

Focused Electron Beam and Elemental Mapping of Palm-top EPMA (Scanning) Equipped with CL Spectrometer (Projection)

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We realized an electron probe microanalyzer (EPMA) and X-ray tube with a palm-top size chamber including the electron source and the sample stage using pyroelectric crystal as the electron source [1,2]. Electrons in the palm-top EPMA and X-ray tube were bombarded on the sample stage and the stainless steel chamber because a rectangular LiTaO_3 crystal was used. Thus, it is difficult to measure Cr, Fe and Ni with the palm-top EPMA. In the present study, we tried to focus the electron beam of the palm-top EPMA on samples by standing a metal wire on the pyroelectric crystal in order to prevent the production of an electric field at the wall of the chamber. We also performed elemental mapping by cathode luminescence (CL) using the pyroelectric crystal and a small camera.

A schematic view of the palm-top EPMA is shown in Fig. 1 (a). +z plane of a single crystal of LiTaO_3 with $3 \text{ mm} \times 3 \text{ mm}$ in x-y plane and 5 mm in z-axis was attached on a Peltier device with silver paste. A gold wire was tightened into a hole in the silver stage with a screw. The silver stage with the gold wire was attached to the LiTaO_3 crystal with silver paste. As for CL spectrometer, a small camera was introduced into the sample chamber with T shape ISO quick release coupling and detachable vacuum joints. Measurements were carried out during cooling the LiTaO_3 crystal by applying -3 volt DC to the Peltier device. Pressure of the sample chamber was set to 2 Pa during the measurements.

Figure 1 (b) shows EDX spectra of titanium plate with and without the gold wire on the LiTaO_3 crystal. Although the gold wire was attached to the LiTaO_3 crystal, Cr, Fe, and Ni K lines from the stainless steel were detected in addition to Ca and Ti K lines. This is because an electric field was produced between the surface of the silver stage and area including the sample stage and the wall of the stainless steel ISO quick release coupling as shown in Fig. 2 (a). On the other hand, Ca and Ti K lines were only detected when we covered the silver stage holding the gold wire with high-vacuum grease. This result indicates that an electric field was produced between the tip of the gold wire and the sample stage and that electrons were focused on the sample as shown in Fig. 2 (b). The spot size of the electron beam was calculated to be $100 \mu\text{m}$ by the distribution of the brightness of the illuminated region in Fig. 2 (b) (Fig. 2 (c)).

We then carried out elemental mapping by applying CL phenomena of rare earth element. Fig. 3 shows photo of TbF_3 and GdF_3 powders with the pyroelectric CL spectrometer. It was observed that TbF_3 and GdF_3 glowed green and red, respectively. The illuminated area of GdF_3 was smaller than that of TbF_3 because the luminescence intensity of GdF_3 was 20 times lower than that of TbF_3 . From this result, we succeeded in carrying out elemental mapping of rare earth elements using the pyroelectric CL spectrometer.

References:

- [1] S Imashuku, A Imanishi and J Kawai, *Anal. Chem.* **83** (2011), p. 8363.
 [2] S Imashuku and J Kawai, *Rev. Sci. Instrum.* **83** (2012) p. 016106.

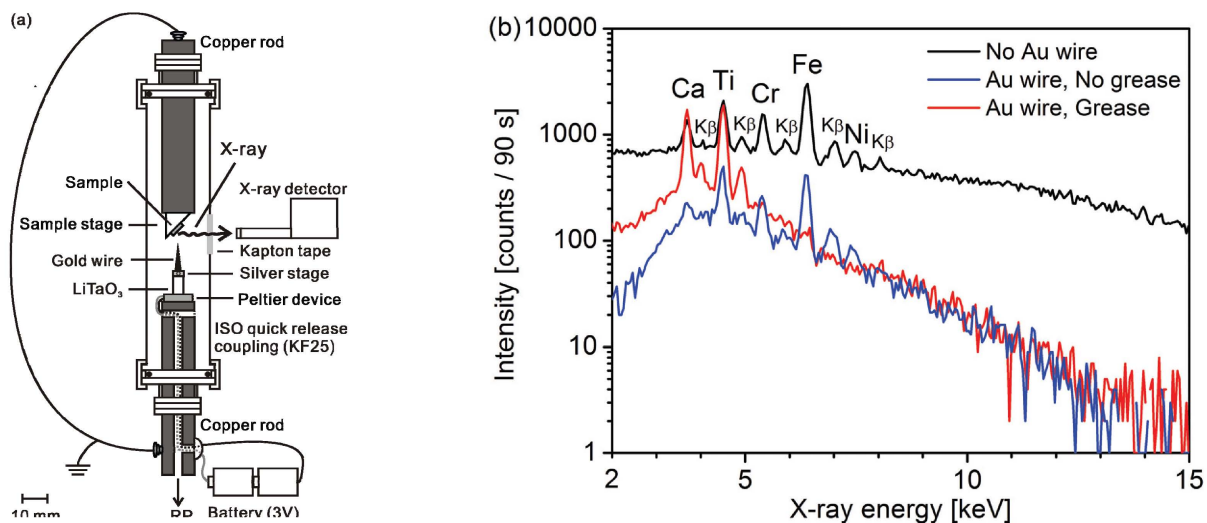


Figure 1. (a) Schematic views of palm-top EPMA. (b) EDX spectra of the mixture of CaF_2 and TiO_2 powders with and without the gold wire on the LiTaO_3 crystal.

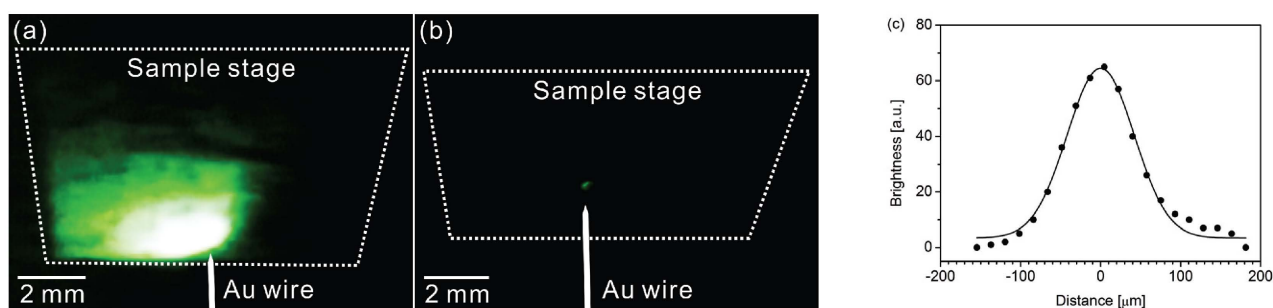


Figure 2. Photos of fluorescent screens during the bombardment of electrons. (a) Gold wire on the LiTaO_3 crystal without covering the silver stage with high-vacuum grease. (b) Gold wire on the LiTaO_3 crystal and the silver stage covered with high-vacuum grease. (c) Distribution of brightness of the illuminated area in Fig. 2 (b)

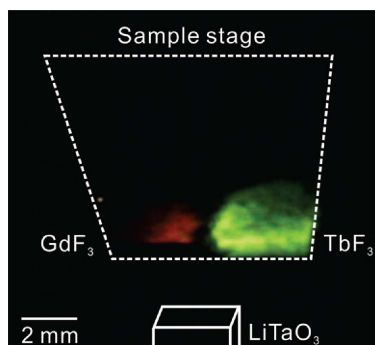


Figure 3. Photos of TbF_3 and GdF_3 powders during bombarding electrons with the pyroelectric CL spectrometer.