EXPERIMENTAL REPORT
RAPPORT D'EXPERIENCE

Programme Committee Proposal Number
N° Projet Comité de Programme

02-02 183

PROJECT TITLE : *TITRE DU PROJET :*

Absorption and anomalous x-ray scattering on Zr-Ni alloys at high temperature

LIGNE :	**D2AM**		**I F**	
INSTRUMENT :	PETITS ANGLES	☐	EXAFS	☐
	7 CERCLES	X	G M	☐
	F I P	☐	S U V	☐

NUMBER OF RUNS USED
NOMBRE DE SESSIONS EFFECTUEES : **21**

STARTING DATE
DATE DE DEMARRAGE : **May, 15 2002**

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Different compositions in the Zr-Ni phase diagram have been studied using x-ray absorption and x-ray diffraction near the zirconium absorption edge (17990eV). The melting point was achieved using aerodynamic levitation and laser heating. The analysis chamber, designed for working on the 7-circle goniometer of the D2AM station, enables combined x-ray absorption and diffraction measurements. The first calculations performed on the data are consistent with other results obtained with x-ray or neutrons scattering.

The high temperature chamber was mounted on the center of the D2AM 7-circles goniometer. The working principle of this device is largely described in details elsewhere[1]. The spherical sample with a diameter around 2.7mm was situated on a levitator in the center of the chamber. This device contains a nozzle for diffusion of a gas jet below the sample. In this study we used a mixture of argon and hydrogen (2.5%) in order to avoid sample oxidation. The gas flow was regulated with a mass flow controller enabling the sphere to remain in a stable position without any contact with the nozzle.
The heating system consisted of a continuous 125W CO_2 laser operating at a wavelength of 10.6µm. The primary laser beam with a diameter of approximately 5 mm was focused on the sample using mirrors in order to obtain a final diameter of about 1mm.

During the experiment, the spot size at the sample position was 0.5mm vertically and 1mm horizontally. Total X-Ray Diffraction (XRD) and Anomalous X-ray Scattering (AXS) measurements were performed using a NaI scintillator coupled to a graphite[002] analyzing crystal giving an energy resolution of about 0.15keV. Absorption measurements were performed in the fluorescence mode using photodiodes included in the levitation chamber.

Zr-Ni Alloys are known to give easily metal glasses by fast quenching, in a wide range of compositions. However, this relative ease for these compounds to form glasses is not completely clarified. This property is connected largely with the alloy viscosity, the mobility of atoms in the liquid playing an important role in the glass forming properties. The degree of mobility depends on the local atomic structure in the material. It was then important to determine the structure factor and the corresponding radial distribution function of these compounds in order to better understand their physical properties.

XRD measurements were first performed on liquid Zr at various temperatures above the melting point (1850°C) at energy of 17.9keV. Scattered x-rays were detected over an angular range of 3-105° giving a maximal Q value around 14.5 Å-1. Figure 1 shows the x-ray structure factor S(Q) for the liquid Zr at 1930°C. The corresponding pair correlation function is presented in figure 2.

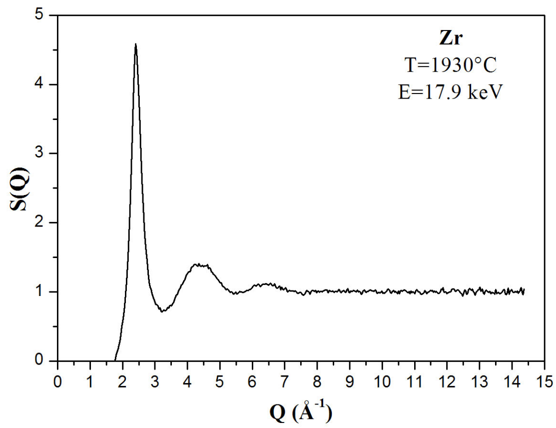

Figure 1 : X-ray structure factor of liquid Zr (1930°C) measured at 17.9keV.

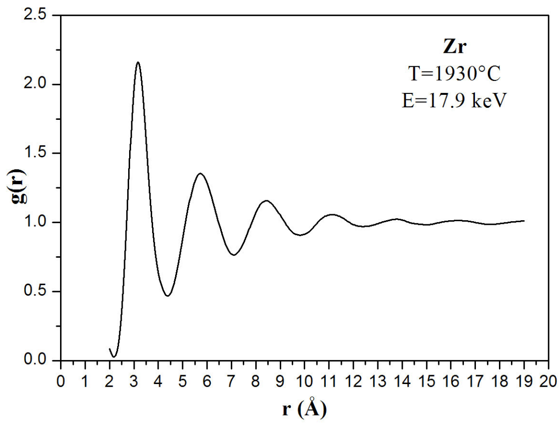

Figure 2 : Pair correlation function of liquid Zr (1930°C).

Non expected structural modifications with temperature have been observed in previous x-ray measurements on liquid Zr[2]. Then it was important to study again the structural behaviour of Zr with the temperature. The first calculation of the distances and coordination numbers are summarized in the following table.
From these new results, we can verify that liquid Zr doesn't show significant change in the structure with the temperature as observed previously. This is consistent with structural results obtained with neutron scattering using electromagnetic levitation[3]

T (°C)	R1 (Å)	R2 (Å)	R3 (Å)	CN
1930	3.20	5.74	8.27	12.3
2050	3.18	5.81	8.33	12.3
2170	3.21	5.79	8.31	12.2
2300	3.22	5.84	8.31	12.5

Concerning ZrNi samples, calculations are in progress. The first calculated distances from the two first peaks in g(r) are summarized in the following table. These values are consistent with the different distances found for liquid Zr at 2050°C and Ni at 1320°C.

Pair	Peak 1	Peak 2
Ni-Ni	2.52Å (2.51Å for liquid Ni at 1320°C)	4.52 (4.58Å for liquid Ni at 1320°C)
Ni-Zr	2.78Å	5.22Å
Zr-Zr	3.19Å (3.18Å for liquid Zr at 2050°C)	5.86Å (5.81Å for liquid Zr at 2050°C)

If aerodynamic levitation and laser heating can be used to work on liquid metals, this technique is not optimal in term of laser power and wavelength. Then the 125W used laser was not sufficient to keep the samples in the undercooled state during extended duration. In further experiments, we plan to combine aerodynamic levitation and RF heating, more adapted method to melt metals.

[1] L. Hennet, D. Thiaudière, M. Gailhanou, C. Landron, J. P. Coutures and D. L. Price, Rev. Sci. Instrum. 73, 125 (2002)
[2] S. Krishnann and S. Ansell, J. Phys.: Condens. Matter 11, L569 (1999)
[3] T. Schenk, D. Holland-Moritz, V. Simonet, R. Bellissent, D. M. Herlach, Phys. Rev. Lett. 89, 75507 (2002)