

BL19B2

X-ray Diffraction and Scattering II

1. Introduction

BL19B2 is a bending magnet beamline for X-ray diffraction and scattering experiments. A versatile high-throughput powder diffractometer (Polaris) is installed in the first hutch (EH1), while a multi-axis diffractometer (Huber) is located in the second hutch (EH2). In the third hutch (EH3), a PILATUS 2M two-dimensional detector is installed for small-angle X-ray scattering (SAXS) experiments. EH3 offers camera lengths between 0.7–3 m, while EH2 provides a 41 m length for ultra-small-angle X-ray scattering (USAXS)^[1]. During fiscal year 2024, beamline optics were upgraded to support high-energy X-ray applications. Concurrently, a feasibility study was conducted in EH2 to analyze the surface oxide film of a Mg-X binary alloy. This *in-situ* grazing-incidence X-ray diffraction experiment was performed using the multi-axis diffractometer in combination with an Anton Paar DHS1100 heating system.

2. Upgrades of beamline optics

Recently, many users have required higher-energy X-rays because of their advantages, such as accessibility to a wide reciprocal space and the possibility of using transmission diffraction geometry. To enable the use of X-rays up to 72 keV, the beamline's optical components were upgraded. This upgrade included a modification of the monochromator, where both Si(111) and Si(311) crystals were installed. These crystals can be exchanged in accordance with the desired X-ray energy: Si(311) is used for X-rays above 37 keV, while Si(111) is used for lower energies.

3. Evolution of the surface oxide film on a Mg alloy: An *in-situ* grazing-incidence X-ray diffraction study

The performance and long-term durability of magnesium (Mg) alloys in high-temperature environments are contingent upon the formation of a stable surface oxide film. This passivating layer is the primary barrier against aggressive oxidation and corrosion, making its characterization paramount for applications in the aerospace, automotive, and railway industries. However, the dynamic evolution of this film's structure and composition under thermal stress remains an area of active investigation.

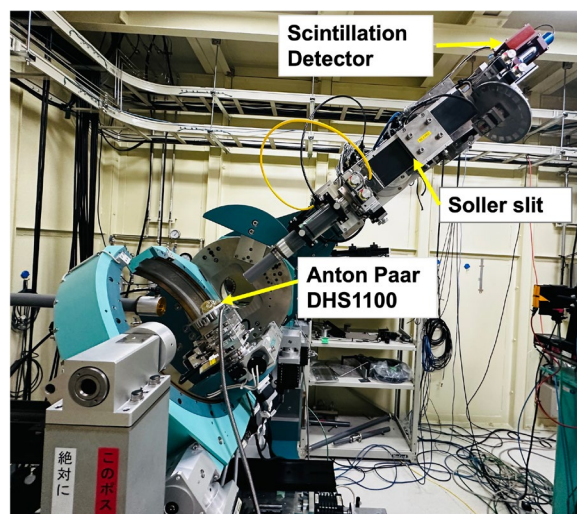


Fig. 1. *In-situ* XRD setup on the versatile multi-axis diffractometer installed at the BL19B2 EH2.

To address this, in the present feasibility study, *in-situ* grazing-incidence X-ray diffraction (GIXRD) is employed to scrutinize the nascent oxide layers forming on a Mg-1Zn-2Y alloy at elevated temperatures. This surface-sensitive technique enables the real-time monitoring of the

alloy–environment interface, offering unparalleled insight into the oxidation mechanism. The primary

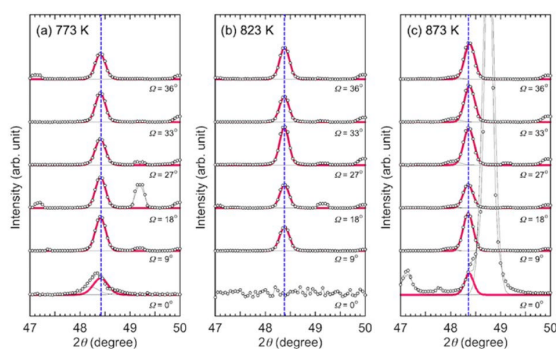


Fig. 2. *In-situ* XRD 2θ – $\sin^2\Omega$ patterns of Mg–1Zn–2Y alloy at (a) 773 K, (b) 823 K and (c) 873 K.

objectives are to (1) delineate the kinetics of oxide growth, (2) identify the emergent crystalline phases within the film, and (3) evaluate the effect of alloying constituents on oxidation resistance. Ultimately, this research provides fundamental data on the surface chemistry of ternary Mg alloys, paving the way for the rational design of materials with enhanced environmental stability.

To evaluate the residual stress within the oxide films that develop on the specimen surfaces at elevated temperatures, we performed GIXRD measurements of the Y_2O_3 film of Mg–1Zn–2Y and Mg–1Zn–2Y–0.7Cu–0.1Be alloys. As shown in Fig. 1, these were conducted using the six-circle diffractometer at beamline BL19B2 of the SPring-8 facility in Japan. We maintained an X-ray beam with an energy of 8.04199 keV using a Si (111) double-crystal monochromator in conjunction with a Rh-coated focusing mirror. For the stress analysis, we employed the 2θ – $\sin^2\Omega$ method, taking measurements at six tilt angles: $\Omega = 0, 9, 18, 27, 33,$ and 36° . Diffracted X-rays were detected through a Soller slit by the FMB $LaBr_3$ scintillation detector with a scan angle (2θ) ranging from 10 to 50°

degrees in 0.05° increments. During the experiment, specimens were heated to 773, 823, and 873 K on an Anton Paar DHS-1100 heating stage [2].

Figure 2 shows the 2θ – $\sin^2\Omega$ narrow XRD patterns of the Y_2O_3 film on Mg–1Zn–2Y alloys at 773, 823, and 873 K for Ω values of 0, 9, 18, 27, 33, and 36° . At 773 K, the peak of the Y_2O_3 film on Mg–1Zn–2Y alloys shifted slightly to a lower angle with increasing Ω (Fig. 2(a)). In contrast, at 823 K, the peak of the Y_2O_3 film showed no change with increasing Ω . Furthermore, at 873 K, the peaks of the Y_2O_3 film shifted slightly to higher 2θ diffraction angles as Ω increased (Fig. 7(c)).

In-situ GIXRD high-temperature measurements showed that Be and BeO accumulate at the grain boundaries of the Y_2O_3 film. This segregation creates high compressive stress and blocks pathways for oxygen diffusion, significantly improving the protective quality of the Y_2O_3 film.

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References:

- [1] <https://user.spring8.or.jp/?p=42152>
- [2] Inoue, S. et al. (2025). *Mater. Today Commun.* **47**, 113098.