

**SEM-EDS analysis of forensic/environmental samples by utilizing
Transition-Edge Sensor Microcalorimeter detector**

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We have developed a new EDS system for SEM utilizing a transition-edge sensor (TES) microcalorimeter X-ray detector. The microcalorimeter is composed of a gold X-ray absorber, a bilayer Au/Ti thermometer, a heat sink and a thermal isolation structure with a SiN_x membrane. The operating temperature of 100 mK is obtained via a dilution refrigerator. The achieved energy resolution was 19 eV for Al K α (1487 eV) at an acceleration voltage of 5 kV. The detector has been successfully mounted onto the JSM 7000/7001(JEOL Ltd., Japan) and SUPRA 40 (Carl Zeiss Inc.) field-emission (FE)-SEM. In the present study, the SEM-TES-EDS systems have been applied to the X-ray microanalysis of forensic and environmental samples. The former samples include automotive paints and Gunshot residue (GSR). The latter sample is a single particle of suspended particulate matter (SPM). These samples were successfully analyzed by the SEM-TES-EDS system at a low acceleration voltage.

Analysis of automotive paint by using the SEM-TES-EDS system gave well resolved spectra of the S-K and Pb-M lines and the Ba-L and Ti-K lines. The energy differences between these two lines were 35 eV and 50 eV, respectively. Similarly, S-K and Pb-M lines and Ca-K and Sb-L lines were also clearly separated in the TES-EDS spectrum of GSRs. These peaks were not discernable by a conventional SSD, owing to its low energy resolution. In addition, we have successfully obtained elemental distribution map of a single particle of GSR by this system visualizing the distribution of S and Pb in the GSR. The map showed that Pb was concentrated in the particles but S distributed homogeneously.

SPM (PM2.5) collected in the urban area of Tokyo was analyzed by using the SEM-TES-EDS system, which showed good spatial as well as energy resolution for the microanalysis of the SPM. The focused ion beam(FIB) was successfully applied to fabricate a cross-section of a single SPM particle. The chemical compositional difference between surface and inner parts, and among different surface parts of the SPM was also determined. Through combination of the FIB technique and the TES microcalorimeter, we achieved a better understanding of the SPM. These results suggest that this system has high potential ability in the X-ray microanalysis of practical samples of various fields.