ESRF	**Experiment title:** Structural investigations on levitated undercooled liquid metallic alloys using x-ray diffraction	**Experiment number**: HS/2548
Beamline: ID15B	**Date of experiment**: from: 26/01/2005 to: 01/02/2005	**Date of report**: 24/02/2005
Shifts: 18	**Local contact(s)**: Guillaume GEANDIER	

Names and affiliations of applicants (* indicates experimentalists):

Prof. Dr. Ivan EGRY (DLR Cologne)*, Dr. Gerhard MATHIAK (DLR Cologne)*, Dr. Jürgen BRILLO (DLR Cologne)*,

Dr. Dominique THIAUDIERE (Synchrotron Soleil, Gif-sur-Yvette)*

Prof. Dr. David Price (CNRS-CRHMT, Orléans and National Laboratory Oak Ridge), Dr. Louis HENNET (CNRS-CRMHT, Orléans)*, Dr. Irina POZDNYAKOVA (CNRS-CRMHT, Orléans)*, Dr. Didier Zanghi (CNRS-CRMHT, Orléans)*, PhD Student Aleksei BYTCHKOV (CNRS-CRMHT, Orléans)*

Report:

The experiments reported here have been performed in the framework of a CNRS-DFG project involving the DLR in Cologne and the CRMHT in Orleans. The two laboratories are specialised respectively in electromagnetic [Herlach] and aerodynamic levitation techniques [Hennet] and they have joined their technologies to develop a new hybrid system combining inductive RF heating with aerodynamic levitation. This new technique offers the advantages of the two approaches previously used: high sample stability, homogeneous heating and good temperature control enabling easy access to the undercooled state of the sample.

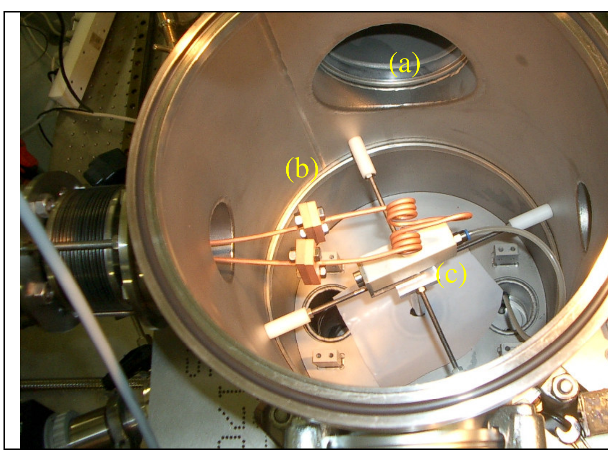

Figure 1. Hybrid system mounted at the ID15 beamline. Incoming x-ray beam (a), RF heating coil (b), Boron Nitride levitator with the spherical sample at the top (c).

A levitation chamber has been developed. This new device is shown in figure 1. Heating coil and levitation nozzle are housed in a vacuum chamber. A spherical sample (diameter=3mm) was levitated using a gas flow coming from a BN nozzle located in the center of the chamber. It was heated to the desired

temperature by means of the RF coil. In order to avoid oxidation, the chamber was initially pumped down to low pressure and then filled with argon (with 2.5% H_2).
During these experiments, we have performed High Energy (90keV) X-Ray diffraction measurements of liquid Al-based alloys Al-Cu, Al-Fe, Al-Ni, Al-Ti and in the system Cu-Ni using an image plate detector. Figure 2 shows S(Q) and g(r) from our preliminary data analysis for pure Al and a eutectic Al-Cu alloy. The quality of the data is remarkable and will allow us to determine atomic distances and coordination numbers.
All these alloys exhibit both, eutectic and intermetallic solid phases. It is one aim of our studies to find out whether traces of these solid phases persist in the liquid phase, i.e. whether metastable clusters of intermetallic composition exist in the melt. In general, the presence of a pre-peak in the structure factor corresponds to compound forming behaviour [Price].

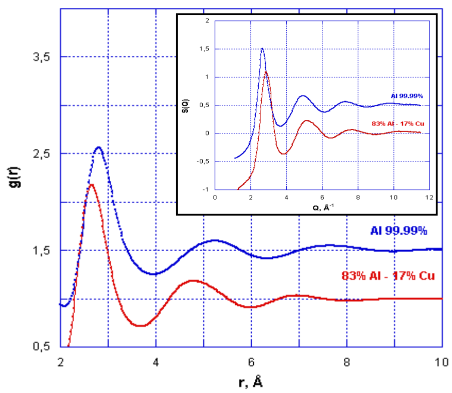

Figure 2. Pair correlation function g(r) of liquid Al of high purity (T=839°C) and Al-Cu of eutectic composition (T=831°C). The first peak at 2.66Å of the Al-Cu alloy corresponds to a combination of Al-Al, Al-Cu and Cu-Cu pair correlations. The insert shows the measured structure factor S(Q) of both alloys. The data processing procedure is described in [Hennet].

A) Al-rich Al-Ni alloys: Measurements were performed on samples with 2.7% and 25% of Ni. The XRD structure factor of Al-25%Ni showed a strong pre-peak, contrarily to the eutectic composition.
B) Al-rich Al-Fe alloys: This system presents some interesting properties such as magnetic ordering due to the presence of Fe clusters. Measurements were performed on samples with 4.2% and 7.5% of Fe. No pre-peak existed in the structure factor as expected for Fe contents less than 8% [Jingyu].
C) Al-rich Al-Cu alloys: Measurements were performed on samples with 17.1% and 33% of Cu. No pre-peak in the structure factor was detected either at the eutectic or at the intermetallic composition.
D) Al-rich Al-Ti alloys: The introduction of few percent Ti in Al has a strong effect on the transport properties in the liquid [Auchet]. Two compositions at 0.5% and 1% of Ti were studied.
E) Cu rich Cu-Ni alloys: There are indications that the Ni atoms exhibit a tendency to form associates [Brillo]. Measurements with electromagnetic levitation with samples of 20% Ni showed no pre-peak.
In the future, measurements with EXAFS shall be performed to investigate more precisely the short-range chemical ordering in these metallic binary alloys. We want to study systematically how the local environment around the absorber changes by going from eutectic to intermetallic composition within the same alloy and by substituting the alloying element. All systems will be measured at different temperatures, including the undercooled regime, where ordering effects are expected to be more pronounced. In combination with the data from the diffraction experiments, we hope to shed some light on the question whether clusters of intermetallic composition exist in the melt.

References

[Herlach] D.M. Herlach, R.F. Cochrane, I. Egry, H.J. Fecht, A.L. Greer: Int. Mat. Rev. 38 (1993) 273-347
[Hennet] L. Hennet, D. Thiaudière, M. Gailhanou, C. Landron, J. P. Coutures and D. L. Price, Rev. Sci. Instrum. 73, (2002) 125.
[Price] D L Price , S.C. Moss, R. Reijers, M.L. Saboungi, S. Susman, J. Phys.: Condens. Matter 1 (1989) 1005-1008.
[Jingyu] Q. Jingyu, B, Xiufang, S. Sliusarenko, W. Weimin: J. Phys.: Condens. Matter 10 (1998) 1211.
[Auchet] J. Auchet, E Terzieff, Journal of Alloys and Compounds 261 (1997) 295-298
[Brillo] J. Brillo, I. Egry, Z. Metallkd. 95 (2004) 691-697.