BL10XU
High Pressure Research

1. Introduction

The BL10XU beamline is dedicated to high-energy monochromatic X-ray diffraction (XRD) for high-pressure research using a diamond anvil cell (DAC). The beamline supports complex sample environments under high-pressure conditions, including a double-sided laser heating system and a 4K-GM cryostat. These techniques enable high-pressure XRD experiments to be conducted over a wide temperature range, from cryogenic temperatures below 10 K to those exceeding 3,000 K. In 2024, a precise external field control environment was developed to expand the range of accessible sample conditions. First, an externally heated DAC was installed to perform stable high-pressure and high-temperature experiments in the 300–1,000 K range. Second, a He-closed-cycle 4K-GM cryostat was equipped with dual diaphragms for pressurization. This allows precise pressure control during high-pressure and low-temperature experiments. The details are described below.

2. Generalization of high-temperature and high-pressure *in situ* X-ray diffraction experiments using an externally heated DAC at BL10XU

Previously, the beamline's standard equipment did not cover the intermediate temperature range of 300–1,500 K. Experiments in this range required users to bring custom-built devices from their own laboratories. Consequently, accessibility was limited to researchers with specialized technical expertise, and experiments remained challenging for novice users. In addition, manual temperature adjustment reduced efficiency and reproducibility, substantially increasing the operational workload, including XRD measurements, and raising the risk of human error.

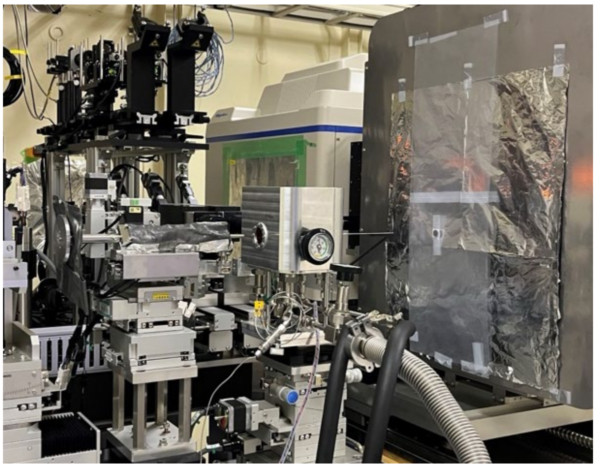

Fig. 1. Overview of the high-temperature and high-pressure XRD system using an externally heated DAC, developed at SPring-8 BL10XU for precise temperature and pressure control up to ~1000 K.

To address these issues, a newly designed externally heated diamond anvil cell (EHDAC) for *in situ* high *P*–*T* XRD was developed and implemented at BL10XU (Fig. 1). This system enables even novice users to readily generate temperatures up to 1,000 K and conduct XRD experiments with precise temperature and pressure control. Temperature is regulated by a controller specifically optimized for the EHDAC, while pressure is controlled via a diaphragm. Both functions can be operated remotely.

The performance of the new system was evaluated using titanium as a sample, for which detailed high-temperature and high-pressure phase diagrams have been reported [e.g., Ref. 1].

Temperatures up to 1,000 K were successfully applied, and changes in diffraction lines associated with the α-Ti to ω-Ti phase transition were observed during heating at constant pressure (see Fig. 2). The phase transition conditions obtained showed excellent agreement with previous studies (Fig. 3). The system demonstrated the capability to control temperature in 10 K increments at constant pressure and to adjust pressure in 0.2 GPa increments at constant temperature. The new system also provided remarkable stability, within ±1 K, under high *P*–*T* conditions.

This system allows even novice users to routinely perform high-precision determinations of phase equilibria and equations of state under high-temperature and high-pressure conditions, as well as *operando* measurements with precise temperature control. It is expected to have a significant impact across a wide range of research fields, including the elucidation of mechanisms underlying novel material synthesis and reaction kinetics.

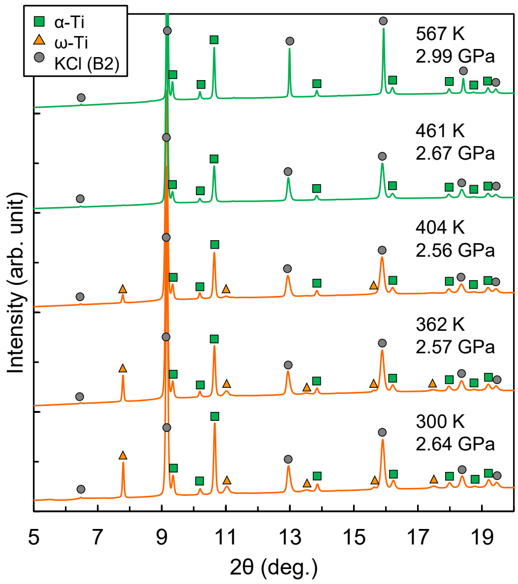

Fig. 2. Changes in diffraction lines associated with the α-Ti to ω-Ti phase transition.

