Newly Developed Time-Resolved Dispersive XAFS System for Laser-Driven Irreversible Processes

The time-resolved dispersive XAFS (DXAFS) system for irreversible processes has been developed at the AR-NW2A. Combining the DXAFS system that can detect one pulse X-ray of duration ~ 100 ps emitted every 1.26 µs from PF-AR with a high power pulsed laser enabled elucidation of laser-driven irreversible processes with nanosecond to subnanosecond time resolution. We applied the system to the laser compression and laser ablation process of metals as an irreversible process, and succeeded in obtaining the time evolution of the processes under extreme conditions from the change of XAFS spectra obtained. In the case of laser compression of copper, it was found that copper was compressed by about 2% in volume after 4 ns from laser irradiation and the pressure was released over 200 ns.

In order to use various materials in real environments, it is necessary to understand and control "irreversible processes" such as fracture, fatigue, and corrosion, which are often accompanied by changes of microstructures and crystal structures caused by diffusion or phase transitions. Though a number of studies on the destruction mechanism of materials have been conducted in order to improve their mechanical properties, most of them are based on ex situ observation of the static state of materials that have already been destroyed, and so an understanding of the mechanism remains limited. Thus, the real-time observation of "irreversible processes", so-called "in situ" observation, remains indispensable for revealing their mechanism and controlling the macroscopic properties of materials after the processes. Real-time observations of "irreversible processes" using the X-ray diffraction technique were recently reported. Ichiyanagi, et al. reported the laser shock-induced lattice response and photo-induced protein reaction by using the time-resolved single-shot X-ray diffraction technique at AR-NW14A [1, 2]. However, structural information in the short range order of materials at the moment of fracture has never been reported, though a high degree of disorder at such moment is expected.

We have developed an *in situ* and time-resolved observation technique using DXAFS for achieving this challenging task at AR-NW2A. We focused on destruction and phase transition among irreversible reactions, which are accompanied with atomic diffusion and/or displacement with a nano-second time scale. The XAFS spectrum of the whole X-ray energy range required can be measured simultaneously without any mechanical movements using the DXAFS technique [3]. For this reason, the whole XAFS spectrum is acquired rapidly with a single shot X-ray pulse by DXAFS measurement using an appropriate X-ray detector.

In our new DXAFS system, destruction or phase transition is triggered by a high-power Q-switch Nd:YAG pulsed-laser (Continuum Powerlite8000) that is synchronized with an X-ray pulse (Fig. 1). PF-AR is a unique synchrotron light source operated in the single bunch mode at all times, and it is particularly suited for time-resolved experiments. A silicon microstrip detector, XSTRIP [4, 5], specifically modified for PF-AR was used as a one-dimensional position-sensitive detector. Since it can detect one pulse of X-ray from PF-AR, the short-est time resolution of the system corresponds to the X-ray pulse duration of ca. 100 ps (FWHM).



Figure 1: Microscope images of sample before (a) and after (b) laser irradiation; conceptual diagram (c) and photos of DXAFS system developed (d), (e).



Figure 2: The EXAFS oscillations weighted by k^3 (a) and the radial structure functions (b) of the copper at various delay times. Solid and dashed lines indicate observed and calculated values for the first shell curve in both figures, respectively.

We have used the developed technique to investigate the laser-induced shock compression of copper. Polycrystalline copper foil of 5 µm thickness was used as the sample and was coupled with an ablator of 10 um-thick aluminum foil. The laser at 1064 nm was irradiated from the aluminum foil side. The shock wave from the aluminum foil applied a high pressure to the copper foil. The pressure was estimated as ca. 20 GPa using the Fabbro-Devaux model [6], though it is rather difficult to estimate a pressure caused by laser-shock because of its short rise time. It should be noted that the Fabbro-Devaux model was reported to be likely to be larger than the actual one. Because the copper foil was destroyed and disappeared (Fig. 1a and b) by the irradiation of the laser, we made disc-type sample changers (Fig. 1e).

Figure 2 shows the observed k^3 -weighted $\chi(k)$ values (a) and the radial structure functions (b) at the delay time of 4 and 200 ns after laser irradiation together with a reference spectrum without laser irradiation (Laser OFF). The solid and dashed lines in the figure indicate the observed and the best-fitted values considering the nearest Cu-Cu interaction, respectively. The coordination numbers, bond length and Debye-Waller factors of the nearest Cu-Cu interaction were obtained from EX-AFS analysis. It was found that the coordination numbers and Debye-Waller factors after laser irradiation are almost similar to the initial state. On the other hand, the nearest-neighbor Cu-Cu bond length of 0.254 nm at 4 ns after laser irradiation is shorter than that of 0.256 nm before laser shock. Then, it almost returned to the initial value of 0.257 nm at 200 ns after laser irradiation. This indicates that the copper after 4 ns laser irradiation is compressed by ca. 2% in volume on average and then the pressure is released at 200 ns. However, the peaks around 0.43 nm corresponding to the third-nearestneighbor distances in Fig. 2b showed a different behavior. The peak remained near that of the initial state (0.43 nm) at 4 ns, and shifted to a longer value (0.44 nm) at 200 ns. These results suggest that the change of

atomic-scale structure induced by the laser shock is not isotropic, in line with the expectation that laser shock produces uniaxial compression.

This kind of information on destruction mechanisms will be crucial for designing structural materials [7]. Furthermore, the developed technique can be applied to various "irreversible processes" that progress with a time scale of ns. For example, we can investigate the dynamics of irreversible structure changes under extreme conditions, such as phase transition, spin-state transition, and fragmentation.

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REFERENCES

- K. Ichiyanagi, S. Adachi, S. Nozawa, Y. Hironaka, K. G. Nakamura, T. Sato, A. Tomita and S. Koshihara, *Appl. Phys. Lett.* 91, 231918 (2007).
- [2] K. Ichiyanagi, N. Kawai, S. Nozawa, T. Sato, A. Tomita, M. Hoshino, K. G. Nakamura, S. Adachi and Y. C. Sasaki, *Appl. Phys. Lett.* **101**, 181901 (2012).
- [3] T. Matsushita and R. P. Phizackerley, Jpn. J. Appl. Phys. 20, 2223 (1981).
- [4] J. Headspith, G. Salvinia, S. L. Thomasb, G. Derbyshireb, A. Denta, T. Raymentc, J. Evansd, R. Farrowa, C. Andersona, J. Clicheb and B. R. Dobsona, *Nucl. Instrum. Methods Phys. Res. Sect. A* **512**, 239 (2003).
- [5] G. Salvini, J. Headspith, S. L. Thomas, G. Derbyshire, A. Dent, T. Rayment, J. Evans, R. Farrow, S. D. -Moreno and C. Ponchut, *Nucl. Instrum. Methods Phys. Res. Sect. A* 551, 27 (2005).
- [6] D. Devaux, R. Fabbro, L. Tollier and E. Barticki, J. Appl. Phys. 74, 2268 (1993).
- [7] M. Kimura, Synchrotron Radiation News 30, 23 (2017).
- [8] Y. Niwa, T. Sato, K. Ichiyanagi, K. Takahashi and M. Kimura, *High Press. Res.* 36, 471 (2016).

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