EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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 report should be submitted electronically to the User Office via the User Portal: *https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do*

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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.

ESRF	**Experiment title:** Investigation of closely packed local structures in a simple metallic glass ($Ni_{62}Nb_{38}$) under pressure	**Experiment number**: HC-4846
Beamline: BM23	**Date of experiment**: from: 28/06/2022 to: 04/07/2022	**Date of report**: 31/08/2022
Shifts: 18	**Local contact(s)**: Joao Elias Figueiredo Soares Rodrigues	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The phenomenon of amorphous-amorphous phase transitions (polyamorphism) has been investigated over the years, mostly in covalently bonded systems with open local structures, such as network forming SiO_2 and GeO_2 glasses [1-7]. Recently, polyamorphic transitions have been reported to occur also in some metallic glasses with closely packed local structures, and the densification in these systems was suggested to follow a very different mechanism [8,9], being associated with drastic changes in the electron orbital configurations. Because of the structural complexity in typical metallic glasses (mostly multi-atomic), the local structure of such densely packed disordered systems and its changes induced by high pressure are still poorly understood. Because of the structural proximity, pressure behaviour of the melt quenched metallic glasses and the corresponding parent liquid. Therefore, studies of the densification processes in the metallic glasses may help for understanding the pressure behaviour of structurally close packed metallic liquids which abundantly exist in nature under extreme conditions (e.g., Earth's liquid outer core, composed mainly by Fe and Ni), but very challenging to study.
Binary metallic glasses such as Ni-Nb systems are among the simplest metallic glasses, and can be a good simple prototype for studying the pressure behaviour of closely packed local structures under high pressure using x-ray spectroscopy techniques (K-edge energies compatible for x-ray absorption through diamond anvil cells). Moreover, Ni-Nb systems belong to one of the few binary metallic glasses that could be cast into large bulk sizes, are at the cutting edge of the materials research with their high compressive strength and corrosion resistance [10]. Studying the pressure behaviour of local structures in this glasses are meaningful also for understanding the microscopic mechanism of pressure and stress properties of these promising materials.

With these motivations, we have perform high pressure EXAFS measurements at the BM23 beamline of ESRF taking advantage of the highly stable energy scanning setp and focusing optics. High pressure EXAFS data have been measurement in transmission mode at both Ni (~8.333 eV) and Nb (~18.98 keV), along two independent runs. Nano-polycrystalline diamond (NPD) anvils of 200 μm culet have been used in order to obtain glitch free EXAFS data. Initially, samples have been loaded together with Ne pressure medium, however, sample loadings have been prematurely failed, possibly due to the Ne penetration into the micro-structures of the NPD anvils. Thus measurements have been continued with new samples loadings with pressure medium of silicon oil. Ni and Nb K-edge EXAFS data have been measurements up to 71 and 60 GPa, respectively. Maximum pressures were limited to those values due to the perforated design of the anvils (also for Nb K-edge, because of the insufficient number of anvils).

Selected high pressure EXAFS data at the Ni and Nb K edges are shown in Fig. 1 and Fig.2 respectably. From the XANES and 1st derivate spectra of both edges, no sign of significant structural modifications have been identified. Quality of the EXAFS signals can be considered to be quite good considering the highly disordered nature of the metallic glass. Increasing amplitudes of the χ (k).k^2 signals and high of the main peak of Fourier transform possibly indicate either the decrease of the disorder or the gradual increase of coordination numbers. Quantitative EXAFS analysis are currently under progress in order to asses the details of the structural changes occurring in this glass under high pressure.
Even-though no clear evidences of drastic structural modifications have been identified from the 1st glance on the data, results of the on-going quantitative EXAFS analysis are still very important for grasping the behaviour of closely packed local structures under high pressure. A good quality article is expected to be published soon after the complete data analysis.

