

NANO-SECOND TIME-RESOLVED SYNCHROTRON X-RAY DIFFRACTION STUDY OF OLIVINE UNDER LASER-INDUCED SHOCK COMPRESSION. T. Mikouchi¹, K. Ohsumi², K. Ichiyanagi³, S. Adachi^{3,4}, S. Nozawa³, S. Koshihara^{3,5}, and M. Zolensky², ¹Dept. of Earth and Planet. Sci., Univ. of Tokyo, Hongo, Tokyo 113-0033, Japan, ²KT, NASA-JSC, Houston, TX 77058, USA, ³ERATO, Japan Sci. and Tech. Agency, Tsukuba, Ibaraki 305-0801, Japan, ⁴High Energy Accelerator Res. Org. (KEK), Oho, Tsukuba, Ibaraki 305-0801, Japan, ⁵Frontier Res. Center, Tokyo Inst. Tech., Oh-okayama, Tokyo 152-8551, Japan (E-mail: mikouchi@eps.s.u-tokyo.ac.jp).

Introduction: Shock experiments on silicate minerals are commonly performed by light gas guns or high explosives, which provide important calibration data on the occurrence of shock effects in certain pressure ranges, and are used to understand shock metamorphism in extraterrestrial materials [e.g., 1]. However, the duration of the pressure pulse by these experimental techniques is within a similar range ($\sim 1 \mu\text{s}$), and much shorter analytical time scales are necessary to clarify shock-induced transient lattice dynamics and the fundamental formation kinetics of shock effects in silicate minerals. Laser shock experiments are a powerful tool to generate shock waves on the order of nano-seconds [e.g., 2]. The recent development of a high-flux white X-ray pulse from synchrotron radiation has permitted *in-situ* nano-second time-resolved X-ray diffraction analysis to understand the lattice dynamics under a high-strain field generated by a synchronized laser pulse [e.g., 3]. We employed this technique to study shock deformation of olivine during nano-second durations, and here report preliminary results.

Sample and Method: The experiment was performed by the pump-probe single-shot Laue diffraction system at the NW14A beamline at the Photon Factory Advanced Ring (PF-AR), High Energy Accelerator Research Organization (KEK), Tsukuba, Japan [3]. The detailed setup at the NW14A beamline can be found in [4]. We prepared thin slices ($\sim 100 \mu\text{m}$ thick) of a well-terminated olivine single crystal (Gilgit, Pakistan: Fo₉₀) cut perpendicular to *c* axis. The typical slice size is a few mm across, and the diamond-polished surface was coated by Pt-Pd ($\sim 1 \mu\text{m}$ thick) as an ablation layer by laser. The slice was covered with the film and mounted on a glass capillary. The experimental configuration is shown in Fig. 1. The synchronization of X-ray and laser pulses is based on the RF master clock that drives the electron bunch in the storage ring. The pump laser is a Nd-YAG laser (Continuum, Powerlite8000), and the frequency, wavelength, pulse width, and the maximum energy of this laser are 10 Hz, 1064 nm, 10 ns, and 860 mJ, respectively. The probe white X-rays are from an undulator with a period length of 20 mm (U20) [4]. The peak is at 16 keV with the gap of 11 mm. The $\Delta E/E$ and photon flux of the probe X-rays are around 15% and 10^9 photons/pulse, respectively. The external trigger of the YAG laser is given at 9.45

Hz, which is divided from the RF master clock. We controlled delay timing between the X-ray and laser pulses by the delay generator (DG535, Stanford Research System, Inc.). The laser and the X-ray were focused down to $300 \mu\text{m}$ and $200\text{--}250 \mu\text{m}^2$ on the sample target surface, respectively.

