ESRF	Experiment title: **EXAFS investigation of a chalcogenide Ge-Ga-Sb-S glass doped with Nd for optical applications**	**Experiment number**: MA-1992
Beamline: BM-08	**Date of experiment**: from: 14/02/12 to: 14/02/18	**Date of report**:
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Results expected and their significance in the respective field of research :

In the proposed investigation, two different aspects of the glass structure will be investigated :
Our objective is **a structural comparison between bulk chalcogenide glasses and thin films for a better understanding of their optical properties**. Therefore, the structure of bulk plates and thin films will be characterized by using EXAFS as a structural probe of the local environment around the Ge, Ga, Sb atoms;
The environment of the Nd^{3+} ions will be studied in order to better understand the interaction of rare earth ions and their local structure surrounding and correlate them with the optical properties.
The structural organisation of amorphous materials is very difficult to study due to the lack of long-range structure typical of crystalline materials. The high brilliance and high energy x-ray beam delivered by the ESRF source will permit **the investigation at the Ge, Ga, Sb and the rare earth (RE) ions K-edge for these glass materials.** We expect data of very high quality obtained at rare-earth K-edge; a much larger k range compared to the L_{III} edge can be accessed since the k_{max} is not restricted by higher energy absorption edges

Results and the conclusions of the study (main part):

In order to compare local structure of glass in bulk and thin film the K edge of **Ge, Ga, Sb, Se and RE** has been investigated for a set of relevant bulk compositions and two deposition techniques (sputtering and pulsed laser deposition). The measurements at Ge, Ga and Se K-edge present good quality for the signal to noise ratio both for bulk and thin films. For the tested deposition conditions, just a minor variation in the average local structure of Ge, Ga and Se can be observed, the small deficit in Se due to film deposition perturb only slightly the structure (see Figure 1 for Ge K-edge).
Moreover on Ge K-edge XANES, we notice a close correlation

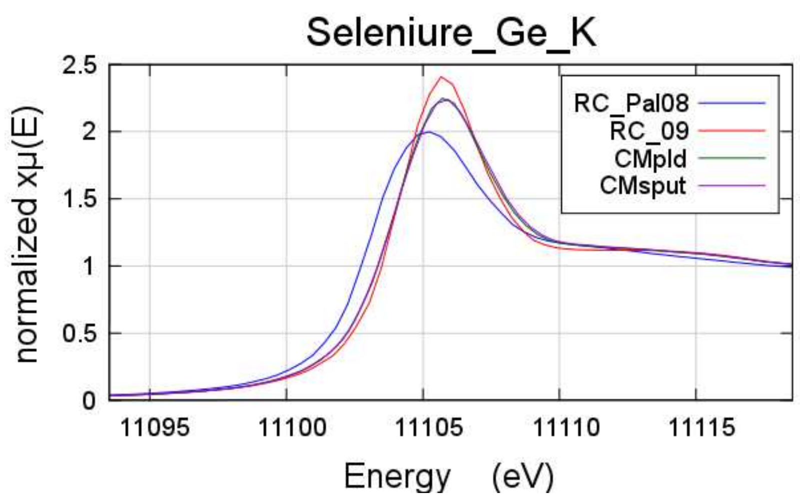

Figure 1 : Comparison between PLD and sputtered film (CMpld, CMsput, respectively) and glass bulk target (RC_09) used for deposition and Ge rich bulk glass (RC_Pal_08)

between shape of Ge XANES and colour of the pellets (directly linked to the glass band-gap related to the selenium variation in respect to stoechiometric composition). Such correlation is illustred in figure 2, showing the fundamental contribution to Ge electron density respect to the material band-gap.

