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In-situ 濃度観測点増加に伴う液体金属の拡散係数測定法の解析精度

Analysis Precision of Diffusion Measurements in Liquid Metals through Increasing In-situ Concentration Measurement Point

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1. Introduction

Diffusion coefficients in liquid metals are important for understanding the solidification process in metallic materials and the atomic transport in liquid metals. In-situ X-ray fluorescence analysis (in-situ XRF) has been proposed as the diffusion measurements in liquid metals by measuring the concentration at only one point in a capillary1). In this study, another X-ray device was installed additionally to double the number of the measurement points. In operating several X-ray devices without a collimator, the precision of the concentration measurement would be declared by detecting an intensity from not intended point, such as another measurement point. The objective of this study is to determine the effect of a collimator on an obtained X-ray spectrum and the precision of the concentration measurement during operating two X-ray devices in in-situ XRF.

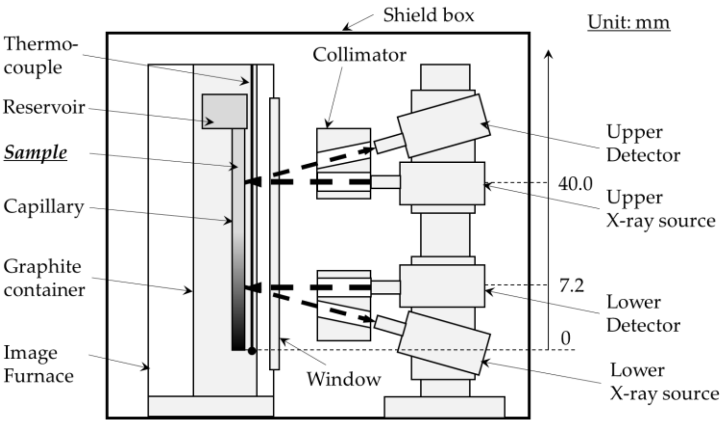

Figure 1. Experimental apparatus of in-situ XRF.

2. Experimental Procedure

Figure 1 shows the experimental apparatus of in-situ XRF for diffusion experiments in liquid metals. Two sets of the X-ray sources (Mini-X2, Amptek Inc.) and the detectors (X-123SDD, Amptek Inc.) were contained in a shield box. The collimator was made of A2017.

A pure Sn rod was cast into 1.5×1.5×10 mm^3 and contained in the capillary, the size of which was 1.5×1.5×63 mm^3. The capillary was heated to 573 K and then its temperature was maintained in vacuum. The upper and lower X-ray sources were positioned at 40.0 mm and 7.2 mm above the bottom of the capillary, respectively. X-ray fluorescence was detected from each detection point with an integration time of 600 s by using the upper detector, while operating only the upper X-ray source or both X-ray sources. By confirming the effect of the collimator, the above measurements were performed with and without the collimator.

3. Results

Figure 2 shows the obtained X-ray spectra from the pure Sn rod. Without collimator, the intensity of Kα emission line of Sn (Sn Kα) detected by operating both X-ray sources was larger than that detected by operating only the upper X-ray source. By adopting the collimator, the detected Sn Kα intensity did not change significantly when both X-ray sources were operated or only the upper X-ray source was operated.

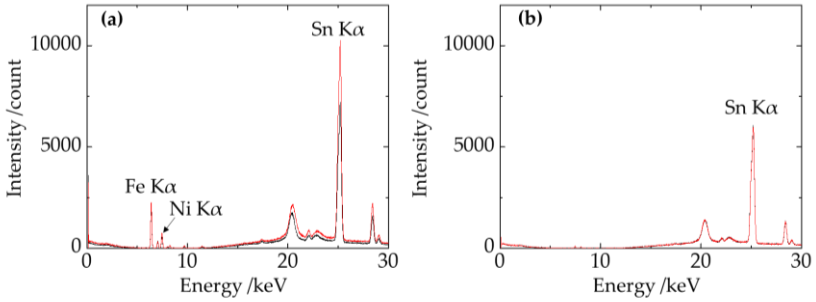

Figure 2. Obtained X-ray spectra from the pure Sn rod; **(a)** detected with no collimator, and **(b)** detected with the collimator. Black line: obtained spectrum by operating only the upper X-ray source. Red line: obtained spectrum by operating both X-ray sources.

4. Discussion

According to **Fig. 2(a)**, the intensities of Kα emission lines of Fe and Ni (Fe Kα and Ni Kα) were also detected. This indicated that an X-ray intensity was detected from not only another measurement point, but also the body of the furnace (made of Ni) and frame of the window (made of Fe) with no collimator. The precision of the concentration measurement in in-situ XRF is declared due to these intensities, which is considered as the background in the spectrum. According to **Fig. 2(b)**, an X-ray intensity from these materials can be eliminated from the detected spectrum by adopting the collimator. Therefore, the precision of the concentration measurement is maintained even though operating two X-ray sources in in-situ XRF.

5. Conclusion

By adopting the collimator, an intensity emitted from not intended point can be eliminated from the obtained X-ray spectrum. Therefore, the precision of the concentration measurement can be maintained when operating two X-ray sources, as that when operating only one X-ray source.

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References

1) Y. Kobayashi, M. Shiinoki, R. Yamatake, T. Masaki and S. Suzuki: Int. J. Microgravity Sci. Appl., **38** (2021) 380302, DOI: 10.15011/jasma.38.380302.