8 P-V-T-ε Materials Structure Science Project

– To bridge a gap between static- and shock-compression experiments –

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8-1 Introduction

Both static- and shock-compression experiments have a long history, and have been closely related to each other. For instance, Hugoniot compression curves measured under shock compression have been used as a pressure scale in static-compression studies. However, significantly different phenomena have been often observed in the two types of experiments for the same samples, and therefore some researchers consider that it makes no sense to compare them. For example, increases in electrical conductivity of some insulators observed under shock compression have not been observed under static compression and some phase transitions observed under static compression have not been observed in recovered samples after shock compression. These discrepancies are likely due to a large difference in strain rate during compression: 10^{-6}–10^{-1} s^{-1} during static compression and 10^{6}–10^{9} s^{-1} during shock compression. There has not yet been enough cooperation among researchers engaged in static- and shock-compression experiments and the relation between the strain rate and the changes in structure and properties is not understood sufficiently.

Synchrotron X-ray techniques are powerful tools for studying the effect of strain rate on the behavior of materials. High-pressure synchrotron XRD under static compression has yielded many important results and significantly contributed to the development of high-pressure science since the 1980s. On the other hand, shock-compression experiments with synchrotron X-rays are still in an early phase of development. There is an urgent need to develop time-resolved XRD with a shock-wave driven laser pulse.

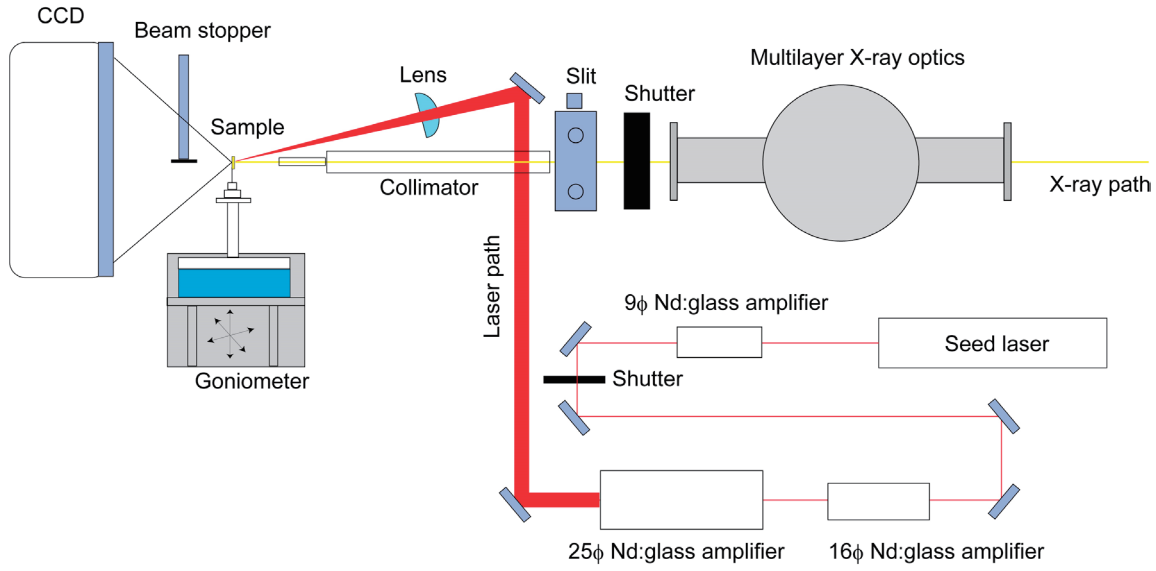

Fig. 1: Schematic drawing of the single-shot time-resolved X-ray diffraction system. The red line and yellow line are the laser and X-ray path. The seed laser is a Nd:YAG laser synchronized with the divided rf master clock from 794 kHz to 9.46 Hz. A single amplified laser pulse of 16 J is taken by the solenoid shutter.

In this project, we have gathered a group of researchers specialized in static- and shock-compression experiments, and are developing measurement systems and conducting XRD, XAFS, and other measurements systematically under static and shock compression. We are mainly focusing on phenomena which need an understanding of the time evolution and/or inhomogeneity, such as the collision of asteroids, mantle convection, and seismic activity (in geophysics) and the deformation and fracture of metals and ceramics (in materials science). The kick-off meeting was held in January 2016 and gathered a total of 34 researchers specialized in static- and shock-compression experiments and XAFS measurements.

