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Study of Crystallisation of Yttrium Garnets $Y_3R_5O_{12}$ ($R = Al, Ga$) by High Temperature EXAFS

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Abstract. In this paper, we present a new high temperature EXAFS device using a laser heating system and aerodynamic levitation for experiments up to 2300°C. We report here the first spectra of Y K-edge for YAG at room temperature and in the liquid.

1. INTRODUCTION

Refractory oxide liquids form a wide range of technologically important materials. Knowledge of the structure at these high temperatures is necessary for an understanding of their physical properties. In the aim to extend the applications of the X-ray absorption fine structure, we will present an original experimental device different from that used by Crozier et al [1]: a levitation cell for radiation experiments at very high temperature. We have develop a high temperature EXAFS device, this technique using radiation synchrotron associated to a laser CO₂ allows to study liquid state (above fusion temperature) and to reach temperature up to 2500°C. In the first part, we present the experimental device and in a second the first spectra with this containerless sample holder.

2. SYNCHROTRON RADIATION AT VERY HIGH TEMPERATURE

The EXAFS experiments were performed using DCI synchrotron radiation source at LURE (ORSAY, France). The DCI storage ring operating at 300 mA and 1.85 GeV. The X-Ray beam was monochromatized by a double Bragg reflexion Si (311) crystals. The energy resolution was estimated to be 2 eV.

2.1 Experimental device

In Figure 1 a schematics of the analysis setup is shown. An aerodynamic levitator allows the spheric sample to float from any contact especially at high temperature.

Figure 1 : Levitation cell for high temperature EXAFS experiments

The detector is adjusted in order to obtain optimum fluorescence emitted by the sample. We used a Si photodiode of 300 μ m thickness to obtain the higher detection efficiency.

This high temperature device presents several advantages : the liquid is neither physically nor chemically perturbed by the container, suppression of heterogeneous nucleation during cooling, suppression of the pollution of the liquid by the container.

2.2. Results and discussion

Rare earth aluminium garnet $\text{Ln}_3\text{Al}_5\text{O}_{12}$ ($\text{Ln} = \text{Y}$) single crystals have been great interest in solid state electronic systems. Temperature dependent X-ray Absorption Spectroscopy offers a great opportunity for structure analysis of YAG processing by sol-gel method [2]. YAG powders were prepared by an organic gel-assisted citrate process [3] A representative plot of the EXAFS interference function $\chi(k)$ is shown Fig 2.

Figure 2 : Spectra of Y K-edge for YAG at room temperature (RT) and 2200°C (HT)

The dominant low frequency oscillation in $\chi(k)$ at high temperature is determined by interference of the photoelectrons for the first shell coordination in comparison with the room temperature where appears the second shell.

A comparison of the Fourier transform at room temperature and in the liquid phase indicates some structural changes with the temperature. Structural parameters obtained by least squares fitting the EXAFS spectra for the coordination shell around yttrium ions. By the fit, the distance yttrium-oxygen is found to be lower for the room temperature: $d(\text{Y-O}) = 0.233$ nm and 0.242 nm at room temperature $d(\text{Y-O}) = 0.295$ nm / 0.319 nm at 2200°C. The interpretation of the increase of this distance is based on the fact that the temperature brings a distortion of the dodecahedron in the liquid phase.

3. CONCLUSION

We have developed a new X-ray analysis cell for synchrotron radiation experiments at high temperature. This study illustrates the possibilities of this new device to study solid state mechanisms such as phase transitions and knowledge of liquid phase. Great improvements in signal -to-noise ratio can be expected with our new cell and for the synchrotron of the third generation.

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