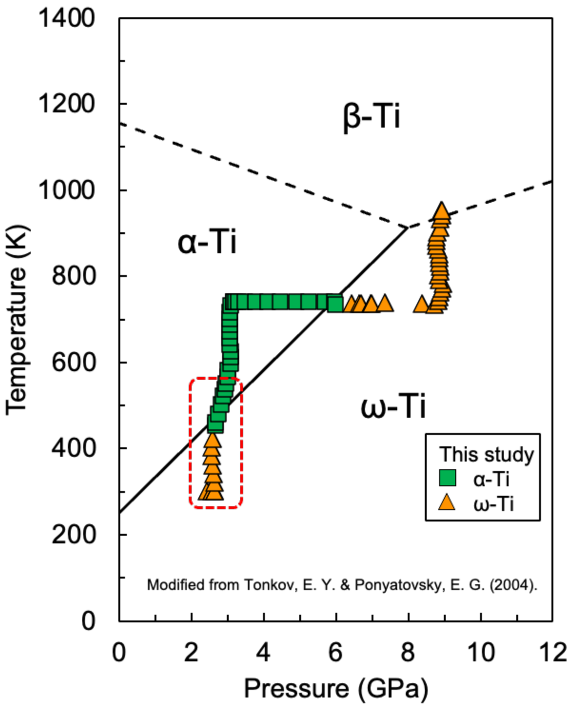

Fig. 3. Summary of the temperature–pressure conditions explored in this experiment. Green squares indicate conditions where α-Ti was observed, and orange triangles indicate conditions where ω-Ti was observed. The red dashed rectangle highlights the region in which changes in diffraction lines were detected, as shown in Fig. 2.

3. Cryostat with dual diaphragms for high-pressure and cryogenic XRD

There is a growing demand for high-pressure and low-temperature XRD experiments under precisely controlled pressure conditions, as well as for experiments in the sub-gigapascal (GPa) range. A He-closed-cycle 4K-GM cryostat has been installed for high-pressure XRD using a DAC (Fig. 4). This apparatus can cool samples in high-pressure cells to below 10 K. The new cryostat is designed to accommodate diaphragms on both sides of the DAC.

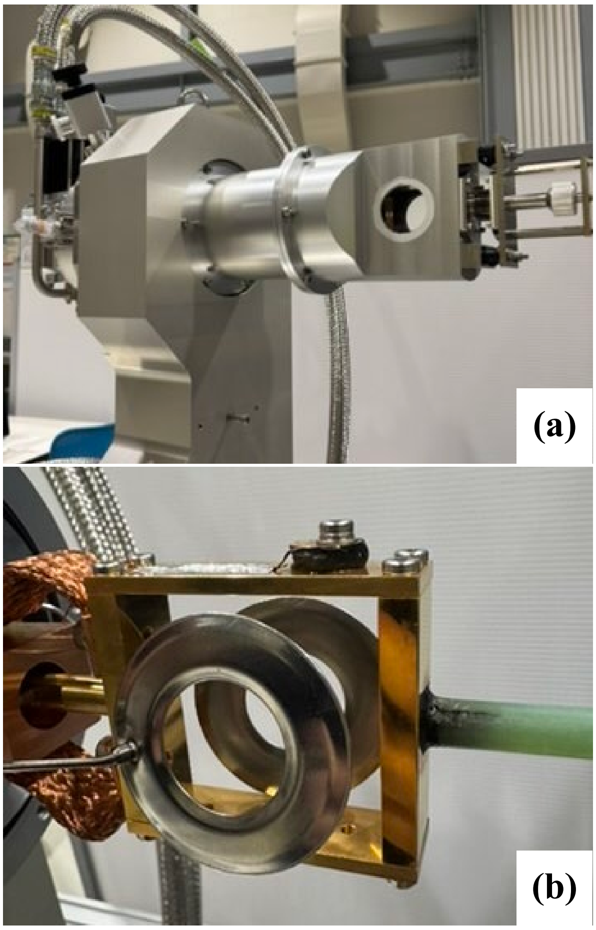

Fig. 4. (a) Photograph of the high-pressure and cryogenic XRD cryostat system installed in SPring-8 BL10XU. (b) Dual diaphragms attached to the cryostat, applicable to a diamond anvil cell.

The dual-diaphragm setup enables bidirectional pressure control of the sample inside the DAC, allowing not only pressurization but also depressurization and fine adjustment to the target pressure. In contrast, the previous cryostat could be equipped with only a single diaphragm on one side, and its function was limited to pressurization.

Controlling pressure is particularly challenging in high-pressure and low-temperature experiments. During the cooling process, the pressure inside a DAC often increases by one to several GPa. This difficulty arises because the high-pressure cell is composed of diamond and various metals with different thermal expansion coefficients. With the single-diaphragm setup, this pressure increase cannot be controlled. The dual-diaphragm configuration is expected to solve the problem of uncontrollable pressure increases during cooling. Moreover, it facilitates precise pressure adjustment to the desired value, enabling reliable high-pressure and low-temperature experiments.

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Reference:
[1] Tonkov, E. Y. & Ponyatovsky, E. G. (2004). *Phase Transformations of Elements under High Pressure*. CRC Press, Boca Raton.