ESRF	**Experiment title:** TIME RESOLVED CRITICAL FLUCTUATIONS IN $FE_3AL(110)$	**Experiment number**: HS - 1483
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Report:

The aim of this experiment was to study the intensity fluctuations related to critical fluctuations in ordered metallic alloys, in particular Fe_3Al (body centred cubic structure), which exhibits an order-disorder phase transition (DO_3-B_2) at about T_C=780 K. The sample ($Fe_{71.5}Al_{28.5}$ single crystal) was prepared and pre-characterized at the Max-Planck-Institut in Stuttgart before the experiment. The idea was to investigate a region in which the relative number of fluctuating domains of a certain size ($\delta N/\langle N\rangle$) is large. This is achieved by increasing the maximum size of the fluctuating domains (by approaching T_C) and reducing the investigated sample volume (thus $\langle N\rangle$). We used a well focused beam (micron size) to probe a very thin layer (sub-micron). The investigated area on the sample is located on the rim of a hole chemically drilled into the sample (similar preparation than for electron microscopy measurements). Using a HR-TEM electron microscope, a uniform thickness and composition (±0.5%) were found on different (10 investigated) spots of the rim (see **figure 1**).

The crystal was pre-aligned before the experiment and mounted into an UHV chamber (Be windows, base pressure in the 10^{-10} mbar range). During the whole experiment mainly the superstructure (113) reflection (characterizing the chemical order) was measured at different temperatures.

Using Compound Refractive Lenses (CRLs) a beam size of 1.5 × 1.7 μm^2 FWHM (horiz. × vertic.) was achieved. Hole profiles were obtained by simultaneously measuring the intensity of a lattice diffraction peak (002) and the transmission through the sample, while scanning the sample horizontal and vertical position (**figure 1**). It was possible to retrieve and fit the thickness on the hole profile, leading to an estimation of the thickness of the investigated area of about 1-1.5 μm limited only by the incident flux.

By reducing the probed volume (1.5×1.7×1.5 = 3.8 μm^3) upon approaching T_C the number of (fluctuating) ordered domains on the length scale of the correlation length ξ is small. Due to the decrease of the number density of these domains, pronounced effects on the measured intensity of a superstructure peak are expected. During the experiment the temperature of the sample was controlled within 80 mK, by using stabilized current power supply for the heater. Note that no feedback was used in this process, avoiding thus oscillations in temperature which could induce oscillations in the measured intensity, mainly near T_c.

The very good crystalline quality of the sample (typical 0.02-0.05° both for lattice and superstructure peak) allows for very large coherently ordered domains (thousands of Å). Thus, the resolution is not limited by the detector slits (set to an acceptance of ~ 0.1°), but by sample mosaicity. At each temperature radial and transverse (rocking) scans were performed as well as time dependent measurements at the maximum intensity of the peak. At the end of each time scan, the alignment of the sample was checked; since no shifts in the peak position were found, the intensity fluctuations measured cannot be attributed to sample misalignment; moreover, the time-scales involved in the fluctuations (~ 100 seconds) are too short for drifting.

Figure 2a shows the measured (integrated) intensity of the (113) superstructure peak at different sample temperatures. Time-scans performed at four different temperatures are shown in **figure 2b**. It is clear that only near T_c strong intensity fluctuations appear (maximum amplitude value). Moreover, by calculating the temporal intensity – intensity correlation function $g^{(2)}(\tau)$-1, it is only near T_c that its values are significantly non-zero.

This experiment was a full success and has shown the possibility to study critical phase transition / fluctuation phenomena using ***partially coherent*** X-rays. Thus, it was not limited by the use of specific sample environment such as Be window or low coherent flux generated by the use of pin-holes as secondary sources. This has been achieved by reducing the probed volume of the sample, using a focused beam and thin high quality crystals. The control of the sample temperature is crucial in evidencing large amplitudes in the fluctuations. A more detailed statistical analysis of the data is currently under way including the development of a theoretical treatment of intensity fluctuations originating from partially coherent beams.

