HC-879 experiment report

A first attempt of a laser shock experiment using time resolved synchrotron radiation XRD as probe was performed on ID09b at ESRF on samples of Fe and Bi.

The study of dynamically compressed materials using laser generated shocks in the ns range attracts nowadays a considerable interest because of the complementary information it can bring to the static studies (phase transitions path and dynamics, nucleation processes) and because of its capability to explore more extremes regimes. However, this science has been so far carried out at the kJ lasers: large dedicated facilities, where the technique used to produce the x-ray probe (k α -k β emission from a laser shot plate) features a poor brilliance. The use of synchrotron radiation (SR) will make this kind of measurements easier, more accessible and with better quality x-ray signals, possibly allowing to address subtle physical issues such as transition kinetics or texturization.

The aim of this experiment was thus to demonstrate the feasibility of this kind of measurements exploiting the capabilities of beamline ID09b in performing time resolved XRD to observe solid-solid structural transitions in the 0-20 GPa range.

The beamline is equipped with a ns laser featuring an intensity of 350mJ in gaussian pulses of around 5ns FWHM, and a focal spot of around 260 μ in diameter (as estimated from the sample damage) corresponding to a maximum power of 1.3 10¹¹ W/cm². The laser can be synchronized to the XR pulses with a required delay.



Fig1: schematic view of Fe and Bi targets.

Samples and hydrodynamic simulations

In this experiment we use direct laser ablation to induce a dynamic loading. Rapidly after the start of the laser pulse, the hit surface is heated to plasma conditions. The formed plasma then absorbs the incident laser energy and expands inducing an elevated pressure in the remaining material, i.e. driving a shock wave.

The samples were commercial Fe and Bi foils (from Goodfellow) coated on both side with CH-N and CH-D ablators and a fine layer of aluminium. The impedance mismatch at the boundary between the metal and the plastic ablator allows to increase the sustained pressure and the Al layer prevents from early laser transmission to the sample.

For the Fe samples plastic deposition on the back side could be etched to allow interferometry measurements of the surface velocity (see next section). This was not possible for the Bi foils which are furnished by Goodfellow with a Polyester support that cannot be detached without damaging the samples.

Hydrodynamic simulations using the code ESTHER predict that with the given laser power, the bcc-hcp transition in Fe (\approx 13 GPa) and the I-III and I-V transitions in Bi (2,5 and 6 GPa respectively) should be observable (Fig.1). The shock in the sample is sustained for a limited time before being extinguished by reflection waves from the rear and the ablated surface and by the laser release wave. According to our simulations for the given laser temporal shape and power, 50% of the sample should remain shocked for around 2 ns.





Fig2: a) simulated laser profile; b) hydrodynamic simulation for a sample of Al-CHN-Fe; c) reachable regions of the Bi phase diagram (green and blue are for CHN-Bi and CHD-Bi samples respectively).

Visar measurements

A VISAR (Velocity Interferometer System for Any Reflector) system was brought and installed on the beamline to check the samples response to the laser.

In particular, VISAR measurements of the reflecting surface velocity allow to re-trace the pressure in the shocked sample, according to the following lows:

$$P = \rho_0 D u = \rho_0 (c_0 + su) u \qquad u = v_s/2$$

Where u is the particle velocity, v_s is the surface velocity, D is the shock velocity, s is a sample parameter, ρ_0 is the ambient density and c_0 is the sound speed.



Fig.3: VISAR measurements of the surface velocity for Fe foils (left) and Al-CH-Fe targets (right)

VISAR measurements were performed both for Fe foils and for Al-CH-Fe targets.

The shock detected by the VISAR (1-4 GPa) is much lower than expected from the simulations and is not very reproducible. This is likely because of laser breakdown, i.e. the laser energy is lost in air and the plasma forms before the surface. This evidence points out that even though the laser energy is not very high, this experiment requires a small sample vacuum chamber. Nevertheless, the laser-induced compression should be observable in the XRD measurements.

