Development of time-resolved small-angle X-ray scattering system using soft-X-ray laser

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Abstracts: *We are developing time-resolved grazing-incidence small-angle scattering (GI-SAXS) system using a soft X-ray laser. As a first step, we carried out GI-SAXS measurements of optical grating and craters on gold surface produced by femtosecond laser ablation. We succeeded in observing the diffraction spots from the grating as expected, but the diffuse scattering from the craters was hidden in thermal noises of a soft X-ray CCD camera.*

The non-thermal (pressure) effect on femtosecond laser ablation has been focused on for development of high-precision micromachining technique with small thermal effect. Since the pressure is always zero at the surface due to boundary condition, the maximum tensile stress is generated not at the surface but around the center depth in the photo-excited volume. The upper part of the photo-excited volume is separated as a thin-layer by merging voids which are generated by aid of the tensile stress. The ablation phenomena is usually called by the spallation in the femtosecond laser ablation dynamics. The spallation dynamics has been studied using time-resolved X-ray microscopy. Nishikino et al. [1] have observed time-resolved X-ray microscopic images and shadowgraphs of the separated thin-layers, and discussed the ablation dynamics in the viewpoints of thermodynamics and thermomechanics so far. Recently, we newly focused on a time-resolved grazing-incidence small-angle X-ray scattering (GI-SAXS) measurement on the femtosecond laser ablation. In principle, the GI-SAXS can measure time-evolution of the morphology of the voids, which would be stretched in normal to the sample surface by the tensile stress, whereas bubbles would be formed when the ablation is dominated purely by the thermal effect. We can quantitatively evaluate the contribution of the tensile stress on the femtosecond laser ablation from the asymmetric morphology of the voids. In this presentation, we report the result of the GI-SAXS measurement, and discuss the possibility and problem on the development of the time-resolved GI-SAXS system.

Figure 1 shows the experimental setup, which was slightly modified from X-ray imaging [1].

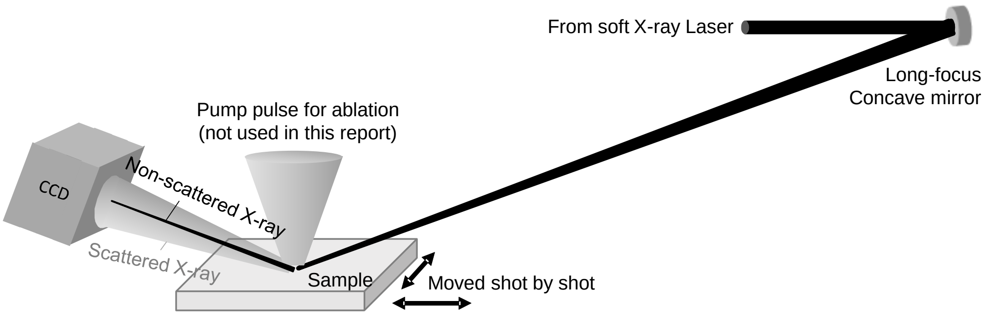

Figure 1 Experimental setup

A soft X-ray pulse with a wavelength λ = 13.9 nm, dispersion $\Delta\lambda/\lambda = 10^{-4}$, and time-duration of 1 ps from the soft X-ray laser in KPSI was guided through a 2-5 mm-thick zirconium filter and a concave mirror with a curvature radius of 3 m to a sample at an incident angle of 19.2° from the surface. The reflected pulse from the sample surface was detected using a soft X-ray charge coupled device (CCD) camera (Princeton, PI-MTE: 2048B) with the sample-to-camera distance of 124 mm. The X-ray pulse was focused to a diameter of approximately 50 μm on the CCD camera, whereas the beam diameters of an ellipsoidal spot at the sample surface was approximately 400 and 100 μm in the major and minor axes, respectively. We used the gold-deposited optical grating for visible and infrared wavelengths (1740 lines / mm) as a standard sample.

Figures 2(a) and (b) show the GI-SAXS profiles of the grating whose groove directions are parallel and perpendicular to the plane of incidence, respectively. The diffraction spots with the number *n* in the figures are attributed to the *n*-order diffraction peaks. Their positions are consistent with theoretical predictions (not shown). The full widths at half maximum of the diffraction spots are determined by the width of the 0-order diffraction pulse focused on the CCD camera, giving the minimum magnitude of the scattering vector *q* of the order of 10^{-4} nm^{-1}.

We tried to observe a GI-SAXS profile of an ablated crater on a femtosecond-laser-irradiated gold, but resulted in unsuccessful. As shown in Figure 2, thermal noise with dozens of counts remains per each pixel even after background signal is subtracted. This level is too large to pick up dispersed scattering signal from disordered system such as the crater. Since structural information is obtained from power-law scattering of intensity against scattering angle, much wider dynamic range of the scattering signal has to be obtained by decreasing the background signal.

References

[1] N. Hasegawa, M. Nishikino, T. Tomita, N. Ohnishi, A. M. Ito, et al.,Proceedings of SPIE 9589, 95890A-1-8 (2015).

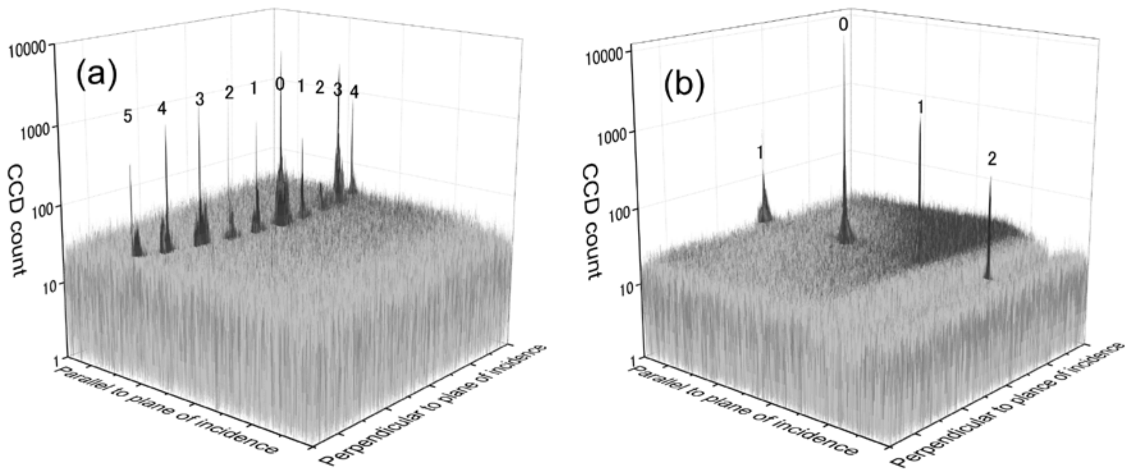

Figure 2 GI-SAXS profiles of the optical grating aligned in (a) parallel and (b) perpendicular to the plane of incidence. The image area is 27.6 x 27.6 mm.