BL08B2
Hyogo BM

1. Introduction

BL08B2 is the Hyogo-prefectural beamline. It is a hard X-ray contract beamline designed for industrial applications. XAFS, X-ray topography, Imaging/CT, XRD and SAXS measurements, which are widely used in industry, can be performed. Over the past few years, research to develop new materials to apply to informatics technologies (materials informatics) has been actively conducted. Since FY2018, the beamline has been supporting research and development by manufacturing through a coalition between synchrotron radiation measurements and informatics technologies. For materials informatics with synchrotron radiation, it is important to prepare large datasets for machine learning. Operando XAFS/XRD and high-throughput SAXS/WAXS measurements have been conducted via automatic data acquisition. In addition, a two-dimensional conversion electron yield XAFS (2D-CEY-XAFS) was installed. This report describes details of the 2D-CEY-XAFS measurement system and examples of its applications to steel corrosion investigations.

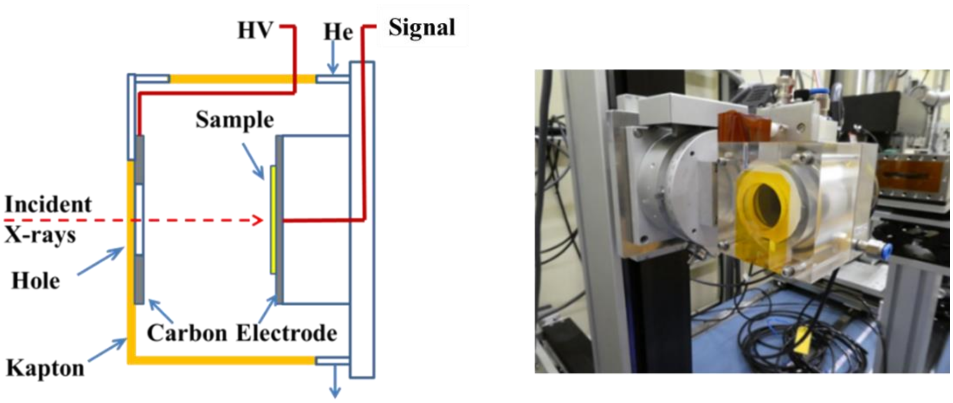

Fig. 1. Schematic diagram of the 2D-CEY detector and side view of the experimental setup at BL08B2.

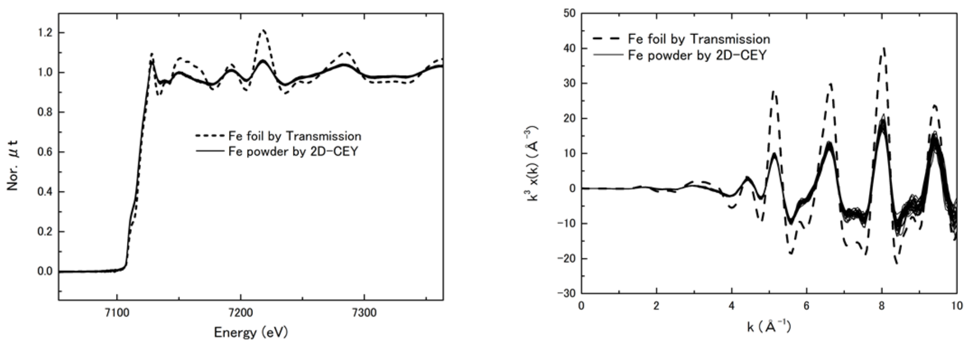

Fig. 2. Comparison of the normalized Fe K-edge XANES spectra and EXAFS spectra.

2. Development of 2D-CEY-XAFS system

CEY XAFS is used to analyze the chemical structure of material surfaces [1]. A conventional CEY detector is not suited for 2D XAFS measurements because it is used in a grazing incidence configuration. Therefore, a new CEY detector was designed for 2D-CEY-XAFS measurements. Figure 1 shows a schematic diagram of the 2D-CEY detector and the experimental setup at BL08B2. The 2D-CEY detector has a φ30-mm window in the center of the detector to the incident X-ray. The sample surface area that can be measured in 2D scanning is 10 mm square. Figure 2 shows the results of the measurements of Fe foils and powders as samples.

3. XAFS analysis of corrosion on a steel surface using machine learning

In general, the corrosion behavior of steel materials varies in the forming process and structure of iron rust layers due to differences in environment and microstructure factors. Machine learning has quantified the atomic valence and bond change of the oxidation state on the surface of steel materials, which are difficult to measure using conventional analysis.

The oxidation state of the corroded steel test piece and the Fe–O bond length were evaluated. The specimen surface before and after corrosion was measured by the 2D-CEY-XAFS method. The region of the specimen surface measured by the 2D-CEY-XAFS method was 10 mm × 10 mm. The X-ray beam size was 0.5 mm × 0.5 mm. The spectra obtained in these measurements were normalized and Fourier transformed using the Larch Library. Nonnegative matrix factorization with the soft orthogonality constraint (NMF-SO) method developed by *Shiga et al* [2]. was used to separate the component spectra in Fe oxides.

Two-component spectra, Component-1 and Component-2, were separated by the NMF-SO method and were similar to γ-FeOOH and Fe, respectively. This result supports the report that rusts mainly composed of γ-FeOOH are formed at the early stage of atmospheric corrosion [3]. By reconstructing the proportion of Component-1, the progress of corrosion in the specimen surface was visualized (Fig. 3). The proportion of Component-1 of XANES and Radial distribution function (RDF) map is close to 1, indicating that the oxidation of Fe and the formation of Fe–O limits the progress.

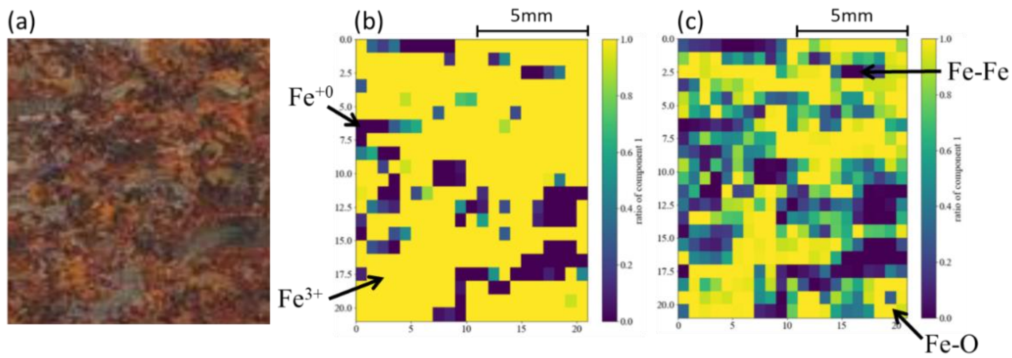

Fig. 3. (a) Photograph of rust on the surface using (b) XANES and (c) RDF score map of steel surface after a 3-hour corrosion test.

The XANES map clearly distinguished the oxidation state on the steel surface. The RDF map also showed that the rust structure differed in the same oxidized region of the XANES map. These results suggest that the combination of synchrotron radiation analysis and machine learning can effectively elucidate rust formation mechanisms in initial corrosion.

This study was conducted by Dr. Takahiro Ozawa of Kobe Steel.

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[3] T. Misawa et al. (1993). *Tetsu-to-Hagané*, **79**, 69-75.