BL05XU
R&D-ID

1. Introduction

In BL05XU, small-angle scattering (SAXS) and wide-angle diffraction (WAXD) measurements are performed at the experimental hutch. In optical hutches, we have installed and tested new optics [1] specifically for the utilization of high-energy X-rays and performed some pilot experiments.

2. Recent activities

We installed a double multilayer monochromator (DMM) to provide a high-flux high-energy X-ray beam in optical hutch 1 (OH1) at BL05XU in FY2020. DMM consists of two multilayer mirrors with 150 Cr/C layers and a d-spacing of 3.33 nm. We obtained the so-called pink beam with a mean photon energy of 100 keV and an energy bandwidth of 0.93% through DMM from the 19th harmonics of undulator radiation with the 1st harmonics of 5.26 keV. The total flux of the pink beam reached 1.3×10^{13} photons/s. We prepared an atmospheric section with a length of about 4 m called a high-energy test bench section upstream of a double-crystal monochromator (DCM) in optical hutch 2 (OH2) for the utilization of the pink beam (Fig. 1).

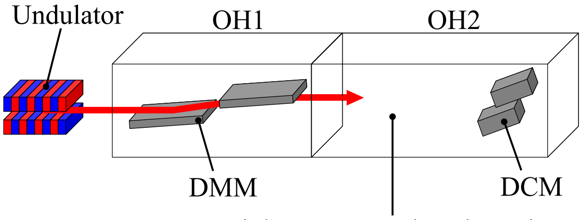

Fig. 1. Schematic of OH1 and OH2 at BL05XU.

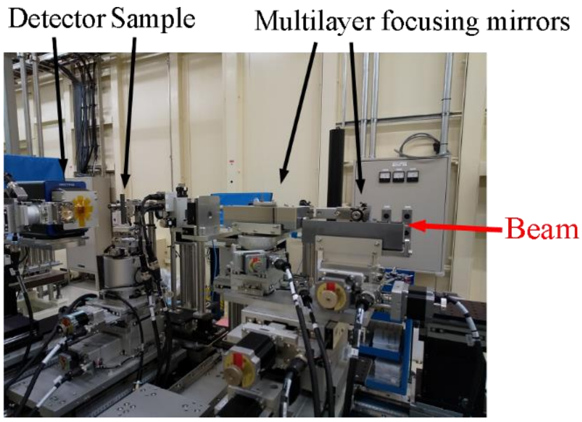

Fig. 2. Photograph of an experimental setup at the high-energy test bench.

In FY2021, we fabricated new multilayer focusing mirrors for the microfocusing of the pink beam. Graded W/C multilayers with d-spacings from ~3.3 nm to ~4.2 nm were coated on elliptical Si mirrors with a length of 200 mm and glancing angles from ~1.6 mrad to ~2.0 mrad of a Kirkpatrick–Baez (KB) mirror arrangement. Designed magnification fac tors of the multilayer focusing mirrors are 1/91.2 and 1/135.3 for the vertical (V) and horizontal (H) directions, respectively. The multilayer focusing mirrors were located at a working distance of 360 mm in the high-energy test bench section (Fig. 2). We irradiated the pink beam to the multilayer focusing mirror and gained a pink microbeam with a beam size of 0.3 × 1.0 µm (V × H) for the full width at half maximum and flux of 1×10^{11} photons/s. The total reflectivity of the multilayer focusing mirrors was 74%.

As a pilot application using the pink microbeam, we have performed X-ray diffraction (XRD) experiments toward nondestructive orientation microscopy (OM). OM based on electron backscatter diffraction is widely used to characterize the mechanical behaviors of polycrystalline materials, such as plastic deformation, creep, fatigue, degradation, and damage. Nondestructive OM has been demonstrated using X-ray diffraction and imaging techniques such as diffraction contrast tomography (DCT), 3D XRD microscopy (3DXRD), scanning 3DXRD, and differential aperture X-ray microscopy. Imaging-based DCT and 3DXRD using as wide an incident beam as the sample size can yield 3D orientation maps with a relatively short measurement time. However, DCT and 3DXRD are applicable only to thin, small, or coarse-grained samples owing to a limitation on the number of grains illuminated by the wide beam. On the other hand, scanning 3DXRD using an X-ray microbeam smaller than the grain size is advantageous for sample and grain sizes at the cost of measurement time [2].

