An X-Ray And Cathodoluminescence Study Of Cassiterite (SnO₂)

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Cassiterite ("wood tin," SnO₂) is the principal ore for tin production. Metallic tin is used in low-melting solder in electronics applications and various industrial and transportation alloys. Tin oxide has been used for gas and chemical sensor devices as particles and films. Indium tin oxide is popular in electronics applications because in thin films it is simultaneously optically transparent and electrically conductive.

A cassiterite mineral from La Paz, Bolivia was obtained from the Smithsonian's National Mineral Collection (NMHC#4903). The cassiterite specimen was cut and polished with the Leica EM TXP. The specimen was immersed in dimethyl methyl phosphate for 24 hours followed by a 500 °C furnace in air for one hour. An FEI Company Quanta 200 field emission scanning electron microscope (SEM) was used to collect electron imagery. The SEM was equipped with a Bruker XFlash 5030 silicon drift detector (SDD) for electron excited x-ray analysis. The instrument was also equipped with a Gatan MonoCL4 elite cathodoluminescence (CL) system. The CL system has an integrated back-thinned Peltier cooled CCD and a high-sensitivity photo multiplier tube [1].

The polished surface of the cassiterite crystal was examined in the SEM with high current and 15 kV accelerating voltage in high vacuum. The panchromatic CL image was collected with 300 µs dwell at 1024² pixels. Fig. 1 displays the secondary electron micrograph (a) and the false colored panchromatic CL image (b) of the same area. One striking difference between the two images is strong CL contrast along the right margin where the polished surface is unchanged in topography. The bright vertical features in the CL image are thought to be healed cracks since they are not visible in the SE image. The thinly banded diagonal features in the CL image are thought to represent zoning imparted during crystal growth [2]. The red highlighted portion of the grayscale image in Fig. 1c represents the area covered by the CL spectrum image. The true color image extracted from the spectrum image is displayed in Fig. 1d. There is no evidence of electron beam damage in this material since Fig. 1b was taken after the spectrum image collection [3]. The cassiterite spectrum is dominated by the intrinsic peak at 2.19 eV with a minor peak in the ultraviolet at 2.8 eV. No compositional explanation for the CL textures was found within the sensitivity provided by a high count rate SDD x-ray detector (Fig. 2), therefore it is likely that the CL activator is present at trace concentrations.

References

- [1] Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.
- [2] W.P. Leeman, et al, *Microsc. Microanal*. 14 Suppl.2 (2008) 38.
- [3] C.M. MacRae, et al, *Microsc. Microanal*. 15 (2009) 222.

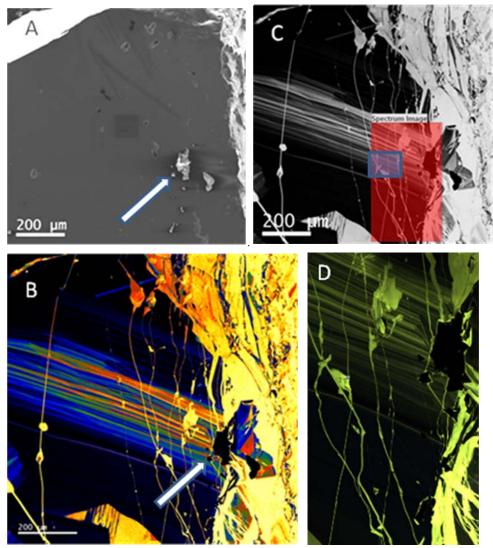


FIG. 1. Secondary electron micrograph (a) of the polished surface of the cassiterite mineral and the false colored panchromatic CL image (b) of the same area (white arrows indicate same feature). Red highlighted region (c) indicates spectrum image location and the resulting true color image (d).

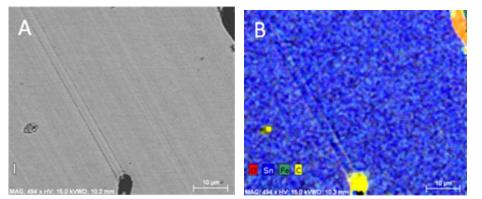


FIG. 2. Backscattered electron micrograph (a) of an area near the center of the specimen (blue highlight in Fig. 1c) and SDD x-ray map overlay (b) of the same area.