ESRF	**Experiment title:** Atomic structure of the ZnMgSc and ZnAgSc icosahedral phases	**Experiment number**: **02 02 608**
Beamline: D2AM	**Date of experiment**: from: 28 January to: 3 February 2004	**Date of report**: 25 Aug 2004
Shifts: 18	**Local contact(s)**: M. de Boissieu	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

T. Ishimasa [(1)], Y. Kaneko [(1)], S. Kashimoto [(1),*], H. Takakura [(2),(3)]*, A. P. Tsai [(4),(5)], M. de Boissieu [(6),]*, S. Francoual [(6),]*

(1) Division of Applied Physics, Graduate school of Engineering, Hokkaido University, Kita-ku, Sapporo 060-8628, Japan
(2) Advanced Materials Laboratory, NIMS, Tsukuba 305-0044, Japan
(3) Research Center for Molecular Thermodynamics, Osaka University, Japan
(4) Materials Engineering Laboratory, NIMS, Tsukuba 305-0047, Japan
(5) Institute of Multidisciplinary Research for Advanced Materials, Sendai, Japan
(6) LTPCM, UMR CNRS 5614, BP 75, 38402 St Martin d'Hères Cedex, France

Report:

We have studied the atomic structure of the $Zn_{80}Mg_5Sc_{15}$ icosahedral phase. This report is following the previous report (previous experiment carried out in July 2003) on which the introduction should be read.

Experimental details:

The experiment has been carried out on the D2AM beamline using the 7-circle diffractometer. We worked at an energy of 9.653 keV, 6eV bellow the Zn-edge to get some insight on the Zn-Sc chemical order. At this energy the f'(Zn) value is equal to –7e to be compared to -3e at 9.3 keV for the previous measurement, i.e there is a contrast of about 4 electrons. We used the same polished i-ZnMgSc single-grain sample [1] and we measured in the theta-scan mode a same set of about 1600 reflections with $Q_{//}$ ranging between 0 and 8 rlu and $Q_{\perp}$ between 0 and 3 rlu. The beam stability was checked by measuring regularly the integrated intensity of a standard reflection, the observed fluctuations being the order of 1%.

The structure analysis will be carried out using the high-dimension approach and a phase retrieving procedure [2]. The work is under progress but fig. 1 illustrates already the good contrast achieved using both data sets. As can been seen in fig.1.a, where the integrated intensity is plotted for a few reflections at that two energies, there are strong relative variations what will put a severe constraint when refining the Zn-Sc chemical order. Fig.1.b is a plot on a logarithmic scale of the relative comparison between both data sets: the measured intensities span almost on 7 orders of magnitude what is a particularly important point since weak reflections are most sensitive to the details of the structure. The contrast variation is also clearly visible in this figure where there is a large spreading of the data around the 'no contrast' black line.

