BL24XU
Hyogo ID

1. Introduction

BL24XU is known as the Hyogo ID beamline. It is one of the contract beamlines designed by Hyogo Prefecture for industrial applications. It is a branched beamline employing a figure-8 undulator light source, a diamond (220) beam-splitting monochromator for branched line A, and a standard SPring-8 double-crystal monochromator (DCM) for mainstream B. The end-station is specialized for high-resolution structural characterization by microbeams and imaging (Table 1).

The BL upgrading of the end-stations and their promotion to industrial users are ongoing in cooperation with the University of Hyogo. A lensless nanoimaging technique called ptychography, a scanning variant of coherent diffraction imaging (CDI), has been open to users since FY2019. Recently, application studies in hard X-ray photoelectron spectroscopy (HAXPES), in collaboration with MAZDA Motor Corporation, have also been performed. Here, we report their research results of FY2020.

2. Lensless dynamic imaging for visualization of mesoscopic dynamics on the millisecond scale

Mesoscale structural heterogeneity and dynamics in materials and devices often play key roles in their properties. Ptychography is expected to be a key tool for visualizing mesoscale features at a spatial resolution of ~10 nm [1]. However, the temporal resolution of ptychography is limited to several tens of seconds because it reconstructs an object image through the phase retrieval (PR) of a set of coherent diffraction patterns collected by the lateral scanning of a focused X-ray beam (Fig. 1 left). Toward the visualization of mesoscopic dynamics on a millisecond scale, we developed

Table 1. Specifications of the measurement techniques in BL24XU.

Measurement techniques	Structural information	Spatial resolution
Projection/imaging microscope/coherent diffraction imaging (CDI)/CT	2D/3D image Field of view: 1 μm–1 mm Absorption, refraction contrast (projection/imaging microscope) Absorption, phase contrast (CDI)	10 nm–0.33 μm
Microbeam SAXS/WAXD/XRF	Periodic/aggregation structures of angstrom–several hundred nm Distribution of crystal grains Elemental mapping	0.5–5 μm
Bonse-Hart USAXS	Periodic/aggregation structures of 16 nm–6.5 μm	bulk
Highly parallel microfocus diffraction, bright-field topography	Local strain analysis, dislocation imaging	0.5–30 μm (diffraction), 0.65 μm (topography)
Near-ambient-pressure HAXPES	Chemical state	30 μm

multiple-shot CDI, a method for reconstructing a movie of structural dynamics from a set of time-evolving coherent diffraction patterns (Fig. 1 right) [2].

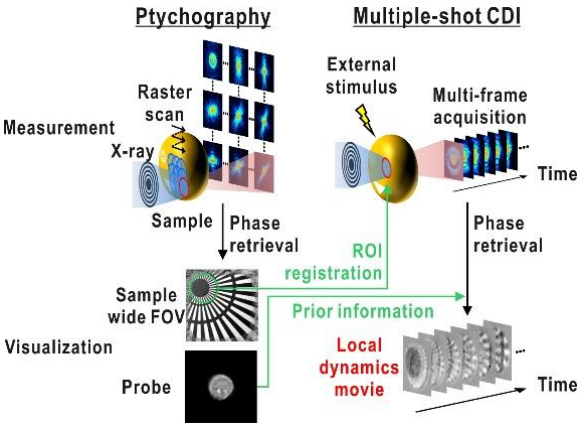

Fig. 1. Ptychography and multiple-shot CDI.

In multiple-shot CDI, there are two major developments: (1) multiple-frame PR algorithm dedicated to dynamic imaging, where the spatiotemporal smoothness of structural dynamics is utilized as a real-space constraint instead of the structural consistency or shape constraints used in conventional PR, and (2) a coherent projection illumination optics to produce top-hat local illumination, which enhances the convergence of the PR. Figure 2 shows multiple-shot CDI results of the Brownian motion of colloidal gold particles (numerical simulations) and a moving tantalum nanostructure (an actual experiment) obtained at BL24XU using 8 keV X-rays. Those results demonstrated the capability of dynamic imaging at frame rates of 10–100 frames per second. The numerical simulation shows that multiple-shot CDI is applicable even to fast dynamics with a root-mean-square displacement of ~3 pixels/frame, where speckles in the diffraction patterns are blurred during exposure. We believe that the combined use of multiple-shot CDI and ptychography will provide deeper insight into the structure–function relationship of materials and devices.

