L.6: Powder x-ray diffraction using focused laser plasma x-ray source

X-ray diffraction (XRD) is a standard tool to get the information on the crystal structure, which is responsible for various material properties. XRD can be done with either single crystal or powder sample. XRD of powder sample gives the diffraction peaks from all the possible crystal orientations simultaneously, whereas in single crystal, XRD from one orientation can be recorded at a time. However, the XRD signal intensity from the powder sample is several orders of magnitude smaller as compared to that from single crystals, due to diffraction at various angles from the powder sample. Thus, XRD from powder sample can be easily recorded using high average flux source, viz., x-ray tube source or synchrotron source, whereas it is difficult to record using laser plasma (LP) based x-ray source due to its low average flux. The above high flux sources have large pulse duration (tens of ps - few μs). On the other hand, the LP x-ray source has very short pulse duration (sub-ps). In addition to this, it has other unique properties such as table top in size, low operating cost and can provide x-ray pulse, which is well synchronized with the laser pulse. This makes it ideal candidate for time resolved x-ray diffraction (TXRD) studies, which is based on pump-probe technique. In TXRD, ultra-fast dynamics of the material is studied by recording the XRD from the crystal by ultra-short x-ray probe after excitation by ultra-short optical pump (laser). The delay between the pump and probe pulses gives the information of crystal structure evolution dynamics after excitation by pump laser beam. TXRD study with powder sample can provide much more information than TXRD of single crystals. One can get time-resolved mapping of phase transitions, charge relocation, etc. in the crystal. In order to proceed in this direction, experimental set-up is arranged and XRD pattern from the powder sample has been demonstrated using LP x-ray source.

When an ultra-short, high-intensity laser pulse is focused on a solid target, plasma is formed at the target surface and it generates hot electrons. These hot electrons interact with cold solid target behind the plasma and generate characteristic K_α and bremsstrahlung radiation. The x-ray flux emitted from laser plasma source was optimized by adjusting the laser pulse parameter such as laser intensity, pulse duration and pre-pulse, etc. Next, to increase x-ray flux on the powder sample, x-rays were collected from the point LP source and focused on the sample. Removing high energy x-ray bremsstrahlung, which creates background was a challenge, as it masks the XRD signal generated from the powder sample. In this report, the XRD pattern from standard Si powder sample using LP x-ray source is presented.

The experiment was carried out using 6.5 mJ, 50 fs, kHz Ti:sapphire laser. The laser beam was focused on a moving Cu wire target using f/8 off-axis parabolic mirror at an intensity of ~3.5×10^{16} W/cm^2. The emitted Cu K_α x-ray photon flux was ~1.2×10^{10} photons/sr/s, which was measured using CdTe detector in single photon counting mode. The emitted x-rays were focused using the polycapillary optics of 50 mm input and 100 mm output focal lengths with a focal spot size of ~500 μm of full width at half-maximum (FWHM). The focused x-ray beam was extracted from plasma chamber through 1 inch diameter, 25 μm thick kapton window.

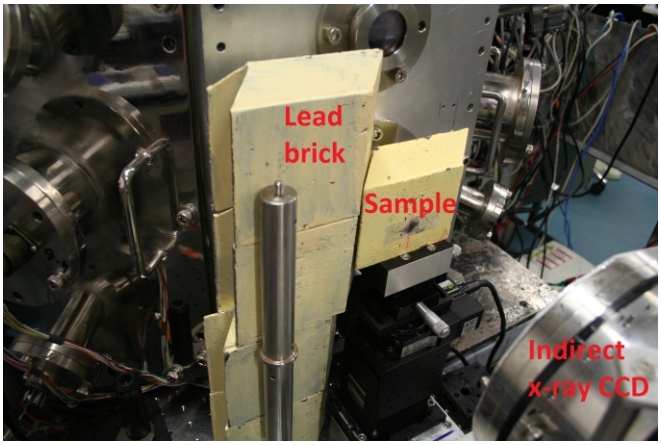

Fig. L.6.1: Photograph of experimental setup.

The powder sample was sandwiched between two kapton foils. The thickness of powder sample was optimized to achieve good diffraction signal and at the same time to reduce x-ray absorption inside the sample. To reduce the high energy background, the sample was mounted on 50 mm thick lead brick containing 2 mm hole at centre as shown in Figure L.6.1. The powder diffraction from Si powder sample was recorded on an indirect x-ray CCD cooled to -40 °C in transmission geometry, which was placed ~60 mm away from the sample. The recorded XRD pattern and its line profile are shown in Figure L.6.2(a) and Figure L.6.2(b), respectively. This diffraction pattern is recorded for 15 minutes of x-ray exposure. The diffraction pattern shows clear diffraction rings. The line profiles are generated by integrating the diffraction rings azimuthally. The powder diffraction pattern from the Si sample shows three peaks at 2θ angle of 28.4°, 47.6° and 56.4° corresponding to the lattice planes (111), (220) and (311), respectively. In future, this set-up will be used to perform TXRD study of powder sample.

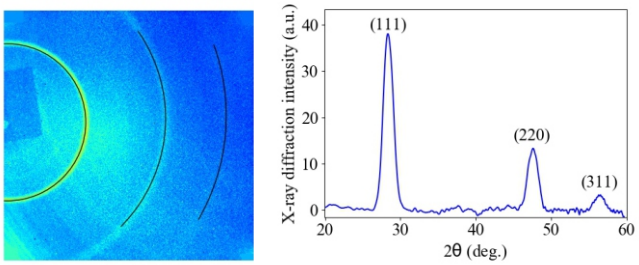

Fig. L.6.2: (a) XRD from Si powder sample recorded x-ray CCD, and, (b) x-ray diffraction intensity line profile.

Reported by:
R. Rathore (ranjana@rrcat.gov.in)