BL12XU
NSRRC ID

BL12XU is one of the two contact beamlines operated by National Synchrotron Radiation Research Center (NSRRC, Taiwan). BL12XU has an undulator source and two branches of the mainline and a sideline (see Fig.1). The mainline has been fully operational since 2001 and used by many domestic / international scientists. Inelastic x-ray scattering (IXS) experiments are mainly performed in BL12XU, and also several other experiments, such as high resolution x-ray optics experiment and micro-imaging, are carried out. In the side line, hard x-ray photoemission spectroscopy (HAXPES) is intensively performed. The sideline is partly opened for users. Some adjustments and commissioning are still made by Max-Planck Institute, Dresden.

Instrumentation:

We made the following upgrading in 2016.

· 5 × 32-ch strip Si detector :

We are developing a Si strip detector, consisting of 5 sensors having 32 strips of 125 μm wide. The detector are developed for (i) high-resolution (~25 meV) non-resonant IXS experiments using five analyzers of diced spherical crystals and (ii) low-resolution (~1.4 eV) experiments using 15 analyzers of bent spherical crystals. For the former, the foci of x-rays on the detector are divided into several lines, allowing us to perform a high resolution experiment with 5 sensors simultaneously (see Fig. 2). In the latter, each sensor collects photons from three analyzers having small offsets in Bragg angles, and thus the reflections from 15 analyzers are detected at the same time. The detector is working well and many of the experiments are made using this detector.

· High-pressure, high-temperature furnace for supercritical water :

We are challenging to derive attosecond (10^{-18} s) time-resolution response function of supercritical water by IXS. For this goal, we need to collect IXS spectra over a wide range in energy and momentum transfer. We have made a furnace and a sample cell, on which IXS experiments are performed in a wide scattering angle, from 2 to 150 degrees (see Fig. 3). Using an aluminum cell, we achieved 210 C and 5 MPa this year. With further modifications on the sample cells, we will try to increase the

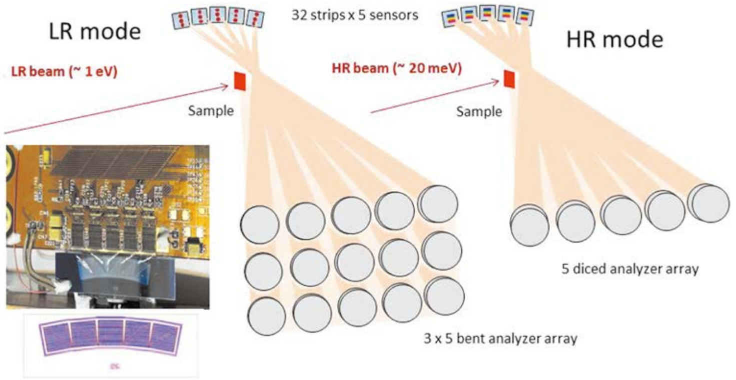

Fig.2 Schematic diagram of IXS experiments with 5 x 32 channel strip detector. Low resolution (LR, dE ~1 eV) beam irradiates a sample and scattered x-rays are collected by 15 bent analyzers in LR mode while high-resolution (HR, ~20 meV) beam and 5 diced analyzers (dE ~ 15 meV) are utilized in HR mode. Highest resolution we achieved so far is 25 meV (overall).(channel cut) monochromator, PRP a phase retarding plate, FM a focusing mirror, and IXS an inelastic x-ray scattering spectrometer.

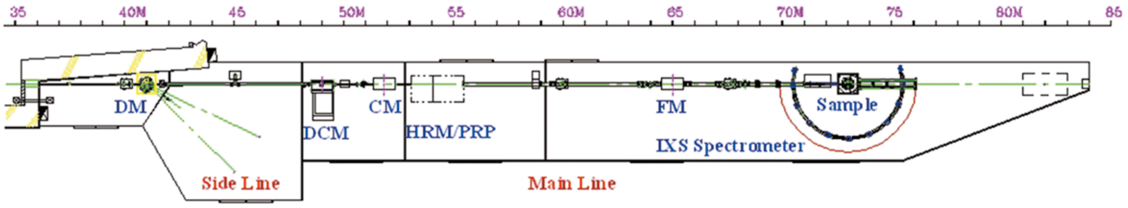

Fig.1 Schematic diagram (top view) of the BL12XU: DM is a diamond monochromator for the sideline, DCM a double crystal monochromator for the mainline, CM a collimating mirror, HRM a high resolution (channel cut) monochromator, PRP a phase retarding plate, FM a focusing mirror, and IXS an inelastic x-ray scattering spectrometer.

