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Development of an Analytical TEM with a TES Microcalorimeter EDS

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Abstract: To realize accurate compositional analysis using a transmission electron microscope (TEM), we applied a transition-edge sensor (TES) microcalorimeter as the detector in energy dispersive X-ray spectroscopy (EDS). We achieved an energy resolution of 7.8eV (FWHM at Si Kα) using this TES-TEM system.

Introduction: X-ray spectroscopy is widely used for compositional analysis in addition to microstructure observation by means of a TEM. However, at present, the accuracy and sensitivity of this method are insufficient for recent advanced materials research, and more accurate analysis is required. One issue preventing accurate analysis is the low energy resolution of the detector. To solve this problem, a TES microcalorimeter has been developed and applied for more than 10 years as the X-ray detector attached with a scanning electron microscope (SEM) [1]. Although, several TES microcalorimeter can now be used with a SEM, there have been few reports on their use with a TEM. We have attempted to use the TES as the X-ray detector with a TEM to simultaneously improve the quality of compositional analysis and achieve high spatial resolution for microstructure analysis [2].

Design of the detector system: To enable the use of a TES microcalorimeter with a TEM, there are several issues to be resolved: (i) the cooling system, (ii) the hardware configuration, and (iii) the detector itself. (i) For the cooling system, we adopted a new liquid He-free cooling system [3]. This cooling system is composed of a Gifford-McMahon (GM) mechanical refrigerator and a dilution refrigerator. To avoid the propagation of mechanical vibration, the GM and dilution refrigerators are separated from each other, and only the dilution part is attached on the TEM as shown in Fig. 1. This system can endure low temperatures of below 100mK for more than 6 months. (ii) For the hardware configuration, in case of the TEM, a specimen is placed in a strong magnetic field. To avoid a mutual adverse effect between the TES device and the lens magnetic field, the detector is placed at outside the TEM column. Because the detecting solid angle becomes very small, on the order of micro-steradians, an X-ray poly-capillary lens is applied to increase the solid angle. (iii) For the detector itself, we used a single-pixel TES device as a first step. The TES thermometer is made of a stack of pure titanium and gold layers, and the absorber is a gold membrane and the size is 150*150*0.5(t) μm.

Results: Figure 2 shows the result of measurements of a silicon device (Si+W) and $BaTiO_3$. These are well-known samples for which a the standard Si(Li) detector cannot separate adjacent peaks, as shown in the figures; i.e., the Si Kα and W Mα lines in (a), and the Ba L and Ti K lines in (b) cannot be distinguished. As shown in the figure, the developed TES detector can separate them clearly. From fig. 2(a), the FWHM of the silicon Kα peak is 7.8eV, which is more than tenfold higher than that obtained

by the standard Si(Li) detector.

Recent progress: For a single-pixel detector, the acceptable count rate is very low, 100 -200 cps. To increase the count rate, we are now implementing a multiple TES array detector [4, 5]. Fig. 3 shows an example of the assembled multiple TES array detectors. In fig. 3, 10 pixels of the TES array are placed at (a). The shape of the probe needs to be a small rod with an approximate size of 1*1*10cm. To overcome the limitation of space, we have employed superconductive 3D wiring and succeeded in detecting X-rays using this detector.

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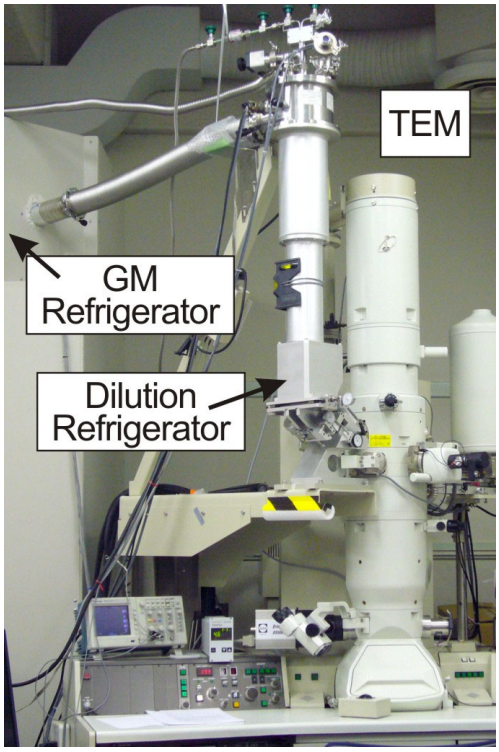

Figure 1. Appearance of the developed TES-TEM system.

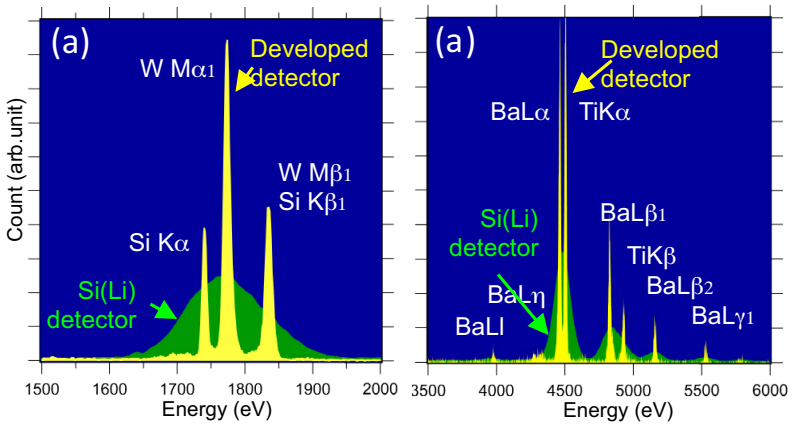

Figure 2. Spectra from (a) silicon device (Si+W), and (b) $BaTiO_3$ obtained using the developed TES-TEM system.

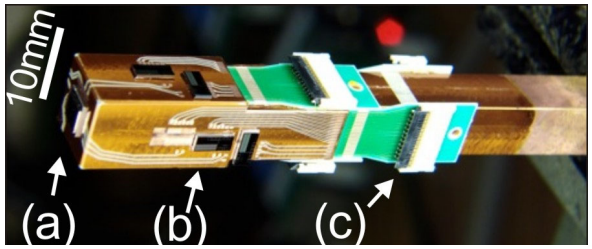

Figure 3. Multiple TES array detector. (a) TES device, (b) SQUID, and (c) edge-connector.

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