ESRF	**Experiment title:** Probing solid-solid and solid-liquid phase transitions in laser-shocked Ni by means of time resolved XAS	**Experiment number**: HC-4722
Beamline: ID24	**Date of experiment**: from: 28/06/2022 to: 04/07/2022	**Date of report**: 08/09/2022
Shifts: 18	**Local contact(s)**: Raffaella Torchio, Jean-Alexis Hernandez, and Nicolas Sevelin-Radiguet	*Received at ESRF:*

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Report:

Nickel is a major engineering material, being the main component of Ni-based superalloys which are widely used when operations at high temperature (> 800 K) are required. Ni is also an important geophysical material, being the main alloying element of iron in the Earth's core. It is therefore mandatory to get a good knowledge of its properties at both high temperatures and high pressures. Whereas its properties have been carefully characterized under static high compression, using both XRD and XAS, the phase diagram and melting curve of Ni under dynamic loading conditions are still poorly known. Moreover, the scarce available data show large disagreement with the existing static high pressure data and microscopic theoretical calculations. It is therfore necessary to further study the high pressure properties of Ni under dynamic loading conditions.

In this work, we have investigated the solid-solid and solid-liquid phase transitions in shock compressed Ni, by coupling time resolved XAS measurements with laser shock compression performed using the ID24's High Power Laser Facility (HPLF). The target assembly and the compression schemes were designed to obtain reproducible single-shot laser-pump/X-ray probe data, allowing to follow the compression and release paths of the Ni sample by changing the delay between the pump and the probe. The target consisted of a 4 µm or 10 µm thick foil of poly-crystalline nickel (Goodfellow, >99,9% purity). The foil was glued to either a 50 µm black Kapton or a 20 µm nano-crystalline diamond ablator on the laser side. On some shots, a 100 µm thick LiF window was also glued on the rear side of the sample in order to maintain the pressure longer. The shock amplitude was tuned by varying the input laser energy (from 6 to 30 J) and the focal spot (250 or 100 µm). For each experiment, the shock states were characterized by velocity interferometry diagnostic (VISAR).

As shown in Figure 1, the single shot XAS spectrum nicely reproduces the features of the reference spectrum measured on the BM23 scanning XAS beamline.

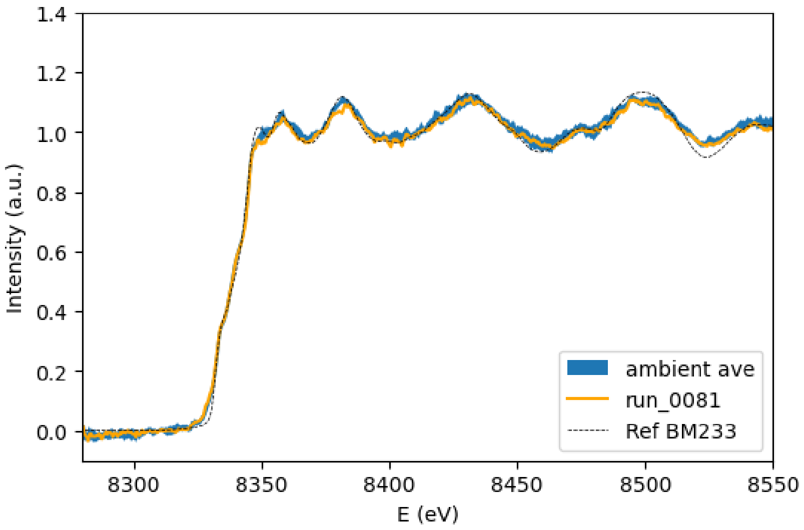
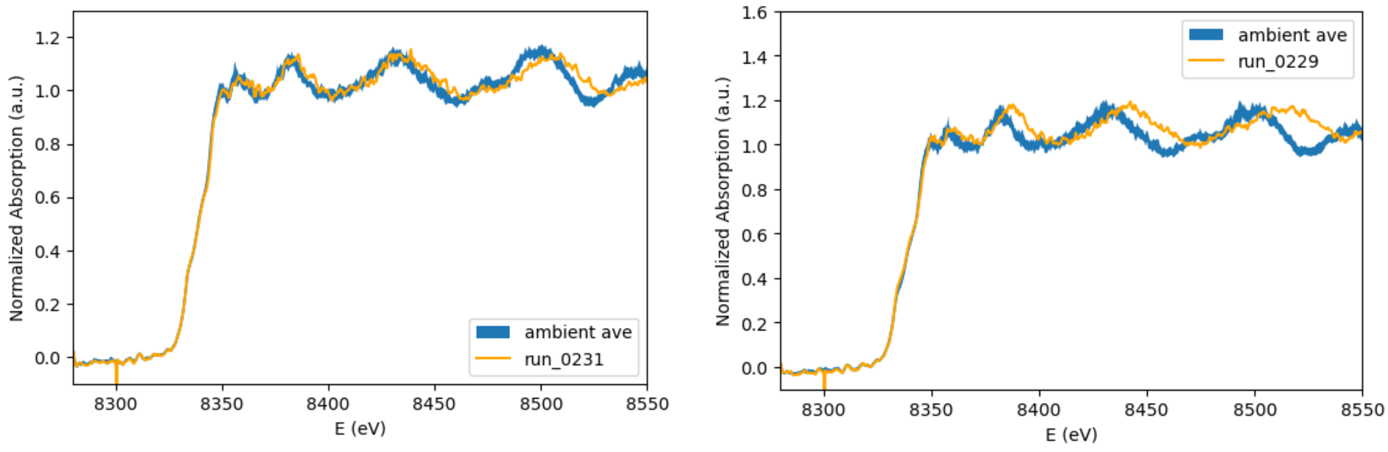