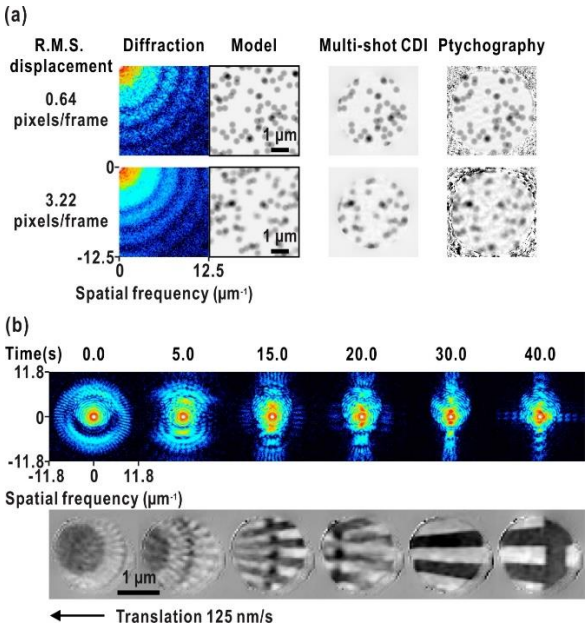

Fig. 2. Multiple-shot CDI of the Brownian motion at 100 fps ((a) simulation) and the moving nanostructure at 10 fps ((b) experiment). Representative frames in the reconstructed movies are shown. The pixel size of the reconstructed movies is ~40 nm/pixel. These movies are available as supplemental files of the original paper [2].

3. HAXPES measurements of highly insulating samples using graphene

One major disadvantage of photoemission spectroscopy is that it is often difficult to properly obtain a spectrum from a bulk insulating material because of the charging effect. Here, HAXPES spectra were successfully obtained from bulk insulating materials coated with monolayer graphene, which is a one-atom-thick material comprising a conductive carbon honeycomb

lattice [3].

In this study, highly insulating materials such as a glass slide (thickness: 1 mm), a $LiNbO_3$ (0001) wafer (0.5 mm), and a 1-mm-thick two-part (epoxy resin and polyamide amine) epoxy adhesive were used as samples. A typical sample structure is shown in Fig. 3. Al foil is inserted between the graphene film and the washer as a buffer, and each sample is approximately 1 cm^2 in size.

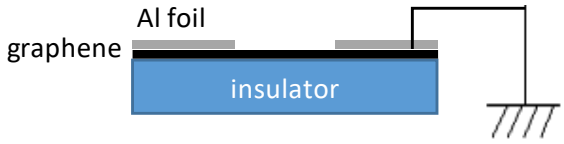

Fig. 3. Schematic of the sample structure.

Figure 4 shows the C 1s HAXPES obtained from an epoxy adhesive sample with graphene at hν=8 keV. Notably, we could measure practically no spectral intensity without graphene; however, the charging effect was counteracted by the graphene layer. The spectrum was curve-fitted using three components at approximately 284.8 (red), 286.0 (blue), and 287.7 eV (green), as shown in the figure. These can be attributed to C-C, C-O, and C=O components, respectively. Charging-free spectra could also be obtained from a glass slide and a $LiNbO_3$ wafer. This method can also be widely applied to various other electron spectroscopic techniques and to the analyses of other insulating materials.

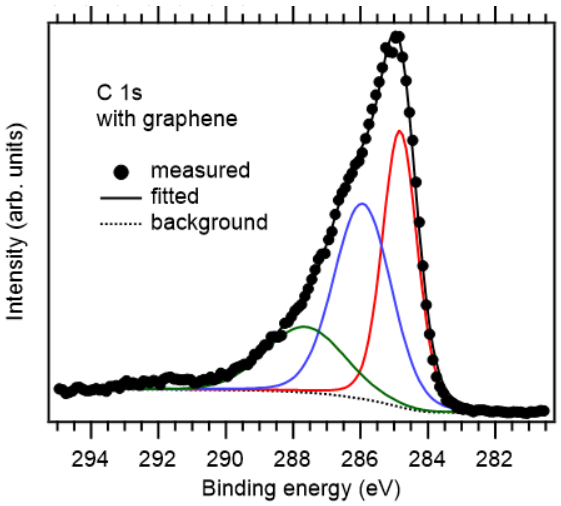

Fig. 4. C 1s HAXPES of epoxy adhesive.

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