ESRF	**Experiment title:** Time resolved critical fluctuations in $Fe_3Al(110)$	**Experiment number**: HS 1776
Beamline: ID22	**Date of experiment**: from: 3 July 2002 to: 9 July 2002	**Date of report**: 16 feb. 2003
Shifts: 18	**Local contact(s)**: Dr. M.Drakopoulos	*Received at ESRF:*

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Report (related to exp.report MI 564):

We report on studies of the critical fluctuations in Fe_3Al (the DO_3 – B2 transition) using a Kirkpatrik-Baez (KB) focusing optics. The KB optics provides a large gain compared to CRL systems (see exp.reports **MI 564** and **HS-1483**).

After moving the KB device from BM5 to ID22, unfortunately, mechanical problems with one of the motors of one mirror delayed us in reaching the minimal (focused) spot size (3 shifts). Since the experimental conditions at ID22 and BM5 are different, stability tests of the KB system were mandatory (3 shifts). The results were totally satisfactory: the obtained spot size (measured by knife-edge scans) on the ID22 beamline was of $1.3 \times 1.4\ \mu m^2$ (**figure 1**) with a good stability (position and intensity) over long periods of time (exp. report **MI 564**).

A new temperature controller was used, allowing to stabilize the temperature of the sample within ±5 mK or better at temperatures around 500ºC. During the measurements, especially near the critical temperature (T_C), spurious jumps in the temperature reading appeared. These spurious jumps were coupled into the controller via spurious noise in the power system of the ID22 hutch. In order to ensure that the feedback of the temperature controller would not induce intensity fluctuations – although they are easily detected – the measurements were performed without feedback loops, while the temperature was carefully monitored. We proved thus that the observed intensity fluctuations are an intrinsic property of the system in the vicinity of T_C (**figure 2**)

At each temperature, mesh scans close to the rim of the circular hole in the sample were performed (**figure 3**). The absorption of the X-rays in the sample was also recorded in parallel, allowing to find and fix the illuminated area (arrows in **figure 3**) for a sample thickness of 2.3 μm. For each temperature long time series were recorded at the Fe_3Al (113) superstructure reflection to evidence the presence of intensity fluctuations only close to T_C (**figure 2**). The sample temperature was carefully monitored and shows no temperature fluctuations large enough to explain the intensity fluctuations.

Note that there is a change in the FWHM of the rocking curve at T_C from 0.15º to values bigger than 1º and an increase of the amplitude of the intensity fluctuations up to 20% or more. The fluctuations are sensibly larger than the corresponding noise level deduced from Poisson statistics which is smaller than 5%. We also mention here that the line shape of the rocking curves changes from Gaussian below T_C to Lorentzian above T_C. This proves once more the critical nature of this transition.

In order to test a thickness dependence of T_C we performed mesh scans in the transverse direction at different temperatures close to T_C. The measurements indicate a small thickness dependence of T_C, but we cannot exclude small temperature gradients in direction radial to the rim at the moment.

We have characterized both order disorder phase transitions DO_3 – B2 (T_C = 468.5ºC) and B2 – A2 (T_C = 651ºC). The temperature dependence of the long range order parameter for the DO_3 – B2 implies that we are close to a tricritical point in the phase diagram. From this we conclude that the composition x of our $Fe_{75+x}Al_{25-x}$ sample was 0 x

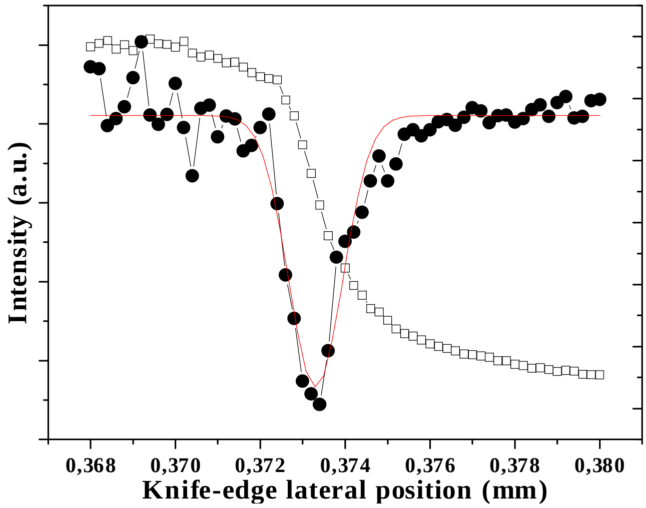

Figure 1: Spot size measured on ID22: transmitted intensity ( ) and its derivative (•) *vs.* the position of the knife edge (in mm). The continuos line is the fit, with a FWHM = 1.27 µm.

