiment Report Form July 1999