Submicron X-ray Diffraction Beamline

Most real materials consist of intrinsic and extrinsic microstructures of mesoscale size that ranges from tens of nm to tens of μm. Material properties depend not only on the local properties around these microstructures such as grain size, dislocations or stacking faults, grain orientation, and residual strain, but also on the evolution under heating, stress or bias. Discovering the fundamental material science associated with microstructures is thus essential for successful applications of these materials.

For this purpose, experimental methods with high spatial resolution and great accuracy, and of a non-destructive nature are required. With the advantage of synchrotron radiation, sub-μm focused X-ray diffraction has become of interest. On utilizing Kirkpatrick-Baez mirrors, sub-μm focusing of either a polychromatic or monochromatic synchrotron beam is achievable.

The diffraction of a polychromatic beam within a single spot basically results in a Laue diffraction pattern recorded with a pixel-array detector of large area. The positions of the Bragg spots in the Laue pattern provide information pertinent to the material identification and orientation, whereas the shape distortion reveals the deviatoric components of the strain tensor. Regarding diffraction with a monochromatic beam, accurate lattice parameters of a unit cell and the dilatation strain are extrapolated. On moving the specimen, a 2D mapping of crystallographic properties is thus demonstrated.

In our constructing beamline ID21, the simulated lateral spot size is about 100 nm × 100 nm for X-rays of energy from 5 to 30 keV. In addition to the sub-μm X-ray diffraction, several effective microscopic examination tools are installed at the end station, which operates under both high vacuum and ambient conditions, as shown in Fig. 1. For further detailed setup and application of this end station, please refer to reference 1.

The main component at the end station is the area detector (Pilatus3 6M, in vacuum), which collects diffraction signals. A scanning electron microscope is used to navigate to areas of interest. The installation of a silicon drift detector (SDD) and a quadruprobe sample system, one of a probe stage and holder as shown in the inset, provides various flexible experiments including 3D Laue diffraction, 3D X-ray fluorescence, near-field X-ray excitation optical luminescence, X-ray absorption, current-voltage from scanning probes, indentation, etc. Spontaneous investigation of structural, elemental, optical, electrical and mechanical properties are hence achieved at the same location on the

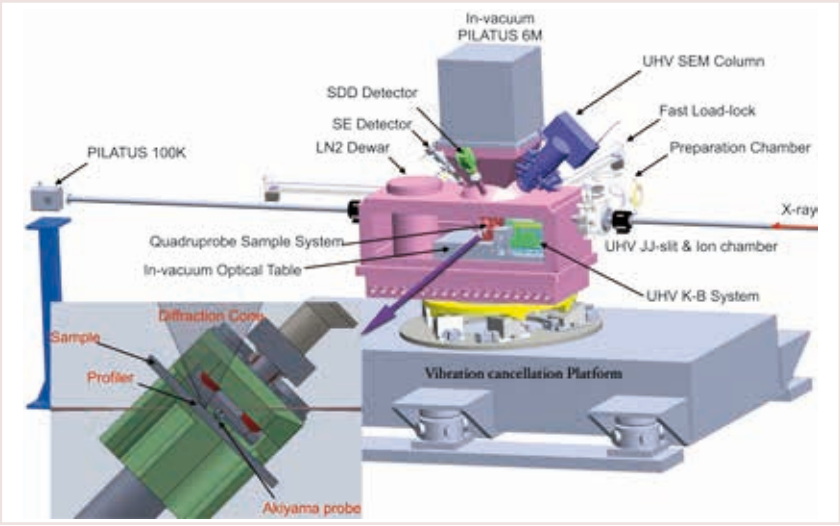

Fig. 1: Drawing of the TPS ID21 end station; the inset illustrates the SPM probe that assists a profile to achieve 3D Laue diffraction.

specimen, which helps scientists to develop the correlation principles. In addition, with use of the quadruprobe sample system and the preparation chamber, heating, external stress and electric field can be applied during the measurement; the growth of a thin film coating *in situ* is also practical. Scientists and engineers are helped to discover not only the growth mechanism in real time but also the working principles of a mesoscale device.