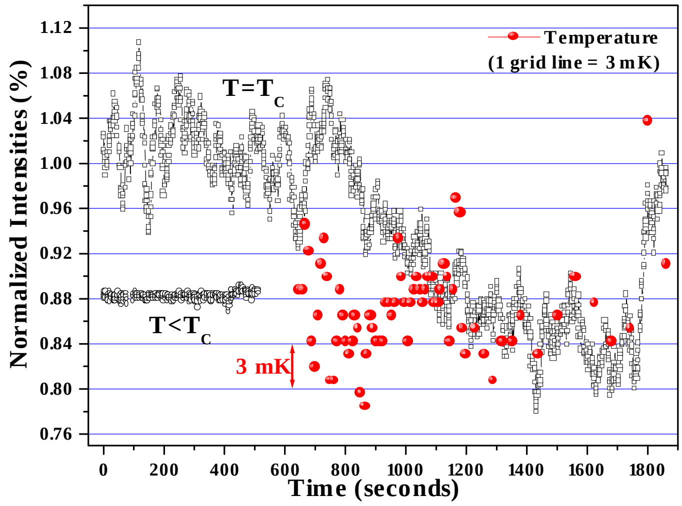

Figure 2: Time resolved diffracted signal at the Fe_3Al (113) superstructure peak, for the sample maintained close to T_C ( ) and far below T_C (O), respectively. The temperature reading close to T_C is also shown (•) on a vertical grid scale of 3 mK. No large enough temperature fluctuations are monitored to explain the intensity fluctuations.

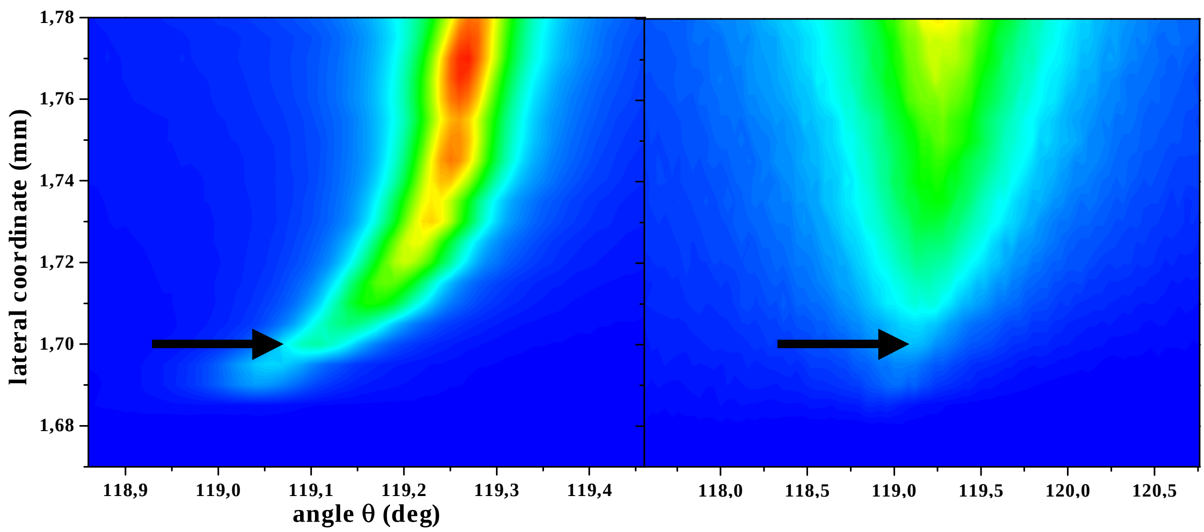

Figure 3: Mesh scans at two different temperatures, slightly below (left) and above (right) the critical temperature. Same color scale is used, but the right intensity was multiplied by 10.

Ref: C.Mocuta, H.Reichert, M.Krech, M.Drakoupoulos, M.Denk, H.Dosch, *Critical fluctuations in Fe_3Al observed by X-ray incoherent micorbeam* (*in preparation* for Phys.Rev.Lett.)