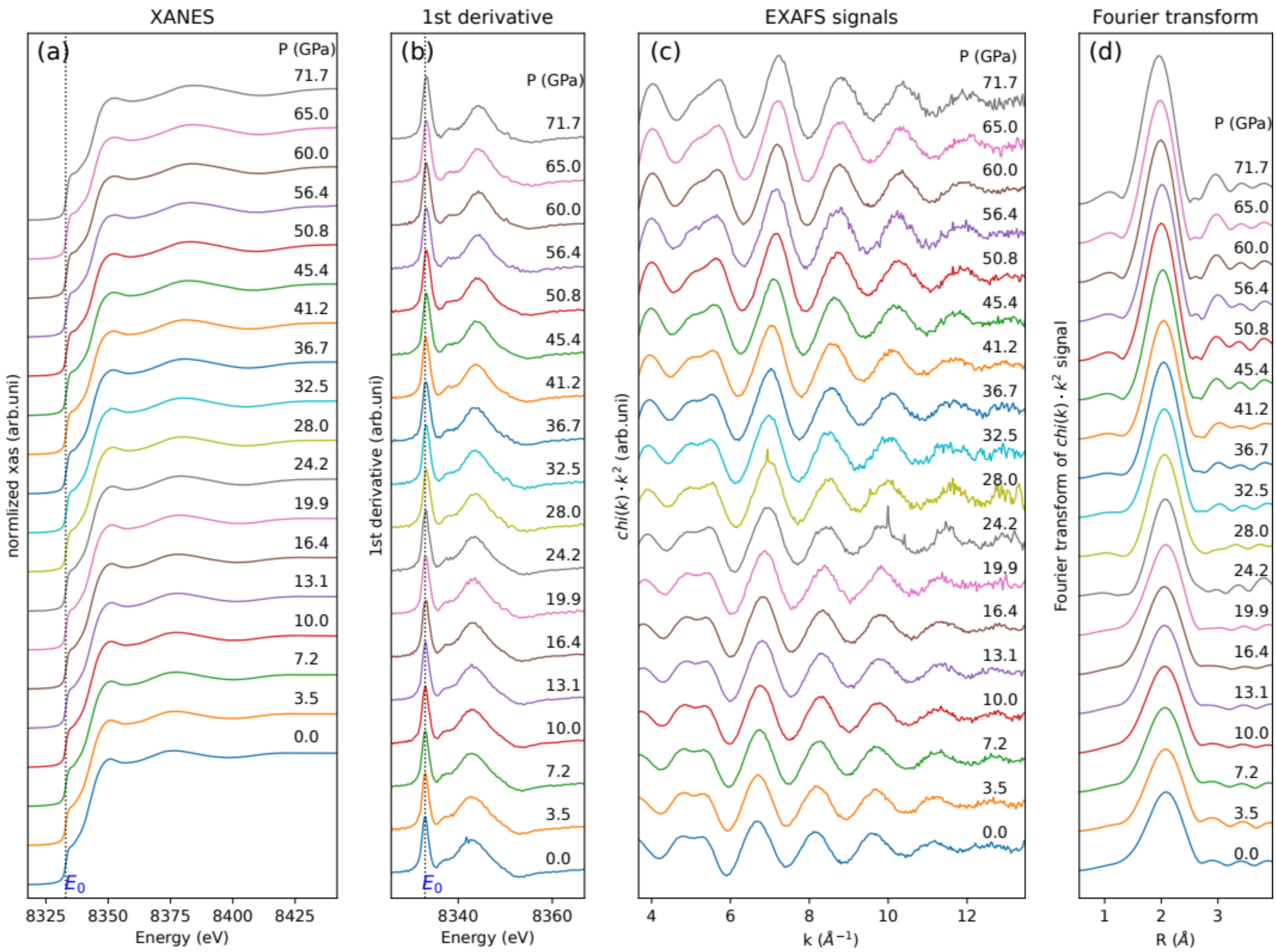

Fig. 1 Normalized Ni K-edge XANES spectra (a) and its 1st derivative (b). Extracted Ni K-edge EXAFS signals (c) and its Fourier transform (d).