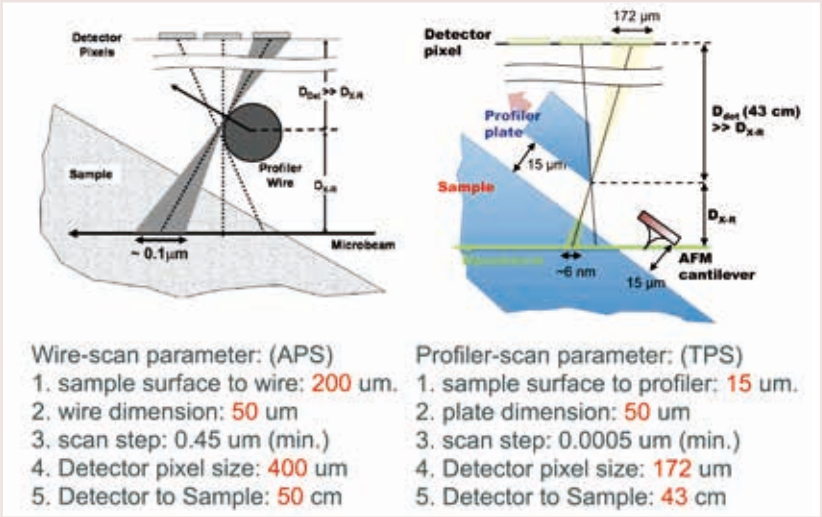

Fig. 2: Depth resolution of the benchmark of the DAXM facility at APS 34-ID-E and our end station.

In our design, two important features transcend the existing sub-μm diffraction facilities around the world, as discussed below.

The first feature is an improved spatial resolution of 3D Laue diffraction. A typical method to realize a 3D Laue pattern is to use a differential aperture X-ray microscope (DAXM). This technique is based on the large depth of penetration of hard X-rays; the reflections at varied depth contribute signals to the area detector. A metal wire that behaves as a differential aperture traverses the surface of the specimen, as shown in Fig. 2. As the wire travels to a specific position, some diffraction signals from particular depths are blocked in the Laue pattern. Analysis of Laue patterns taken with varied wire positions via an image reconstruction algorithm yields diffraction information at varied depths. In combination with 2D scanning and depth-resolved imaging, it yields 3D crystallographic information.

The greatest depth resolution of DAXM, 500 nm, is exhibited at Advanced Photon Source (APS), USA. In our design, we decrease the height of the wire specimen from 200 μm to 15 μm and the wire scanning step from 500 nm to 50 nm with use of a piezoelectric scanning probe system instead of a stepper motor used in APS. According to a geometric calculation, a depth resolution about 50 nm would be achieved. In brief, a 3D mapping of crystallographic properties with resolution 100 nm × 100 nm × 50 nm would satisfy most conditions of material science and engineering, especially for semiconductor thin films and nanocrystals, nano-scaled composites and ceramics, and the electrode connection in 3D very-large-scale integration.

The second important feature of our design is the 3D X-ray fluorescence. For most real cases, users not only are interested in structural information in a real material but also seek to realize the corresponding elemental evolution in the same region of analysis. The lateral spatial resolution of the fluorescence depends on the spot size of the focused X-ray, whereas the depth resolution depends on an orthogonal confocal detection system, which collects fluorescence signals along the penetration of incident X-rays. On utilizing a polycapillary together with a pinhole, sub-μm depth resolution of the confocal detection becomes achievable. The depth resolution is significantly improved from greater than 20 μm to the sub-μm range. In addition, as another SDD is employed to detect fluorescence, the detection limit of an element concentration would attain about 10 ppm. These conditions help scientists to discover the elemental distribution inside ceramics, composites and metals, or at the interface of biomaterials.

Reference

1. T. Feder, Physics Today **67**, 45 (2014).