

Luminescence-Based Imaging Analysis of Metals

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Steel products can suffer from various defects caused by non-metallic inclusions, including hydrogen cracking, fatigue damage, reduced ductility, and brittleness at low temperatures. During manufacturing processes, these inclusions may lead to surface defects, wire breaks in drawing operations, and blockage of nozzles in continuous casting systems [1]. Therefore, identifying and characterizing non-metallic inclusions is essential for quality control in steel production. Traditional methods for studying these inclusions involve examining polished steel surfaces through techniques such as optical microscopy and scanning electron microscopy (SEM), which is a time-consuming process. The author focused on cathodoluminescence (CL) imaging as a rapid determination of non-metallic inclusions in steel because CL imaging can be obtained by capturing luminance from the inclusions as a result of electron bombardment for a short time (~ 1 s). In this study, the feasibility of CL imaging for identifying $\text{MgO}\cdot\text{Al}_2\text{O}_3$ and Al_2O_3 inclusions was investigated. These inclusions are formed through reactions between $\text{MgO}\cdot\text{C}$ refractories and molten steel during aluminum deoxidation. They act as critical points where wire fracture can initiate in subsequent drawing operations, negatively impacting the final steel product quality. The author also applied ionoluminescence (IL) imaging, which produces images based on light emission from materials under ion bombardment, to the three-dimensional imaging of $\text{MgO}\cdot\text{Al}_2\text{O}_3$ inclusions.

A sample containing $\text{MgO}\cdot\text{Al}_2\text{O}_3$ and Al_2O_3 inclusions was prepared by mixing metal powders composed of Fe (68 wt%), Cr (20 wt%), Ni (10 wt%), Mn (1 wt%), and Al (1 wt%). The mixed power was then placed in a MgO crucible, and melted at 1560 °C for 30 minutes under an argon atmosphere. The sample surface was polished to a mirror finish using emery paper and subsequently polished using 1 μm diamond slurry. CL images were obtained through a quartz viewing port attached to the SEM system using a digital mirrorless camera equipped with a zoom lens (Fig. 1(a)). The camera was sensitive to wavelengths in the range of 420–680 nm. For IL imaging, ion guns generated by an ion gun were irradiated onto the sample at 5 kV and 10 mA under an argon atmosphere at 5×10^{-4} Pa. IL images were acquired through a quartz viewing port mounted on the measurement chamber using the same type of camera and lens used for CL imaging (Fig. 1(b)). In addition, surface morphology and compositional analysis were performed using a SEM equipped with energy-dispersive X-ray spectroscopy (EDX).

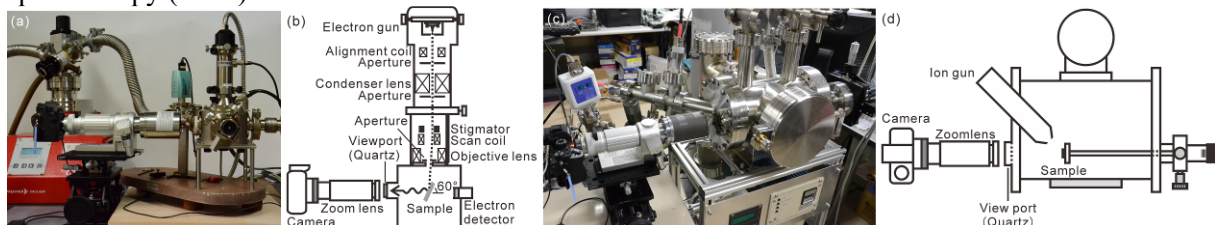


Fig. 1 Photographs and schematic views of the custom (a,b) CL [2] and (c,d) IL systems [3].

The sample contained $\text{MgO}\cdot\text{Al}_2\text{O}_3$ (area 1) and Al_2O_3 (area 2) inclusions as shown in Fig. 2(a), which was confirmed by EDX point analysis. The $\text{MgO}\cdot\text{Al}_2\text{O}_3$ and Al_2O_3 inclusions exhibited green and red luminescence, respectively (Fig. 2(b)). Luminescent colors and spectra of these inclusions consistent with those reported previously [4]. The CL image was obtained with a exposure time of 0.2 s, demonstrating that CL imaging enables rapid identification of $\text{MgO}\cdot\text{Al}_2\text{O}_3$ and Al_2O_3 inclusions in steels.

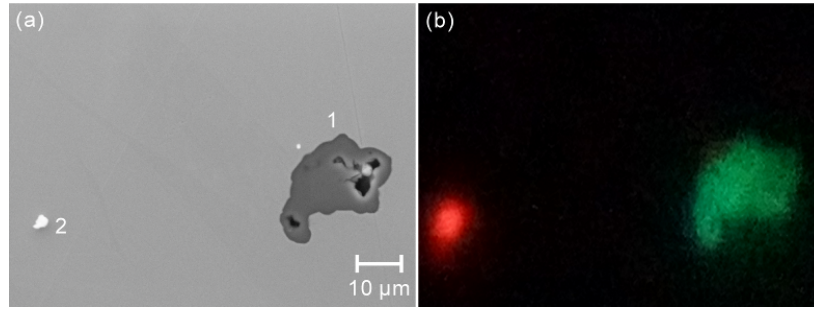


Fig. 2 (a) SEM and (b) CL images of the sample. The exposure time for the CL image was 0.2 s.