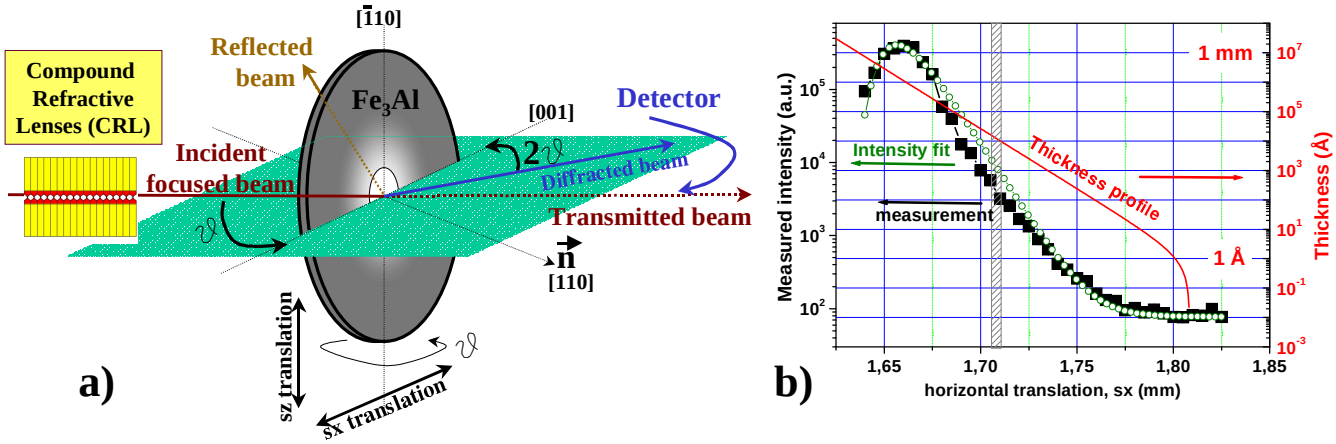

Figure 1: a) sample geometry for the diffraction experiment; b) hole profile obtained by scanning the sample position at the lattice peak (002) position. The edge position is sx=1.8 mm. The hatched area (around sx=1.705 mm) indicates the measured position at the rim of the hole.

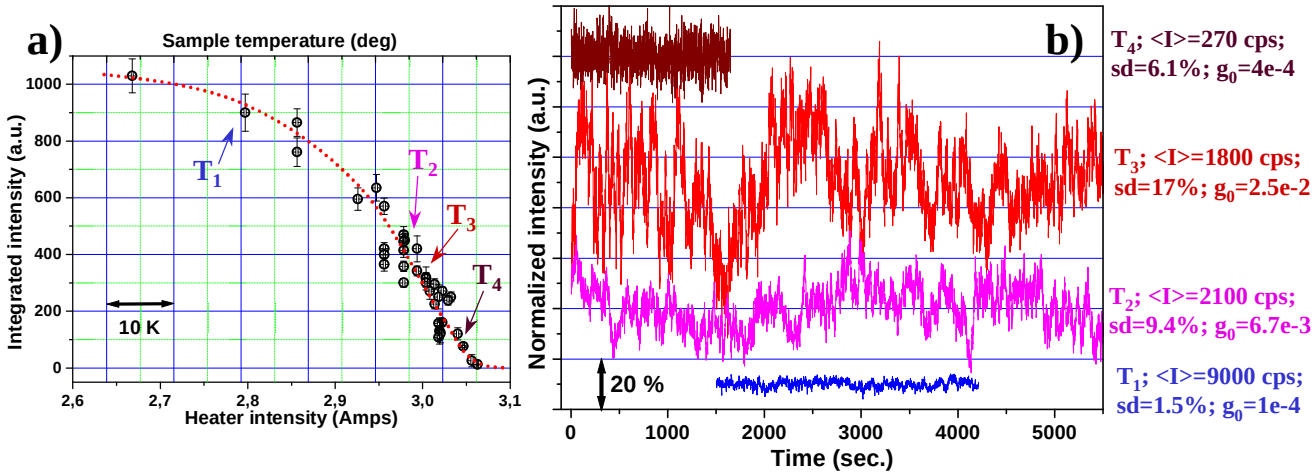

Figure 2: a) integrated intensity of the superstructure (113) peak measured at different temperatures near T_c. The dotted line is just a guide for the eye. For the temperatures denoted by T_1 to T_4, timescans are shown in figure b), using the same relative scale. The average intensity was normalized to 1. The values of the average intensity (<I> in counts/s), standard deviation (sd), and $g^{(2)}(\tau \rightarrow 0)$-1 (denoted by g_0) are shown.