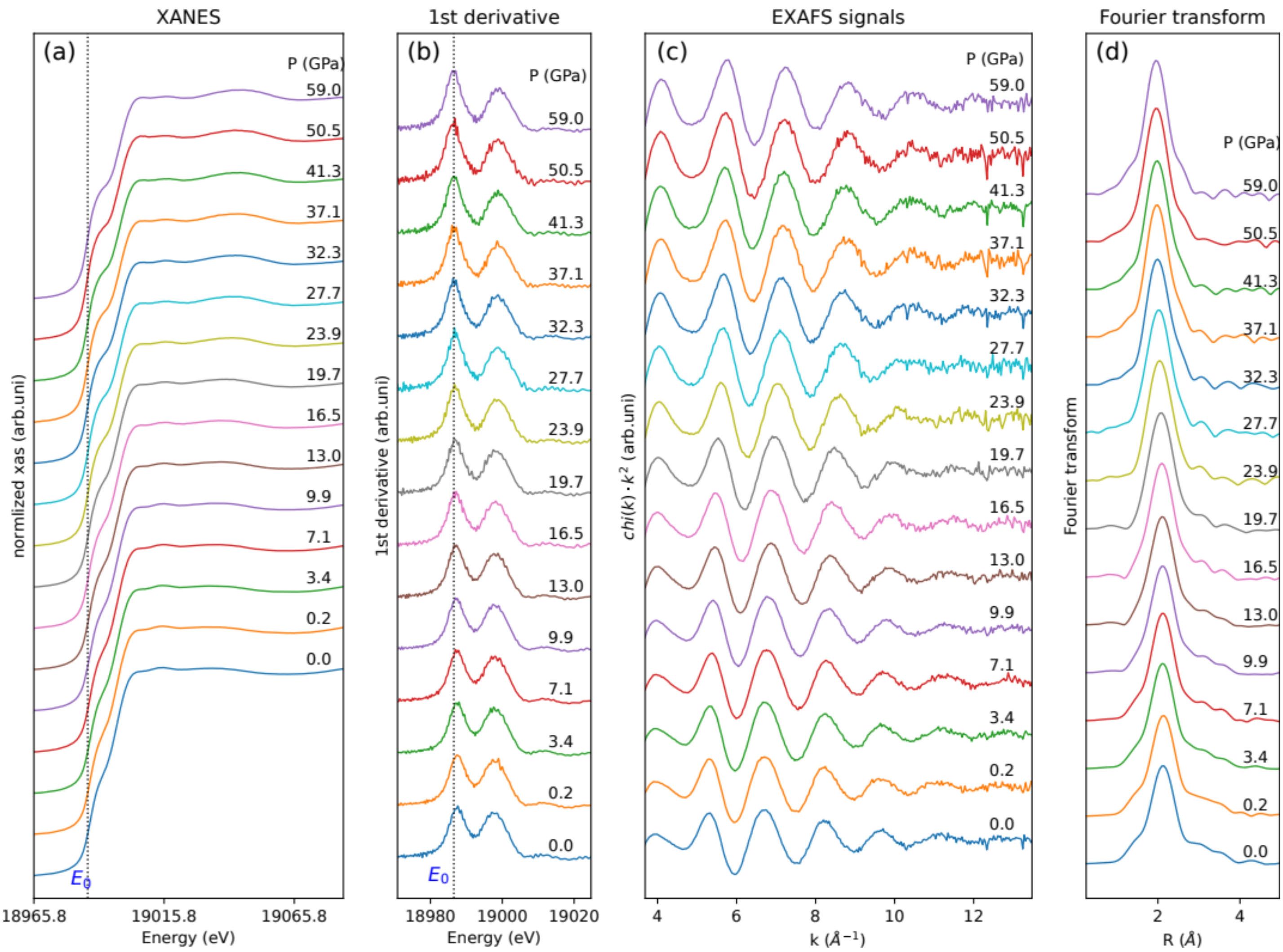

Fig. 1 Normalized Nb K-edge XANES spectra (a) and its 1st derivative (b). Extracted Nb K-edge EXAFS signals (c) and its Fourier transform (d).

References:

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