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Report:

In this experiment, we wanted to measure “in situ” the dynamics of the thermal fluctuations in an ordering alloy close to the critical point. The system chosen is an “Heussler” ordering alloy, and all measurements have been carried out close to the (1/2 1/2 1/2) superstructure peak.

Experimental methods.

The sample was heated in a very precise and stable furnace (+/-.001K). T_c is estimated from the decrease of the Bragg intensity close to the critical point (Tc=619.45K for the sample studied).

Monocbromatization of the beam was ensured by a Si(111) monocbromator ($\delta\lambda/\lambda=1.4\,10^{-4}$).

In order to obtain a (partially) coherent beam, slits have been carefully aligned. The horizontal width of the source was limited to 200µm by closing primary slits. A curved SiC mirror focused the beam in the vertical direction. Such setup and alignment are necessary because of the very anisotropic geometry of the source ($800*50\mu m^2$). In this case, reasonable coherence is achieved, without loosing too much intensity, with a 8µm diameter Gold pinhole placed 20 cm before the sample.

CCD “deep depletion” 2d detector.

As the diffuse intensity scattered is small, a direct illumination “deep depletion” 2d (584*376 pixels, 22µm*22µm each) CCD detector was used for the first time. Much of the experimental time was spent in testing, characterizing and programming this new detector.

We have checked the excellent spatial (25µm FWHM) resolution of the detector. About 30% of the 8Kev X-rays are detected. For low counting rates, or for frequent readings of the detector, individual absorbed X-rays can be well identified. In our case, this provides a very good method for noise suppression: if the intensity is less than .l X-ray per pixel per second, long measurements and large amount of pixels can compensate for low intensity if noise can be suppressed. For this pupose, frequent readings of the detector are necessary. In order to save memory space, only the pixels larger than three times the average noise have been recorded.

We have then used a “droplet algorithm”, where every x-ray is identified on the CCD detector. This method isolates the small regions (droplets) where electronic charges have been detected. We have shown that the number of x-rays absorbed in each region can be estimated (if less than 10), so that individual x-rays can be located with a high spatial resolution. This method will strongly increase the efficiency of further measurements.

The static intensity can be obtained with very good dynamics (down to less than 10^{-4} X-rays/pixel/set.).

Results.

The scattered intensity is recorded on the 2d CCD detector, centered on the superstructure peak. A typical result is given on Fig. 1. In the central part, a nearly stable speckle structure is observed. This gives an estimate of the degree of coherence of the measurement: about 20% of mean square deviation (a +/-45% intensity variation). This central structure is elongated perpendicular to the [1/2 1/2 1/2] oriented surface, and this is explained by some Zn concentration gradient due to Zn oxide formation and Zn evaporation, which gives surface ordering.

The scattering of critical fluctuations dominates in the low-intensity regions of the detector, as this is suggested by the isotropic q^{-2} behaviour observed.

From time series, photon counting correlations can be estimated. Typical results are given on Fig.2. Close to the Bragg peak, intensity variations are roughly periodic, and this can be related to the observed variations of the monitor. They are explained by small sample and pinhole movements (a few micrometers) in the irregular structure of the incoming beam, due to speckle scattering from unpolished Berylium windows. The main results for critical fluctuations are obtained in the low intensity part of spectra, and the correlations have to be estimated from long time measurements and by averaging correlations over a large number of pixels (about 30000 for Fig. 2-b), which is only possible after the droplet treatment.

We are writing a paper about the directly illuminated “deep depletion” CCD detectors, and, from the data analysis already performed, as well as from other experiments on this system, improvements in the quality of results can be obtained from new experiments.

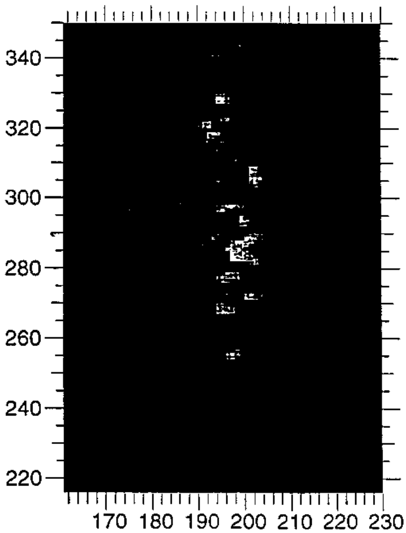

Fig. 1: The speckle structure in the central part of the detector.

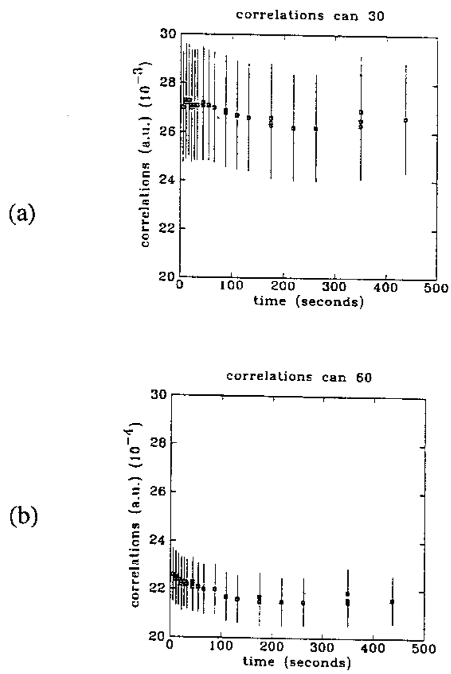

Fig. 2: Intensity correlations observed (a) -In the central part of the detector. (b) -At larger angles.