

EXPERIMENTAL REPORT

RAPPORT D'EXPERIENCE

Programme Committee Proposal
Number
N° Projet Comité de Programme

PROJECT TITLE : *TITRE DU PROJET :*

In situ absorption and diffraction of X-ray at high temperature.

LIGNE :D2AM

INSTRUMENT :	PETITS ANGLES	<input type="checkbox"/>	EXAFS	<input type="checkbox"/>
	7 CERCLES	x	GM	<input type="checkbox"/>
	BIO-CRISTALLOGRAPHIE	<input type="checkbox"/>	SUV	<input type="checkbox"/>

NUMBER OF RUNS USED 15 shifts

NOMBRE DE SESSIONS EFFECTUEES :

STARTING DATE 1-Juillet 98

DATE DE DEMARRAGE :

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ABSTRACT

We have developed at the CRMHT a setup based on laser heating and aerodynamic levitation in order to gain an insight of materials at very high temperature. This experiment permits to investigate the structural properties, especially short and long-range order in partially disordered specimen. The analysis chamber, which has been designed for working on the 7-cradle goniometer of the D2AM station, consists in an isolated cell performing in-situ experiments combining X-ray absorption and diffraction. Up to now, three teams had used levitation (electromagnetic, aerodynamic or acoustic) associated with a convenient heating (electromagnetic, laser irradiation) for performing one of the X-ray techniques, but not any has combined two or more among these X-ray spectroscopies. We have studied the structure of some solid and liquid refractory oxides at temperatures up to 2750°C : ZrO₂, melting temperature $T_m = 2715^\circ\text{C}$; Y₂O₃, $T_m = 2440^\circ\text{C}$; YAG, yttrium aluminum garnet, $T_m = 1945^\circ\text{C}$; Gd₂O₃, $T_m = 2440^\circ\text{C}$; Ho₂O₃, $T_m = 2420^\circ\text{C}$ and Er₂O₃, $T_m = 2420^\circ\text{C}$.

The levitation of the spherical sample is performed with a gas jet (argon or helium) to steadily bear a spherical sample in energy well. A convergent-divergent diffuser creates a stable vortex ring that traps the sample. This levitator allows the sample to float in a central position independently from any contact. A similar system has been used for shaping the spherical sample from compact powders that have been used in X-ray characterization experiments. The heating is obtained by the irradiation of the sample by a continuous wave (cw) 100 W CO₂ laser (SYNRAD). For security reasons, the invisible CO₂ laser beam was displayed by a red He-Ne laser beam. Our device has been configured in order to provide a large open solid angle for the photodiodes used as detectors for XAFS experiments in fluorescence mode.

X-ray absorption spectra have been recorded in the fluorescence mode with large area silicon photodiodes (Hamamatsu). The position of the detectors has been adjusted close to the sample in

order to achieve an optimum fluorescence signal emitted by the sample. The photodiode supports were water-cooled. Beryllium windows have been used to transmit X-rays and to absorb visible light. Oscillation functions of the yttrium K-edge recorded at 2200°C above melting for an Yttrium Aluminum Garnet (YAG) sample have been measured. With a record time of 5s per point, the signal to noise ratio seems to be sufficiently high to exhibit the EXAFS oscillations above $k = 8 \text{ \AA}^{-1}$ characteristic of the yttrium-yttrium bond

In order to perform time resolved diffraction measurements on sample during heating and cooling, we have used a Position Sensitive Detector (PSD) developed by J.F. Berar. A gas (argon) detector is used with an angular aperture of 30° (angular resolution in 2θ of 0.1°) located at a distance of 130 mm of the spherical sample. The measurement of the position of a photon results from the determination of the delay time between two signals emitted by the incoming photon. 50 diffraction patterns have been recorded during the cooling time of 2.5s (50 ms per diagram) of a liquid drop of Ho_2O_3 , showing the solidification of the drop. From the diagram, we suggest the following phase transitions : the first, towards a mixing of high temperature cubic and hexagonal phases, the second towards a mixing of low temperature cubic and hexagonal phases and the last, towards a low temperature cubic phase.

We have also recorded diffusion spectra on various liquid oxides with a scintillator coupled to a photomultiplier°. The spectra were recorded on an angular range of 120° giving a complementary information to the pair function data calculated by EXAFS analysis. The interest of measurement of X-ray diffraction oscillations results from the reliability of the information obtained at low k values.

Publications related to these experiments during 1998 and 1999

I Conferences as invited speaker

1998

C. Landron, L. Hennet, J.P. Coutures and J.F. Berar - 2nd International Conference on Synchrotron Radiation in Materials Science, Kobe, Japan - "*Aerodynamic levitation studies of ceramics at high temperature : XAFS and XRD*". Publication in : Japanese Journal of Applied Physics. Invited speaker.

C. Landron - 18th European Crystallographic Meeting, Prague, République Tchèque - "*In-situ phase transition characterization probed by synchrotron radiation*". Pub. In a book. Edited by K. Hazek. Plenary lecture.

C. Landron - Gordon Conference on High Temperature Materials, Processing and Diagnostics, Plymouth, New Hampshire, USA - "*A structural approach to laser heated liquids by synchrotron radiation*". Invited speaker.

C. Landron, L. Hennet, J.P. Coutures, M. Gailhanou, M. Gramond, J.F. Berar and K. Sakurai C. Landron. - Ninth International Workshop on Fine Ceramics, Nagoya, Japan - "*refractory oxide investigation by X-ray at high temperature*". Publ. in Nov 98, éditées par K. Koumoto, L.M. Sheppard and H. Matsubara, in Mass Charge Transport in Ceramics, Ceramic Transaction, Volume n. Pub. The American Ceramic Society, Westerville, USA, (1998) P.ppp. Session chairman, invited speaker.

1999

C. Landron. - ninth International Symposium on Nondestructive Characterization of Materials, Sydney, Australia - "*Aerodynamic levitation. A new opportunity in nondestructive Characterization at high temperature*". Proceeding edited by : R.E. Green. Member of the international organizing committee.

C. Landron. - 18th International Union of Crystallography Congress, Glasgow, UK - "*Measuring XRD/XAS from high temperature oxides*". Proceeding edited by : C.C. Wilson, K. Shankland and T. Csoka. Published in Acta Cryst., **A55**, (1999) P. 189. Invited speaker.

II Papers

1998

C. Landron, L. Hennet, J.P. Coutures, M. Gailhanou, M. Gramond, and J.F. Berar, ESRF Newsletter - 30, 39, - "*The high temperature project at the french D2AM-CRG beamline*"

C. Landron, L. Hennet, J.P. Coutures, M. Gailhanou, M. Gramond, and J.F. Berar. - Europhysics Letters - **44**, 429, - "*Contactless investigation on laser heated oxides by synchrotron radiation*"

1999

C. Landron, L. Hennet, J.P. Coutures and J.F. Berar. - Japanese Journal of Applied Physics - **38-1**, 87, - "*Aerodynamic levitation studies of ceramics at high temperature : XAFS and XRD*".

L. Hennet, C. Landron, P. Berthet, J.P. Coutures, T.E. Jenkins, C. Aletru and G. N. Greaves. - Japanese Journal of Applied Physics - **38-1**, 115, - "*X-ray Diffraction and Near Edge Structure of iron oxides and alumina at high temperature using aerodynamic levitation and laser heating*".