ESRF	**Experiment title:** "In situ study of the ordering transition B2-Heussler at the critical point"	**Experiment number:** 02-02-66
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Shifts: 9	**Local contact(s):** F. Bley	*Received at ESRF:*

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Report:

In this experiment, a careful high resolution study of the region close to the (1/2 1/2 1/2) superstructure peak position corresponding to the Heussler transition of the $AuAgZn_2$ alloy had to be carried out.

For that purpose, the new direct illumination CCD camera of 22.5μm resolution was used. Its images were exploited directly by the "droplet" method, where the impact of X-rays on the CCD is isolated and memorized. This method was developped originally for very low counting statistics, in order to suppress the noise of the CCD cameras. In the case of Bragg peaks enlarged by finite domain size, this method is also very efficient for the observation of a large dynamics of intensity (at least a factor of 10^4).

The shape of the beam on the optics was roughly a square, of size between .8 and 3mm, depending on the resolution needed, and the dimensions of the beam at the sample were 130(h)*80(v)μm2.

The Heussler transition was close to 350°C, and the "in-situ" furnace was regulated within +/-.01°C. Two main features can be distinguished in the results of this experiment:

1-Above and very close to T_c.

The critical scattering has been observed with a very good precision and with high accuracy and a large dynamics. For the sample-detector distance of .4m, and correlation lengths (ξ) varying from 30 to 500Å could easily be observed. Fig. 1 shows an incomplete set of results, where the critical scattering is plotted versus the scattering vector, for temperatures varying between 353°C and 351.1°C (T_c+.08 to T_c+2). From our results, we can verify that all static characteristics (ξ, and also I_0 or χ) have the critical exponents of the Ising model. For instance, Fig.2 is a plot of the maximum intensity (I_0) observed versus the temperature.

According to the Ising model, I_0 should diverge as $(T-T_c)^{\gamma}$, with $\gamma=1.241$: $I_0^{-1/1.241}$ is linear versus temperature, and one can deduce $T_c = 351.02(1)°C$.

This verification of Ising properties in such a large temperature domain is very important. In ordering systems, the elastic deformation-ordering coupling can lead to non-Ising behaviour or to first order phase transition. Moreover, a strong streak (parallel to the scattering vector, but also perpendicular to the surface) was observed in previous experiments close to T_c. This latter was sometimes interpreted as a second fluctuation distance (analog to the central peak in ferroelectic transitions). Here, the sample was coated with a 70Å thick layer of Al, and this scattering had disappeared. This shows that it is simply surface contamination.

2-The dynamics of ordering.

The dynamics of the transition was observed by quenching the sample. The distance from sample to detector was 1.3m, and domain sizes of .2mm could easily be observed.

The system has a fast evolution close to T_c, and this makes the observation of large ξ and sharp second order transition possible: a slower system should have an apparently "rounded" transition. Close to T_c, we have observed the domain (D) size increase. D is close to experimental resolution after a few minutes. The interpretation of the results is under way.

Only two remarks are necessary here:

1)-In the area detector, resolution in the direction perpendicular to the scattering vector is lowered by the scattering of a few mosaic domains, observed for long ageing times (and large D). This can be eliminated by reducing the surface irradiated by closing slits in order to reduce the beam size to the dimensions of a mosaic grain (20-30mm) :in these measurements, filters are necessary when using the area detector, and the intensity can be reduced.

(2)-For the study of slower dynamics, the sample has been quenched at "low" temperatures (T_c-100K). After this type of quench has been done, some "streak" scattering has again been observed. It is interpreted as a decohesion of the surface layer of Al (transformed into Al_2O_3 at this temperature) due to expansion differences between alumina and the alloy. For this reason, deep quenches should be avoided.

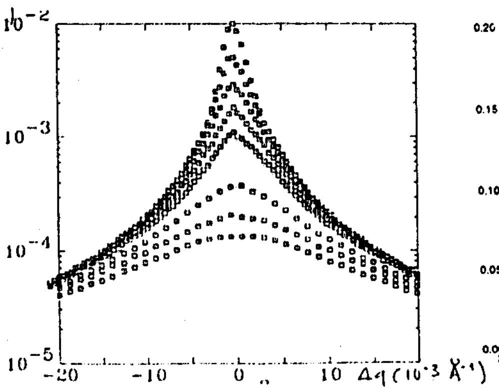

Fig1-The critical scattering at various temperatures.

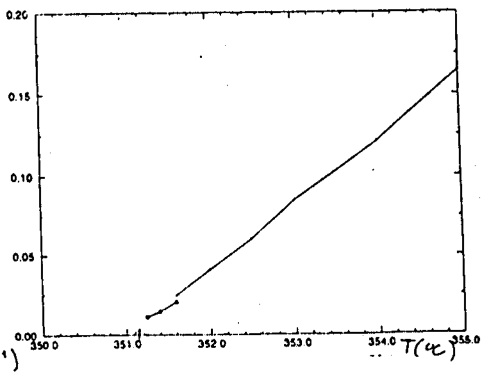

Fig2. Plot of $I_0^{-1/1.241}$ vs T showing Ising behaviour.