In FY2020, we conducted scanning 3DXRD experiments at the high-energy test bench and successfully demonstrated nondestructive XRD OM for a solder joint of an electronic component on a printed circuit board (PCB) extracted from a computer peripheral without any sample cutting. The degradation of solder joints for electronic components on PCB caused by the difference in the linear coefficient of thermal expansion between the solder and the PCB has been a serious problem in electronics industries for a long time. It has been known from EBSD OM studies that intergranular and intragranular misorientations and grain refinement grow owing to the thermal-mechanical fatigue of the solder. Unfortunately, cross sectioning necessary for EBSD OM has prevented us from revealing the detailed mechanisms of the thermal-mechanical fatigue of solder joints, because we cannot trace the fatigue process with such destructive microscopy. We achieved nondestructive XRD OM for a coarse-grained solder joint with a grain size of more than 100 μm, which is, however not subjected to thermal-mechanical fatigue. As a next step, we need higher-resolution XRD OM to observe grain refinement caused by thermal-mechanical fatigue.

In FY2021, we conducted scanning 3DXRD experiments with the pink microbeam and small solder balls as a test sample. Solder balls with a diameter of ~30 μm were encapsulated in a glass capillary of 500 μm diameter. The capillary was mounted on a sample rotation stage and irradiated with the pink microbeam. Diffracted beams from individual balls or grains were detected as diffraction spots on an area detector. Diffraction spots from grains were collected during multiple rotations of 180° and translations of the sample. Detected diffraction spots were assigned to each grain using multi-grain indexing. After the indexing, the orientation of each grain and the number of detected diffraction spots, N, from each grain were determined. N is important because $N'=N/M$ corresponds to the confidence index of EBSD OM, where M is the theoretically expected number of diffraction spots from each grain. Figure 3 shows the maps of orientation and the normalized number of detected diffraction spots, N', which were measured for ~1 hour. It is found that the ~30-μm-diameter balls are reconstructed, and some balls consist of multiple grains. N' of ~0.8 at most was obtained for grains larger than 10 μm, which implies that grains with a size of 10 μm are observable with confidence. It is expected from these results that the grain refinement of the solder caused by thermal-mechanical fatigue can be nondestructively observed using the pink microbeam.

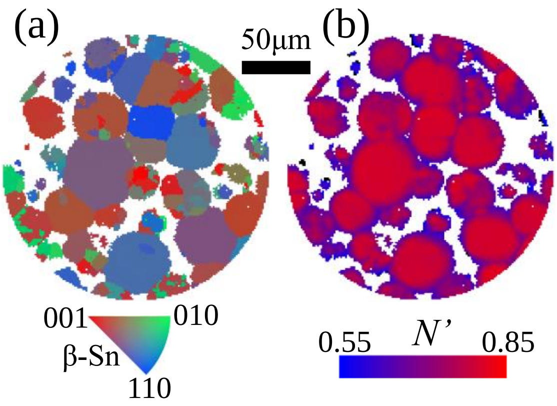

Fig. 3. (a) Orientation map of solder balls obtained by nondestructive XRD OM and (b) map of N'. The pixel size is 1.2 × 1.2 μm. The orientation is represented by the inverse pole figure.

Hoshino Taiki[*1] and Hayashi Yujiro[*2]

[*1] SR Imaging Instrumentation Team, Physical and Chemical Research Infrastructure Group, Advanced Photon Technology Division, RIKEN SPring-8 Center

[*2] SR Materials Science Instrumentation Team, Physical and Chemical Research Infrastructure Group, Advanced Photon Technology Division, RIKEN SPring-8 Center

References:

[1] Yumoto, H. et al. (2020). *Proc. SPIE* **11492**, 114920I.

[2] Hayashi, Y. et al. (2020). *Science* **366**, 1492–1496.