EUROPEAN SYNCHROTRON RADIATION FACILITY
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF. Once completed, the original report should be sent, together with 5 reduced (A4) copies, to the User Office.

In addition, please send a copy of your file as an e-mail attachment to reports@esrf.fr**,** using the number of your experiment to name your file. This will enable us to process your report for the ESRF Annual Report.

Reports accompanying requests for additional beam time

If your report is to support **a new proposal**, the orginal report form should be sent with the new proposal form, and a copy of your report should be attached to each copy of your proposal. The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.
- bear in mind that the report will be reduced to 71% of its original size. A type-face such as "Times", 14 points, with a 1.5 line spacing between lines for the text, produces a report which can be read easily.

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ESRF	**Experiment title**: STRUCTURAL STUDY OF THE INCORPORATION OF RARE-EARTH SPECIES IN AMORPHOUS SOL-GEL FILMS BY XAFS SPECTROSCOPY.	**Experiment number**: HS-1119
Beamline: ID26	**Date of experiment**: from: 23/2/00 to: 29/2/00	**Date of report**:
Shifts: 18	**Local contact(s)**: P-E Petit	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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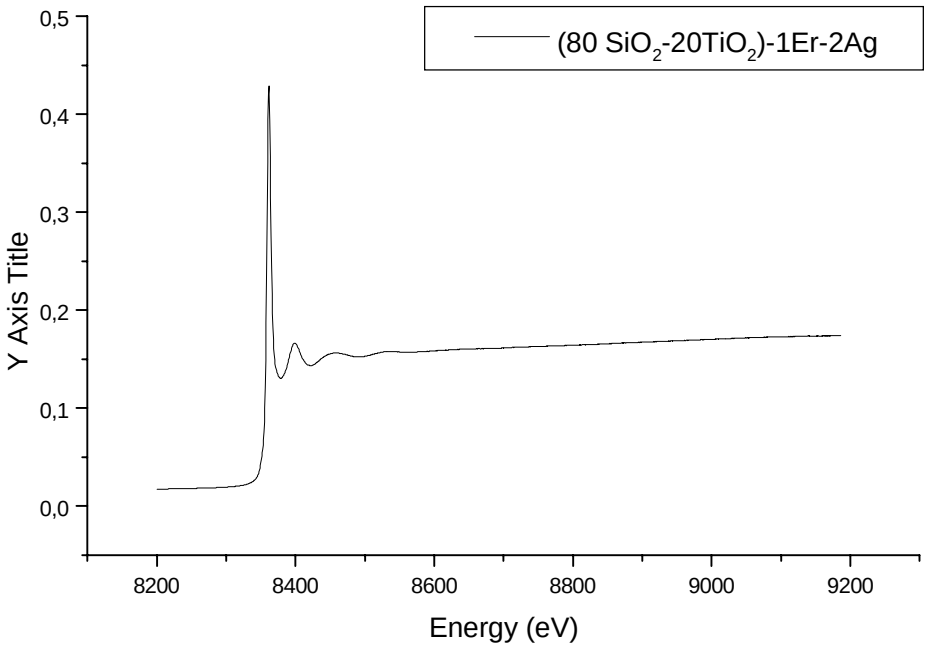
Report:

The present project deals with the study of the effect of nanometer-sized Ag particles on the fluorescence behavior of Er^{3+} ions in SiO_2 or SiO_2-TiO_2 glass matrices, in particular with the possible relationship between the structural incorporation of the Er^{3+} ions and the presence of the Ag particles. The aim of this research is to determine the nature of the near neighbor vicinity of the Er^{3+} ions in those glassy networks by EXAFS spectroscopy at the Er L_{III} edge (8.358 keV), as a function of the glass composition and heat treatment.

The investigated samples were films ca. 300 nm thick, deposited by spin-coating on glass substrates, in the SiO_2-Er_2O_3:Ag and SiO_2-TiO_2-Er_2O_3:Ag systems, using sol-gel processing, with tetraethylorthosilicate, titanium isopropoxide, and Er and Ag nitrates as precursors, in ethanol/water media, under acidic conditions. The films were heat treated at 600°C for 15, 30 or 60 minutes; some were only dried at room temperature.

A series of 33 samples was analyzed as a function of the TiO_2 content, Er content, Ag content and heat treatment.

EXAFS spectra were recorded in the fluorescence mode, at the Er L_{III} edge (8.358 keV), in the range 8.2-9.2 keV. A Si(220) double crystal monochromator, with an entrance slit of 0.4 mm and the undulator u42, as the insertion device (which was later changed to the u35 undulator, due to some technical problems), were used and the detector was equipped with a 9 μ Fe filter (in order to avoid the Compton signal). Energy resolution was estimated to be ~0.4 eV. The samples were analysed at grazing incidence and the measurements were first carried out at 55 K, in order to attenuate the thermal vibrations and therefore reduce the Debye-Waller factors. However, it was later realized that no real advantage was obtained for these samples and the subsequent measurements were then carried out at room temperature, where the time required to position and align the samples was much shorter. Nevertheless, good quality spectra have been obtained (see figure). Preliminary EXAFS analysis, obtained with the XAFS program, showed different behaviour for the room temperature and the heat-treated samples, but further analysis is necessary to characterize the nature of the atomic coordination around Er and the role of Ag.



Some XAFS spectra were also recorded at the Ag K edge (25.51 keV). These measurements were carried out at room temperature and the XANES portion of the spectra allowed us to obtain a reliable fingerprint of the silver oxidation state: amorphous and crystalline metallic patterns were easily recognized in samples with different heat treatments. Further investigations are necessary to elucidate the nature of the atomic coordination around Ag.