XRD static and dynamic measurements

XRD measurements were performed at E=15 keV using the beam from the U17 undulator with the Ru multilayer, characterized by \approx 3% bandwidth and pulse flux of 4.10⁸ ph/pulse.

This experiment is performed using pump-probe method: the samples are initiated by laser pulses and the ensuing structural evolution recorded with XR "flashes" at a given delay.

Most importantly static XRD tests on our targets demonstrate that a clear diffraction pattern can be obtained with few or even one single XR pulse. This result is very encouraging as the data quality is much higher than in previous laser-shock experiment using in-situ XRD where the XR probe is obtained by emission.

CHN24µm Fe15µm



1 XR pulse

СНN 17µm Вібµm



2 XR pulses

Fig.3: XRD static measurements for a target of Fe (left) and Bi (right) using 1 and 2 XR pulses respectively.

Dynamic XRD measurements were performed with laser-XR delays between 5,8 and 14,2 ns in steps of 2,6 ns. The instrumental time resolution in a pump and probe experiment is the convolution of the laser and the XR pulse length and their relative jitter; the time step with which the laser can be moved is calculated by a macro.

Because the laser damages the target, the sample needs to be moved in order to perform multiple XR pulses acquisition. The macro on ID09b allows to move one sample holder motor (horizontal or vertical) in synchronization with the XR and the laser pulse and to sum up the acquisitions along the movement on a single image.

Our XRD images were collected summing over 2-10 XR pulses while the sample was moved by 500 μm at 1Hz.



Fig.4: schematic view of laser-XR tested delay.

Several problems were encountered during the dynamic combined laser-XR measurements:

- 1) for all the targets we observed that the plastic ablator detaches after the first laser hit. Once the plastic is detached the ablation is ineffective therefore summing up many laser-XR pulses on an image plate has likely hidden any structural effect from the first pulse.
- 2) after the observation in point 1) we tried to perform a single shot experiment, so we modified the macro in order to have one single laser-XR pulse. Nevertheless the system never gave less than 2 pulses even though the log.file reported that it had performed one pulse per image.
- 3) sometimes the laser did a discharge before starting the macro, damaging the sample in the initial position. To avoid this problem we let the discharge occur on the sample holder.
- 4) the Fe target likely moved towards the CCD because of the laser shot (see below).

In the Fe targets no compression (peak movement towards high angles) is observed but rather the opposite effect (peak movement towards low angles) which is likely due to the sample movement towards the CCD after the laser shot. In Fig.5 top panel, an example of this is shown for the (110) reflection.

Bi targets were more stable thanks to the thick polyester support, XRD reflection movement towards low angles was never observed.

Moreover when only 2 pulses were summed – thus limiting the effect described in point 1 - a slight compression of $V/V_0= 0.993$ could observed (Fig.5 bottom panel). Using the Bi equation of state from McMahon et al. PRL 2000, this corresponds to a pressure of 0.2-0.3 GPa.



Fig.5: dynamic XRD measurements. Top: (110) reflection from a Fe target; bottom: (012) reflection from a Bi target. We are currently try to model the laser breakdown to check if this result is compatible with the VISAR observations.

Conclusions, plans for future experiments and problems to solve

-Despite the problems encountered the observation of a small compression in Bi is an encouraging result and demonstrate that this kind of experiments are feasible.

-We are designing a small **sample vacuum chamber** so to avoid laser breakdown. We will have to consider the available space and the optical needs (focalization lenses...).

-We are planning **off-line LASER-VISAR experiments** with another laser to validate the target design and simulations. However for the success of a future experiment it will be essential to foresee similar experiments with the laser integrated on the ID09b beamline. These tests are very important to understand the sample response to the laser shots.

-Thanks to the high flux and resolution of ID09b, which allows for a sufficiently clear XRD image with only one XR pulse and considering the above mentioned laser damage on the plastic ablator, it seems that the best way to perform the experiment is in single shot.