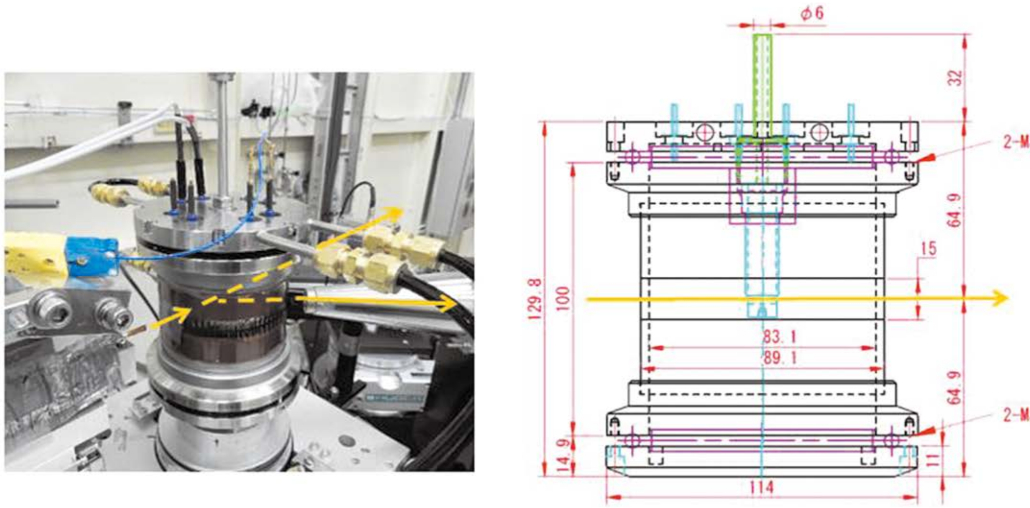

Fig.3 Photo and drawing of high-pressure, high-temperature (< 22 MPa, 370 C) furnace to produce supercritical water. Inner cell seals HP-HT waters. Wide opening is made to cover a wide momentum range.

temperature and the pressure up to 370 C and 22 MPa, at which supercritical water is available.

Experiments:

In 2016, we had 8 experiments of non-resonant IXS, 2 of x-ray emission, 13 of resonant IXS (or resonant emission), 2 of high-resolution x-ray optics, 3 of micro-imaging, and 7 of HAXPES. Interesting examples are introduced below.

· **Suppression of X-ray-induced dissociation of H_2O molecules in under pressure:**

Fukui et al. investigated molecular dissociation induced by 10 keV x-ray irradiation in dense ice at pressures up to 40 GPa at 300 K. The dissociation yield estimated from the oxygen K-edge x-ray Raman spectra, showed that the molecular dissociation was enhanced up to 14 GPa and gradually suppressed on further compression to 40 GPa (see Fig. 4). The molecular dissociation was detected for a rather narrow pressure span of 2–40 GPa [Fukui et al, Sci. Reports **6:**26641 (2016)]

· **1s3p Resonant Inelastic X-ray Scattering of Cobalt Oxides and Sulfides:**

In order to establish detailed reference materials for cobalt-based systems, Samarai et al. applied 1s x-ray absorption spectroscopy (XAS) and 1s3p RIXS to cobalt oxides (CoO, Co_3O_4) and sulfides (CoS and CoS_2). The Co^{3+} ions in Co_3O_4 contained a large nonlocal peak intensity in the pre-edge and that the resonant 3p final state of Co_3O_4 was dominated by

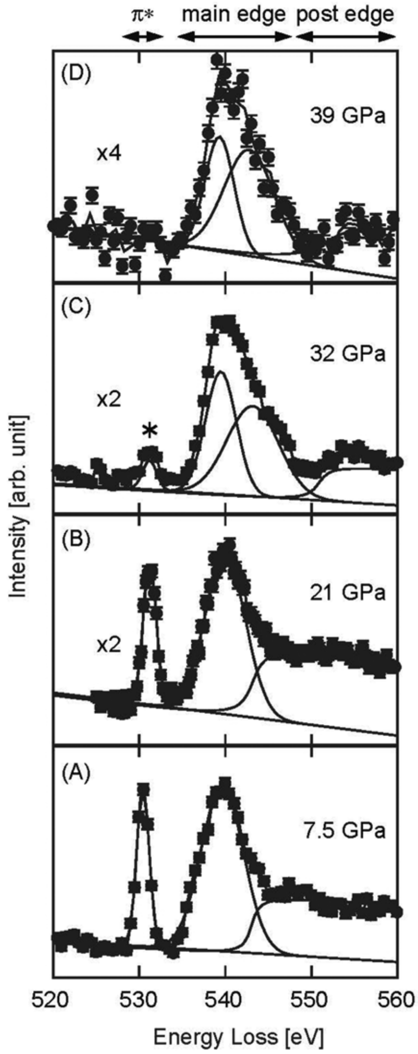

Fig.3 XS spectra on H_2O at high-pressures. As pressure increases, a peak at 530 eV substantially becomes weaker compared to the main peak, representing $2H_2O \rightarrow 2H_2 + O_2$ dissociation suppressed [Sci. Reports 6:26641 (2016)].

the two exchange-split peaks of the tetrahedral Co^{2+} site. In contrast, the 3p final state of CoS and CoS_2 showed a single asymmetric peak due to the large screening of the 3p-3d exchange interaction. [M.Al Samarai et al, J. Phys. Chem. 120, 24063 (2016)]

· **Transparent Conducting Oxide Induced by Liquid Electrolyte Gating :**

Optically transparent conducting materials are essential in modern technology. Recently, it has been shown that ionic liquid gating can induce a reversible, nonvolatile metallic phase in initially insulating films of WO_3. ViolBarbosa et al. have used hard x-ray photoelectron spectroscopy and spectroscopic ellipsometry to show that the metallic phase produced by the electrolyte gating does not result from a significant change in the bandgap but rather originates from new in-gap states. These states produce strong absorption below ~1 eV, outside the visible spectrum, consistent with the formation of a narrow electronic conduction band. [C. ViolBarbosa et al, Proc. Natl. Acad. Sci. 113, 11148 (2016)]

N. Hiraoka, H. Ishii, Y.-F. Liao, M. Yoshimura, T. Tatsumi,
K.-D. Tsuei
NSRRC, Taiwan