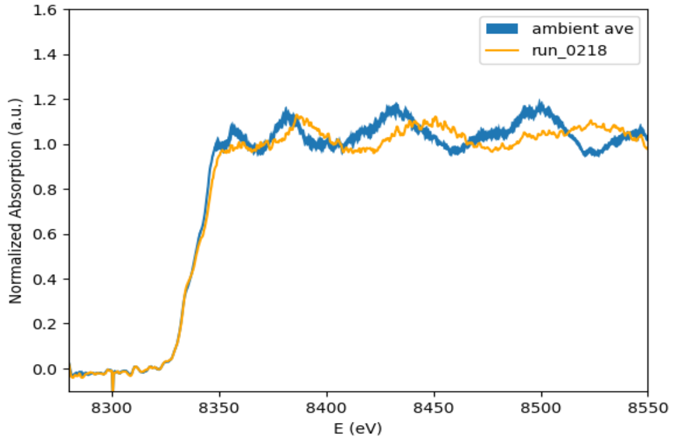
Figure 1: Comparison of the ambient average XAS spectrum (average of 10 spectra) and a single shot cold XAS spectrum recorded on ID24 dispersion beamline, with the reference Ni spectrum recorded on the BM23 scanning beamline.

As the pressure increases, one can see in Figure 2 that the peaks of the fcc Ni tend to broaden and shift to higher energies. These effects correspond to interatomic distance compression and increase of temperature respectively, in agreement with what was previously observed in both static (Boccato *et al*) and dynamic compression (Torchio *et al*) experiments. We did not observe any phase transition from the initial fcc Ni phase, but this conclusion should be mitigated given that we could not fully sample the required phase diagram region. This was due to an experimental issue. The glue layers used to bond the samples were burnt by the X-ray beam resulting in a modified hydrodynamics.



~18 GPa

~40 GPa



>50 GPa

Figure 2: Evolution of the XAS spectra as the shock pressure increases.

As a consequence, the reproducibility of the experiments was quite poor, and it was almost impossible to probe at a given delay, due to the spreading of the shock breakout resulting from the debonding of the ablator and Ni layers. Further experiments are therefore required to probe all the intermediate delays and conclude whether or not any solid-solid phase transition occurs. In order to avoid the chronometry problems encountered during this first experiments, future experiments should be performed with deposited Ni samples. Deposited test targets have already proved to work properly, moreover, a fast and synchronized shutter will be installed before the target to avoid radiation damage.

We observed the occurrence of partial melting above 50 GPa, but unfortunately, we could not reach higher pressures due to a problem with the HPLF laser system, which had a maximum energy limited to 30 J instead of 50 J. Therefore, we could not probe the full melting of the Ni layer. This problem was fixed in the following weeks, and an energy upgrade above 50J is foreseen in 2023.

As a conclusion, we obtained interesting results during this first experiment which demonstrated that very good quality XAS spectra can be obtained under laser shock compression with the HPLF laser system. Unfortunately, we were not able to achieve all of our goals due to target and laser energy issues, but we plan to submit a new proposal, to perform new experiments with full energy capability on Ni deposited samples.