



	Experiment title: Strain effects in order-disorder fluctuations	Experiment number: HS-1849
Beamline: ID01	Date of experiment: from: 13.11.02 to: 20.11.02	Date of report: 25.08.03
Shifts: 18	Local contact(s): <b>Dr. Olivier Plantevin</b>	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists):  Harald Reichert*, Ingo Ramsteiner*, Cristian Mocuta*  Max-Planck-Institut fuer Metallforschung Heisenbergstr. 1 D-70569 Stuttgart Germany		

## Report:

A focal point in the research of our group is the short range order of disordered binary alloys. For the last decades, the most powerful experimental approach to this subject has been the diffuse scattering of X-rays and neutrons. This technique yields the pair correlation function of the system and thereby allows to access the effective pair interaction parameters. In the past we have conducted several diffuse short-range order scattering experiments at various synchrotrons on a wide selection of alloy systems (e.g. Cu-Mn [1]) using high energy X-rays and 2D detectors. For the interpretation of our data we have developed novel methods based on a reciprocal space fluctuation wave formalism [2].

Previous results of this work indicate a major influence of strain effects on the behaviour and coupling of the fluctuation waves. In conventional diffuse scattering experiments, however, the data represents an average over various local configurations due to only partial coherence of a standard synchrotron beam. This does not allow to observe coupled concentration wave fluctuations. Thus, we proposed an X-ray intensity fluctuation spectroscopy (XIFS) experiment to perform a time resolved measurement of the speckle pattern (using a fully coherent beam) of a Cu<sub>3</sub>Pd sample .

The first beamtime in the context of this project [3] proved the excellent coherence qualities of beamline ID01, but suffered from sample and instrumental problems. Encouraged by the promising beam characterization we modified our setup and refined the sample preparation for the second beamtime, which is accounted for in this report. An energy of 8keV was chosen. Coherent experiments demand low X-ray energies, as the transverse and longitudinal coherence are proportional to  $\lambda$  and  $\lambda^2$  respectively. With respect to even lower energies the wavelength choice is limited by the detection efficiency of the vacuum compatible Princeton CCD available at ID01, which peaks at 10 keV, and the penetration depth into the sample, which was mounted in transmission geometry. The main challenge in sample preparation was

to produce a single crystal with a thickness of about 1  $\mu\text{m}$ . To this end electrochemical thinning methods (also used for transmission electron microscopy) have been employed and produced samples of good quality, as verified during the beamtime. The diffracted signal was detected with the Princeton CCD mentioned above, which was mounted on the detector arm of the in-vacuum diffractometer of ID01. In the transmitted primary beam downstream of the sample a vacuum compatible cyberstar detector was mounted as a monitor counter. It was equipped with a set of high precision slits, so the coherence could be checked anytime by pulling the sample out of the beam and scanning the Fraunhofer pattern from the beam defining aperture with the offset translation of the detector slits. At ID01 the complete diffractometer sits in a vacuum vessel, which eliminates air scattering and absorption. Two differential pump stages compensate the difference in pressure between the diffractometer vessel and the ring vacuum, allowing to remove all windows between sample and undulator. As verified in the first experiment the coherence at ID01 is excellent (20  $\mu\text{m}$  transverse coherence and more) and would allow a beam of corresponding diameter. The size of the illuminated spot, however, is additionally limited by the speckle size  $s_{\text{Speckle}} = (\lambda * \text{distance}) / \text{spotsize}$ . The size of individual speckles on the detector should not be smaller than the pixels of the CCD (55  $\mu\text{m}$ ), otherwise the speckles are being averaged by the detector. The wavelength cannot be increased further due to reasons explained above, and the sample-detector distance is limited by the dimensions of the vacuum vessel. The only free parameter left is the spot size, which has then to be decreased to less than 2  $\mu\text{m}$ . Decreasing the pinhole size to this value cuts the flux on the sample by orders of magnitude. The tight restrictions on beam size combined with the tiny scattering volume led to a diffraction intensity, which was smaller than expected from pre-experimental estimations. Unfortunately, it was not sufficient to record a short-range order speckle pattern. We still believe the experiment to be a fascinating application for coherent synchrotron radiation. With today's synchrotron sources, however, it is not feasible yet.

- [1] H. Reichert, V.N. Bugaev, O. Shchyglo, A. Schöps, Y. Sikula, H. Dosch, Strain-induced nonanalytic short-range order in the spin glass  $\text{Cu}_{83}\text{Mn}_{17}$ , *Phys. Rev. Lett.* **87**, 236105 (2001).
- [2] V.N. Bugaev, H. Reichert, O. Shchyglo, A. Udyansky, Y. Sikula, H. Dosch, q-space configurational energy and short-range order in alloys with atomic size mismatch, *Phys. Rev. B* **65**, 180203, (2002).
- [3] H. Reichert, I. Ramsteiner, M. Denk, Strain effects in order-disorder fluctuations, Experimental report HS-1549 (2002).