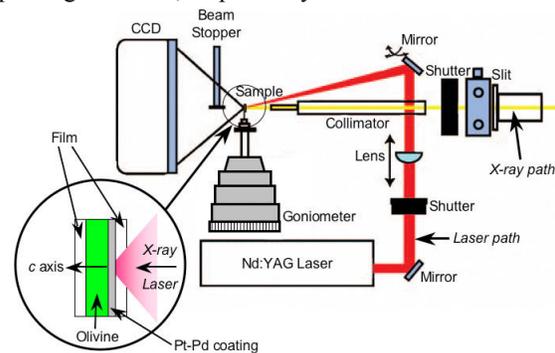


Fig. 1. Schematic illustration of the pump-probe single-shot Laue diffraction system developed at the NW14A beamline, PF-AR, KEK [3].

Results: At first, we obtained Laue diffraction images under shock loading with a 6 ns delay time with laser energy of 50 mJ, 100 mJ, 200 mJ, and 500 mJ. These laser energies correspond to shock pressures of 1.2, 2.0, 3.4, and 6.5 GPa, respectively [5]. All obtained patterns display a mixture of sharp diffraction spots with streaks (6 ns of Fig. 2). Because the sample thickness was about $100 \mu\text{m}$, ~ 15 ns is required for the shock pulse to travel from the ablation surface to the other side of the sample. Therefore, the sharp diffraction spots are from unshocked areas where the shock wave has not yet reached, and the streaks are from the shocked areas near the ablation surface. Although the experimental energy range covers a wide range of shock pressures, there are no significant differences in diffraction patterns among shocked samples analyzed.

We next performed a series of X-ray diffraction exposures with variable delay times with the laser energy at 50 mJ; the resultant images are shown in Fig. 2. The observed shift depends on the laser-induced shock-loading process that propagates into the sample from the ablation surface. Because each laser-pulse run destroyed the samples, Fig. 2 was constructed from six different runs with different delay times. The images obtained from 6 ns and 12 ns experiments clearly show

the presence of sharp diffraction spots with streaks, which result from the developing shock pulse in the olivine crystal. By contrast, the image from 30 ns displays broad diffraction patterns. This is probably due to partial decompression after the entire sample was shocked. This observation implies achieved shock pressure lower than 15 GPa (Hugoniot Elastic Limit (HEL) of olivine [e.g., 6]), consistent with the calculated pressure (1.2 GPa) by this laser energy (50 mJ).

Discussion and Conclusion: This study demonstrates that *in-situ* nano-second time-resolved X-ray diffraction analysis of olivine is possible by synchronization of X-ray and laser pulses, and we could successfully obtain 0-30 ns Laue diffraction images. The time-resolved analysis of shocked olivine by the 50 mJ laser (1.2 GPa) revealed the developing shock deformation in olivine. This shock pressure is close to the estimated shock pressure of Stardust particles during aerogel capture (~1 GPa) [7]. In this preliminary experiment, we did not obtain a delay time longer than 30 ns, and could not observe the diffraction pattern when the sample was completely decompressed. In a future experiment, we plan to take longer delay time shots, which

will reveal cell parameter change (if present) of olivine by capture heating and shock [8,9].

To our knowledge, this is the first *in-situ* nano-second time-resolved X-ray diffraction analysis of a natural silicate mineral. This system can be applied for other important rock-forming minerals to understand their properties under shock-compression conditions. Furthermore, the system can be used for other time-resolved X-ray analysis such as scattering and absorption experiments [4], and will be a powerful tool to further characterize shocked materials.

References: [1] Bischoff A. and Stöffler D. (1992) *Eur. J. Mineral.*, 4, 707-755. [2] Langenhorst F. et al. (1999) *Earth Planet. Sci. Lett.*, 173, 333-342. [3] Ichihyanagi K. et al. (2007) *Appl. Phys. Lett.*, 91, 231918. [4] Nozawa S. et al. (2007) *J. Synchrotron Radiat.*, 14, 313-319. [5] Fabro R. et al. (1990) *J. Appl. Phys.*, 68, 775-784. [6] Syono Y. et al. (1981) *J. Geophys. Res.*, 86, 6181-6186. [7] Zolensky M. et al. (2006) *Science*, 314, 1735-1739. [8] Foster N. J. et al. (2007) *Meteorit. and Planet. Sci.*, 42, A51. [9] Ohsumi K. et al. (2008) *LPS XXXIX*, Abstract #1808.