8-2 Upgrade of single-shot time-resolved X-ray diffraction system under shock compression

Time-resolved X-ray diffraction, combined with the shock-wave driven laser pulse and short X-ray pulse, is a powerful tool for understanding the high-speed structural changes of condensed matter under shock compression. The measured signal from the shock-compression state cannot be accumulated repeatedly because the sample is completely broken after shock compression. Therefore, the high-flux X-ray pulse is extremely useful to observe the irreversible structural change under shock-wave loading. The PF-AR is operated in single-bunch mode throughout the year. The combination of a nanosecond high-power laser and a picosecond X-ray pulse from the PF-AR is suitable for studying shock-wave induced structural dynamics, such as shock-induced phase transition, elastic-plastic deformation, and shock fracture.

Previously, we used a two-step amplified Nd:YAG laser with a maximum energy of 1 J/pulse for driving a shock wave into the sample [1-3]. The value of shock pressure depends on the power density (W/cm^2) of the laser pulse. We have recently installed a high-power Nd:Glass laser system (NPG760, Continuum Co. Inc.) at the experimental hutch of the NW14A beamline of the PF-AR. The Nd:Glass laser is composed of a Q-switch Nd:YAG laser oscillator system and three-step amplifier components (Fig. 1). The amplifier system consists of φ9, φ16 and φ25 mm rods of Nd-doped laser glass surrounded by flashlamps. The pulse width, wavelength, and maximum pulse energy are 12 ns, 1.06 μm, and 16 J/pulse, respectively. The Nd:YAG oscillator is synchronized with a divided 508 MHz rf master clock at 9.46 Hz. The delay timing between the laser pulse and the X-ray pulse is changed by a digital delay generator (DG645, Stanford Research System, Inc.). An amplified laser pulse is taken from the pulse train using a solenoid shutter. The amplified laser pulse can be fired with a minimum time interval of about 15 min. The beamline provides a white X-ray pulse in the 13–18 keV energy range using an undulator with a period length of 20 mm and with a pulse duration of about 100 ps. It has been possible to make single-shot time-resolved X-ray diffraction measurements with the upgraded system since the spring of 2016.

8-3 Development of synchrotron X-ray multiple-measurement system under static compression

XAFS enables us to selectively probe the electronic state (i.e., valence) and short-range structure (i.e., bond length and coordination number) of a specific element contained even dilutely in a sample. On the other hand, XRD enables us to probe the long-range structure (and intermediate-range structure) of the host phase. These complementary measurements provide information on the inhomogeneity and its structural scale.

Upgrading the NE5C beamline of the PF-AR, we have developed a high-pressure in-situ XAFS-XRD measurement system with a world-famous large-volume press, called “MAX80”. The monochromator system was reactivated and confirmed to provide monochromatic X-rays at 10–50 keV. The XAFS measurement system can be switched to the XRD measurement system with white X-rays within about 5 minutes by moving the first crystal of the monochromator. To test the system, high-pressure XAFS-XRD measurements at room temperature were conducted on tellurium. Figure 2 shows the XAFS spectrum measured at 4.4 GPa. The pressure dependence of bond length estimated from the XAFS spectra is consistent with those from the XRD patterns and in the literature [4].

SAXS enables us to observe the structure of materials over a nanometer scale. A high-pressure in-situ small-angle and wide-angle X-ray scattering measurement system was developed at the BL-18C beamline of the PF to observe the intermediate state during a pressure-induced

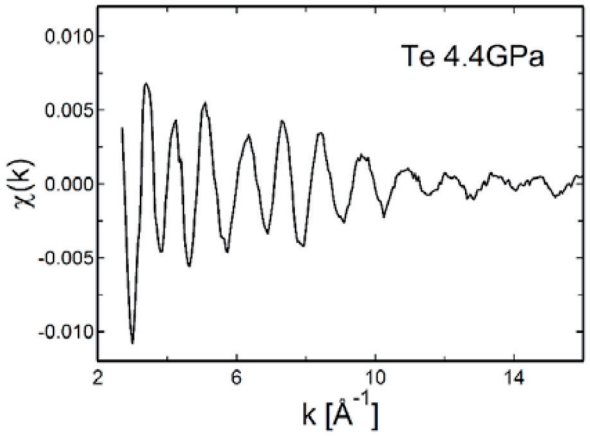

Fig. 2: XAFS spectrum of tellurium at 4.4 GPa.

phase transition in non-crystalline materials. An additional collimator was set between the sample and the X-ray shaping collimator in order to remove scatterings from the shaping collimator. A vacuum chamber was installed downstream of the sample to remove scatterings from air. Imaging plates can be set to the positions for small-angle scattering (camera length ~1250 mm) and wide-angle scattering (camera length ~170 mm) in the chamber. High-pressure in-situ measurements on SiO_2 glass were conducted with a diamond-anvil cell and revealed that the low-Q scattering intensity shows a maximum during the phase transition between fourfold- and sixfold-coordinated amorphous polymorphs. Detailed analysis suggests that inhomogeneity at a sub-nanometer scale (~7 Å) exists in the intermediate state [5].

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