ESRF	**Experiment title: Pressure-induced** a -g **phase transition in mixed-valence Ce : A resonant inelastic x-ray scattering study**	**Experiment number**: HE-1127
Beamline: ID12	**Date of experiment**: from: 23/10/2001 to: 30/10/2001	**Date of report**: 15/11/2001
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Report:

We report here the investigation of the α/γ valence transition in pure Ce under pressure by resonant inelastic x-ray scattering .

The sample used was a 25 micron thick Ce foil from GoodFellow initially contained in an argon-filled ampoule. Maximum care was taken in order to avoid oxidation by air during the experiment preparation. Also, the sample was loaded in a glove box under argon atmosphere directly from the ampoule into a compact Mao-Bell diamond anvil cell prepared with 600 microns-culet diamonds and using a 3mm diameter Be gasket. The choice of small-sized Be gasket was dictated by the low energy (~4.9 keV) of the Ce L_α emission lines which were to be measured in this work. Paraffin oil was used as an oxygen-free pressure medium thus ensuring no further degradation of the sample quality during the experiment. Pressure was monitored by conventional ruby fluorescence technique.
It appeared however that the Ce foil was already oxidized in the ampoule, as it will become clear in the following and the expected transition which occurs in pure Ce could not be observed, although the quality of the measured spectra are satisfactory considering the sample environment . Nevertheless, this demonstrates the feasibility of such high-pressure experiments by inelastic x-ray scattering, even at these low energies (below 5 keV).

The inelastic scattering spectra were measured in the compact UHV-compatible spectrometer developed by the Laboratoire de Chimie Physique, which was hooked up to the ID12 beamline. The spectrometer consists of a 0.5 m-radius cylindrically-bent quartz analyzer in the Rowland circle geometry operating using the 20-23 reflection and a gas-filled detector. The monochromatized incident beam was focused vertically down to 140 microns. The spot size of 25 microns foreseen before the experiment could not be obtained due to a technical difficulty with the beamline. The incident beam entered the cell in the gasket plane (perpendicular to the diamond axis) and the emission was detected at 90° scattering angle in reflection mode.

The absorption spectra at the Ce L_3 under pressure are shown on Figure 1 both in the total yield and the partial yield modes. The spectra are compared to a reference CeO_2 sample. The close resemblance in the near edge region between the two sets of spectra, in particular the existence of two peaks at 5730 and 5740 eV characteristic of CeO_2, indicates the oxidized nature of our sample (the uprising trend at higher energies comes from background-subtraction corrections). These absorption spectra can be confronted to the ones obtained by Lengeler *et al.* in the Ce (see Ref. [1]) which shows that the white line of pure Ce-γ presents a single and intense peak located at 5724 eV with no comparison with the present data. The same conclusion can be derived from the partial yield data. These spectra were measured by setting the analyzer to the maximum of the $L_{\alpha 1}$ emission line while scanning the energy across the absorption edge. In that way, the core hole lifetime broadening effect is almost completely suppress yielding to a strong enhancement of the spectral features. Again, one could note that the two sets of spectra superimpose each other within the error bar.

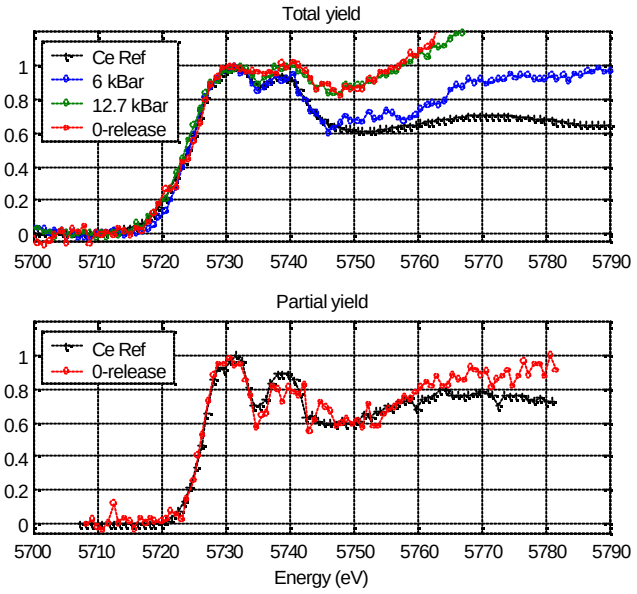

Figure 1 : Total and partial yield Ce L_3 absorption edge in an reference Ce foil and in the pressure cell

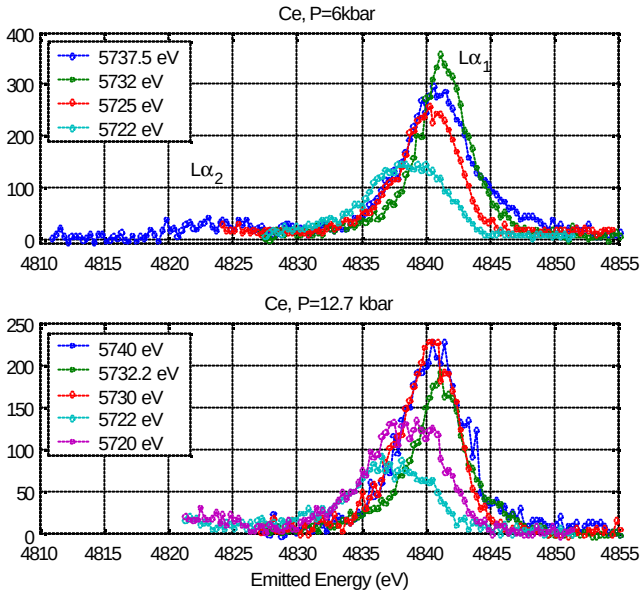

Figure 2 : Resonant inelastic x-ray scattering spectra in Ce at 6 and 12.7 kBar.

The resonant emission spectra of Ce under pressure are displayed in Fig. 2. In the fluorescence regime, the countrate was about 15 counts / sec. This intensity diminished by about a factor of two in the resonant mode as expected from the self absorption effect. At 12 kBar; another factor of two were lost because of the closure of the diamond anvils gap, which became smaller than the vertical beam size. The shift of the emission line with the incident energy is a typical fingerprint of the resonant regime. No clear resonant features could be observed. The poor statistics essentially due to the mismatch between the sample size and the x-ray spot, has limited us to a restricted number of resonant spectra, which did no permit to extract detailed information concerning the electronic structure.

Besides the problems related to sample quality; this challenging work, the first of its kind in this energy range, has proved the potentiality of inelastic x-ray scattering to study the electronic properties of sample under extreme conditions. A gain of about two orders of magnitude in the emitted intensity is expected with a vertically and horizontally focused beam spot, whose dimensions correspond to the sample in the pressure cell. We are also presently investigating a more reliable sample preparation method in order to avoid its oxidation for a future attempt.

[1] B. Lengeler, G. Materlik and J.E. Müller, Phys. Rev. B **28**, 2276 (1983)