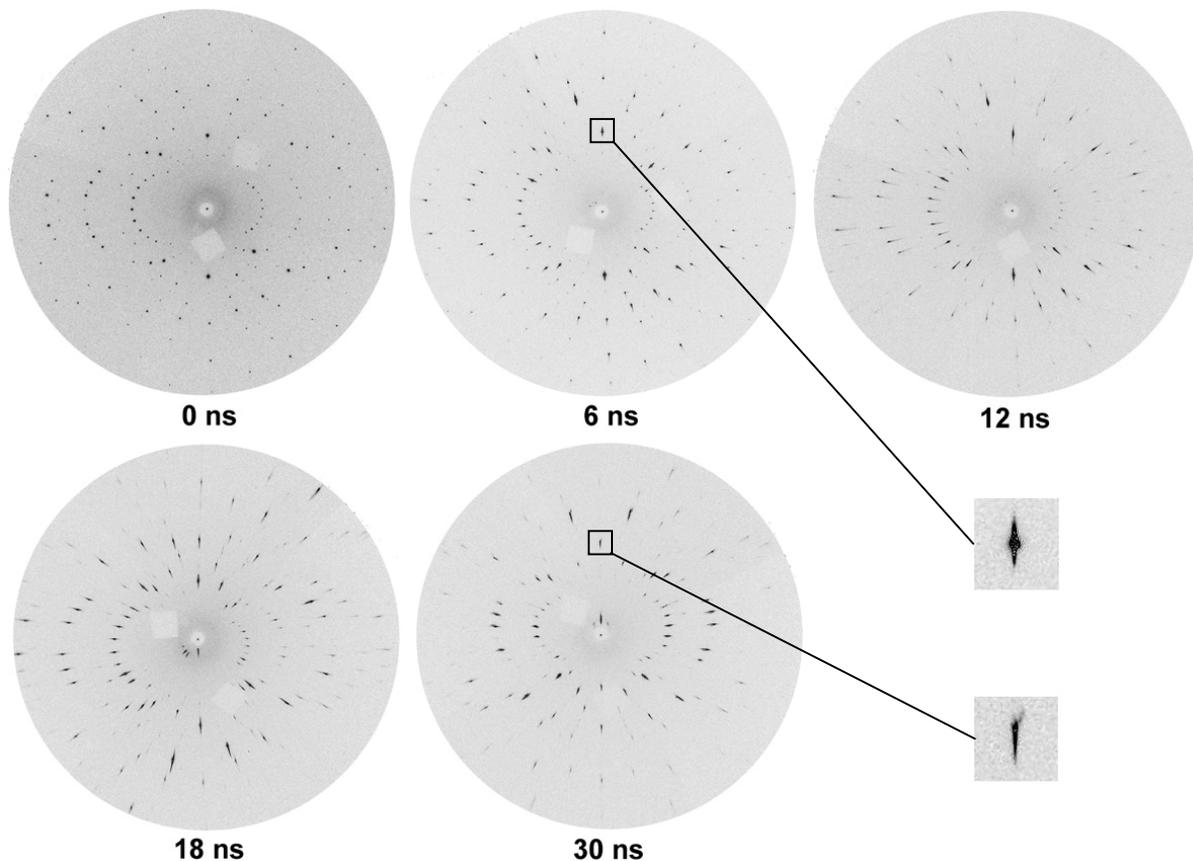


Fig. 2. Laue patterns of the olivine single crystal by time-resolved synchrotron X-ray diffraction measurements. The data at each delay times are taken as a snapshot. The delay times are 0, 6, 12, 18, and 30 ns. The enlarged reflection from 6 ns and 30 ns show different characteristics corresponding to microstructural changes.