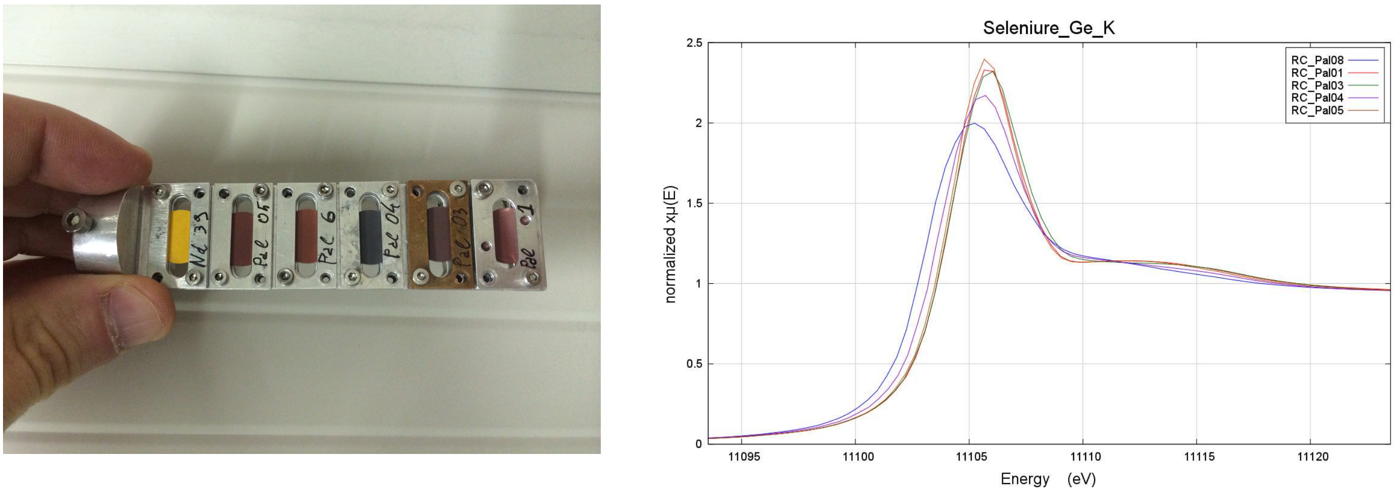

Figure 2 : correlation colour of the pellets and Ge XANES position for different bulk compositions (from $Ge_{20}Se_{80}$, $Ge_{20}Sb_5Se_{75}$, $Ge_{20}Sb_{10}Se_{70}$, $Ge_{20}Sb_{15}Se_{65}$, $Ge_{20}Sb_{20}Se_{60}$).,

However, for Sb and RE K edge results have been less satisfactory. For Sb-K edge, spurious features likely due to monochromator glitches that are not normalized through the inhomogenous sample (pressed powder) appeared between 30680-30830 eV. The spectra measured in transmission as well as in fluorescence mode are however exploitable except for one sample having the lowest Sb concentration.
Regarding RE K-edge only for high concentrated bulk sample an acceptable signal has been recorded (See figure 3). Indeed, the EXAFS signal is strongly damped by disorder and consequently, a very high signal to noise ratio is required to obtain reliable results. Such a condition is very difficult to obtain working at such high energy (~42000 KeV).
In the case of thin films, we just made an attempt to measure spectra in fluorescence mode. However, most of the signal reaching the detector comes from the chalcogenide matrix where the rare earth ions are diluted and there is also a strong Compton scattering signal from the substrate. The signal from the RE ion was finally too low to collect data with sufficient statistics in a reasonable collection time.
From the preliminary analysis of the Nd:bulk sample, it is clear that EXAFS signal disappears very early around k =11 $Å^{-1}$ corresponding to ~ 450 eV from the edge. In such case, the limitation in k for L_3 edge are bearable and at L_3 edge due to the minor penetration of X-ray in the film and the lack of fluorescence emission from the other glass components should make possible to measure RE EXAFS signal.

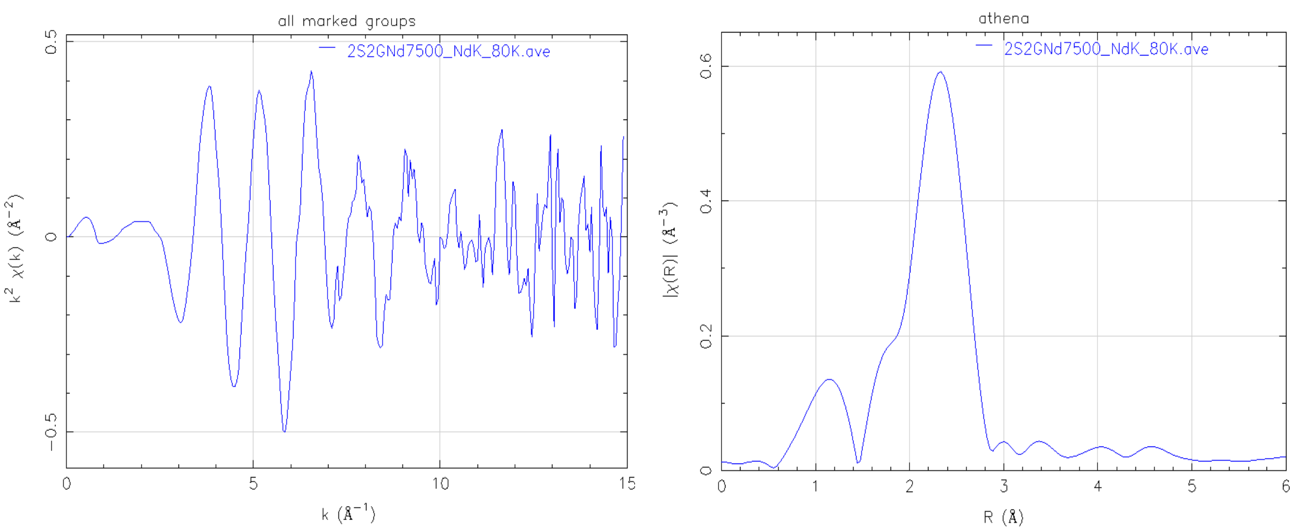

Figure 3 : Nd K edge EXAFS signal for bulk glass.