Three-dimensional imaging of the $\text{MgO} \cdot \text{Al}_2\text{O}_3$ inclusions was conducted by capturing two-dimensional IL images every 10 min during argon-ion bombardment. The analyzed $\text{MgO} \cdot \text{Al}_2\text{O}_3$ inclusions (Fig. 3(a)), which were confirmed by EDX point analysis, exhibited green luminescence consistent with that observed in the CL image (Fig. 2(b)). As the ion beam irradiation progressed, the green luminescent region gradually changed (Figs. 3(b-d)), and the luminescence disappeared after 120 min of irradiation. By compiling these two-dimensional IL images, a three-dimensional image of the $\text{MgO} \cdot \text{Al}_2\text{O}_3$ inclusions was constructed as shown in Fig. 3(e). The shapes of the $\text{MgO} \cdot \text{Al}_2\text{O}_3$ inclusions were in good agreement with previously reported shapes [5]. Therefore, IL imaging can provide a three-dimensional information on $\text{MgO} \cdot \text{Al}_2\text{O}_3$ inclusions more rapidly than conventional three-dimensional imaging methods, such as serial sectioning and X-ray computed tomography.

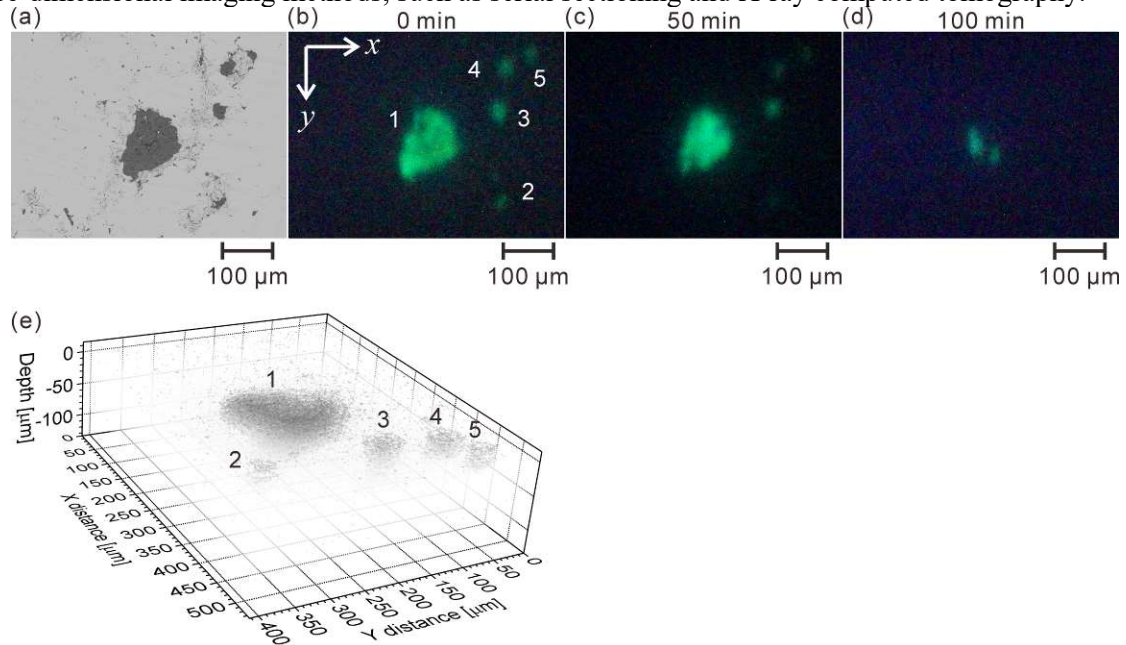


Fig. 3 (a) SEM and (b) the corresponding IL images obtained after argon-ion bombardment for (b) 0, (c) 50, and (d) 100 min in an area of the model sample. (e) Three-dimensional image of $\text{MgO} \cdot \text{Al}_2\text{O}_3$ inclusions in the area constructed based on their two-dimensional IL images [3].

Acknowledgements

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References

- [1] L. Zhang and B.G. Thomas, *ISIJ Int.* 43 (2003) 271-291.
- [2] S. Imashuku, K. Wagatsuma, *ISIJ Int.*, 62 (2022) 891-896.
- [3] S. Imashuku, *Metall. Mater. Trans. B*, 55 (2024) 2459-2466.
- [4] C.M. MacRae and N.C. Wilson, *Microsc. Microanal.* 14 (2008) 184-204.
- [5] G. Okuyama, K. Yamaguchi, S. Takeuchi and K.-i. Sorimachi, *ISIJ Int.* 40 (2000) 121-128.