ESRF	**Experiment title:** ***Short-range order of Cr and Mo in amorphous steels by EXAFS. Influence of structural changes in the ductility and mechanical properties of steels***	**Experiment number**: MA-656
Beamline: BM25A	**Date of experiment**: from: 24/10/2008 to: 28/10/2008	**Date of report**: March 1st, 2009
Shifts: 12	**Local contact(s)**: Jon Ander GALLASTEGUI	*Received at ESRF:*

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Report:

The aim of the experiment was to obtain information about the local atomic environment of Molybdenum, Chromium, and Iron atoms in melt-spun ribbons of amorphous steels of Fe(71.2-x)C(7.0)B(3.3)P(5.5)Cr(2.3)Mo(x) with the Mo concentration varying between 0 and 6.5 at. %. These compositions were previously characterized by Mössbauer spectroscopy in our lab and by x-ray diffraction at the beamline BM16 at ESRF and showed different iron environments that correlated with changes in the glass forming ability reported in the literature [1]. Therefore the main result searched for with this proposal was the assessment of the local environments of Mo sites, in order to establish how the atomic configuration changed upon doping and how these changes influence macroscopic properties such as the critical size of amorphous casting and the domain distributions observed using Mössbauer spectroscopy.

To this aim, 12 shifts of (7/8+1)-hybrid mode beam time were alloted at BM25A. The experiment required two different setups:

1) Transmission detector: in order to obtain the EXAFS data at Mo K-edge
2) Fluorescence multichannel detector: in order to obtain the EXAFS data at the Fe K-edge

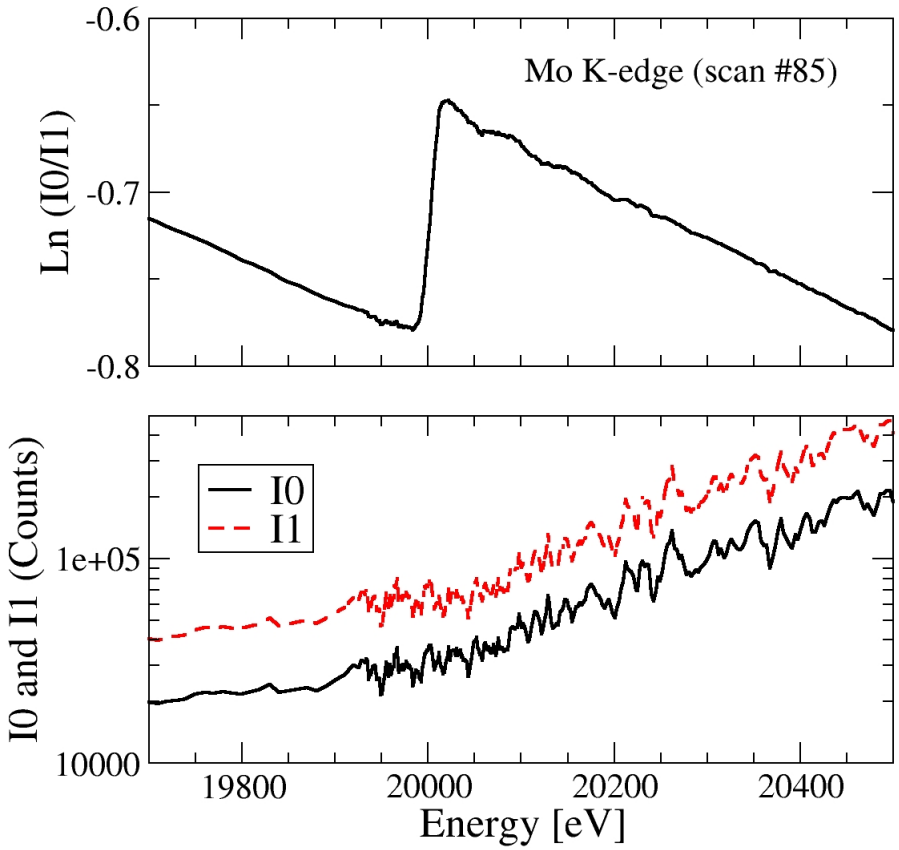

Fig. 1: Scan 85, corresponding to EXAFS measurements at the Mo K-edge in a sample with 6.5at.% of Mo. The upper pannel shows the EXAFS spectrum, obtained from the incoming (I0) and transmitted (I1) intensities, displayed in the lower pannel.

Unfortunately, due to unexpected delays in the delivery of the multichannel detector the fluorescence signal had to be acquired with a single channel detector, which resulted in spectra of poor statistics and unreliable for further data treatment.

The lack of stability of the I0 (incoming intensity) signal made unsuccessful all attempts to analyze the x-ray absorption data obtained in transmission geometry, corresponding to the Mo K-edge XANES and EXAFS spectral regions, even for the largest Mo concentration (6.5 at. %). Figure 1 exemplifies the challenge presented during the experiment, given the high correlation between the I0 and I1 (detected intensity) signals and the large oscillations observed in both signals, which affected dramatically the quality of the EXAFS spectra (see upper pannel). Beside this, the pre-edge shows as well anomalous noise levels.

This issue might be related to deficiencies in the monochromator instrumentation, which according to our local contact will be replaced in the near future.
Tests performed on ribbons with the same composition and same synthesis batch on BM29 warrant the quality of the samples and the feasibility of the proposed experiment. A continuation proposal will be submitted in order to repeat the experiment in another beamline.

[1] *Mössbauer characterization of an amorphous steel with optimal Mo content*, L. Facchini, P. Bruna, E. Pineda and D. Crespo, J. Non-Cryst. Solids, 354, 5138 (2008)