Development of high resolution micro X-ray diffractometer for pinpoint structural measurements at BL40XU of the SPring-8

Haruno Murayama¹,², Nobuhiro Yasuda¹,², Jungeun Kim¹,², Yoshimitsu Fukuyama¹,², Shigeru Kimura¹,², Yoshihiro Kuroiwa¹,³, Yutaka Moritomo¹,⁴, Yoshihito Tanaka¹,⁵, Koshiro Toriumi¹,⁶, Masaki Takata¹,²,⁵
¹CREST, ²JASRI/SPring-8, ³Hiroshima Univ., ⁴Tsukuba Univ., ⁵RIKEN/SPring-8 Center, ⁶University of Hyogo

In order to characterize nano-materials and their ultrafast phenomena (e.g. amorphous-crystal change on recording DVD media and photo-induced metal-insulator transition), we have been developing high resolution XRD measurement technique. The measurements are named “X-ray pinpoint structural measurements” and the high resolution means nanometer spatial and pico-second time resolution. The pinpoint X-ray structural measurement system has been constructed on BL40XU at the SPring-8, which is the high flux beam line using a helical undulator. Single pulse X-ray of the SPring-8 can give 40 ps detecting time. Furthermore, combination of high precision goniometer and micro-focused X-ray by using Fresnel zone plate can give ~100 nm spatial resolution.

The pinpoint X-ray structural measurement system is composed of an X-ray pulse selector (XPS), a Si(111) channel-cut monochromator, a CCD detector, an imaging plate detector, a sample cooling system (T=90-300 K), and a femtosecond Ti:sapphire laser system. Figure shows the high resolution micro X-ray diffractometer. X-ray through a zone plate is focused on a sub-micron single crystal. The high precision goniometer has θ-2θ rotation stages and 100-nm-resolved XYZ sample positioning stages. Especially, the θ stage uses an air-bearing stage (Canon AB-100R), which is very low eccentricity within ±100 nm/360°. The high spatial resolution diffractometer can align micro-focused X-ray with a sub-micron single crystal, which is as small as a single grain of powder diffraction sample. A high resolution type zone plate, whose Ta thickness is 0.75 µm was designed to produce sub-100 nm beam by 8 keV X-ray. The ideal X-ray diffraction efficiency of the zone plate is 12 %.

The pump laser pulse initiates phase transition and the probe X-ray pulse takes snapshots of the transition process with time delay between the laser pulse and the X-ray. Intense X-ray is required, since repetitions of the pump-probe cycle are not desirable. Ideally the phase transitions are permanently reversible processes, however numbers of repetition cause irreversibility.

A high flux type zone plate, whose Ta thickness is 2.5 µm was designed for high X-ray diffraction efficiency at 15 keV. The zone plate achieved the diffraction efficiency up to 20 %. The beam size and flux density was 1.4×2.4 µm² and 3×10¹⁴ photons·s⁻¹·mm⁻², respectively. Data collection of sub-micron single crystal was performed and availabilities of X-ray micro-focusing by the high flux type zone plate is discussed.

Figure. sketch of the high resolution micro X-ray diffractometer.