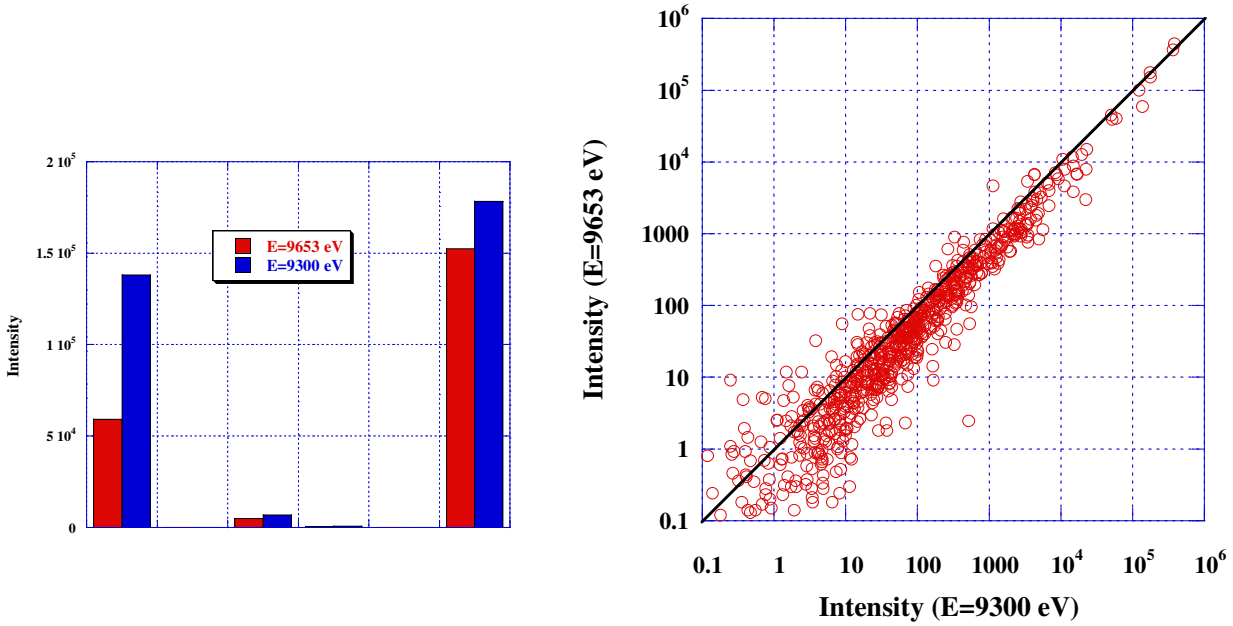
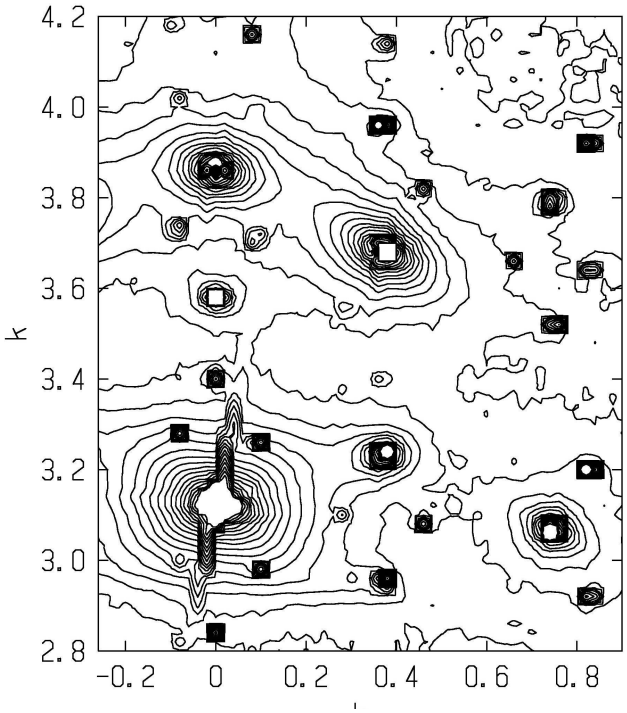


Fig. 1. a-(left): Comparison of the integrated intensities at the two energies for a few reflections; b-(right) : Log-Log plot of the intensity distribution measured at the 2 energies. If there was no contrast variation, all points would lie on the straight black line.

Most of the data have been recorded in a single assymetric unit. For a few reflections, we could measure the accessible equivalent ones (because we were working in reflection geometry, only reflections with an angle χ between 90 and 45° were selected). As a general trend, it seems that whereas strong reflections with equivalent positions have equal integrated intensities, there are significant intensity variations for weak reflections. This is most likely due to multiple scattering effects, a problem which is enhanced by the high density of Bragg reflections in this phase.



Finally, we have performed complementary measurements in the ZnAgSc icosahedral phase. This phase is supposed to be almost isostructural with the ZnMgSc phase [3], although the Ag content is slightly larger than the Mg one. This complementary data set should allow to locate the Ag or Mg atoms in the structure. We could check the structural quality of this phase and measure, in particular, the diffuse scattering around several Bragg reflections. As shown in fig. 2, a significant amount of anisotropic diffuse scattering intensity has been observed, most likely related to phason fluctuations.

Fig 2 : Diffuse scattering intensity map measured in the ZnAgSc icosahedral phase

References

[1] Y. Kaneko, Y. Arichika, T. Ishimasa, Phil. Mag. Lett , 2001, 81, 777
[2] H. Takakura, A. Yamamoto, M. de Boissieu, A.P. Tsai Ferroelectrics, 2003, in press
[3] S. Kashimoto, R. Maezawa, Y. Kasano, T. Mitani, T. Ishimasa, Jap. J. of Appl. Phys, 2003, 42, L1268