

Type of presentation: Poster

IT-5-P-2719 Development of an Analytical TEM with a Transition-Edge Sensor type Microcalorimeter EDS detector

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X-ray spectroscopy is widely used for compositional analysis in a TEM. However, the accuracy and sensitivity of this method has not been realized to the required level from recent advanced materials research. One of the main reasons preventing accurate analysis is the low energy resolution of the detector itself. The energy resolution of a standard SSD(Si(Li) type) detector is approximately 130eV, which results in considerable peak overlap. To solve this problem, we have attempted to use a superconductor transition-edge sensor(TEs) type microcalorimeter with a TEM as an EDS detector to improve the quality of compositional analysis(1).

Figure 1 shows an outlook of the first prototype of the TES-EDS mounted on a TEM. The characteristic points of this system are as follows: (i) Cryogen-free cooling system, based on a combination of a mechanical (GM type) and a dilution refrigerator, is newly developed(2). (ii) An X-ray polycapillary is applied to increase detecting solid angle. Figure 2 is an example spectrum from silicon device (Si+W) taken for system confirmation. It is well-known that a standard Si(Li) detector (dotted line) cannot separate adjacent peaks; i.e., the Si Ka and W Ma lines are overlapped to each other. As shown in the figure, the developed TES detector (solid line) can separate them clearly. The FWHM of the silicon Ka peak is 7.8eV, which is more than tenfold higher than that obtained by the standard Si(Li) detector.

The spectrum shown in Fig.2 was taken with a single-pixel TES detector mounted on the TEM (not STEM) with LaB6 thermal emitter (Fig. 1). The acceptable count rate of this detector is very low, approximately 100 cps., From these reasons, an EDS map couldn't be obtained. In order to obtain EDS map with sufficient count rate, we are now developing a multiple-pixels detector system mounted on a STEM. Figure 3 is a current situation of the developing new system; we have succeeded to obtain an X-ray map with a single pixel TES detector and confirmed the mapping function can correctly work. Multipixel detector system is now under developing to increase count-rate in order to obtain an EDS map effectively.

References:

- (1) T.Hara, et al.; "Microcalorimeter-type energy dispersive X-ray spectrometer for a transmission electron microscope", J. Electron Microsc., 59(1),(2010),17-26
- (2) K.Maehata, et al.; "A dry 3He-4He dilution refrigerator for a transition edge sensor microcalorimeter spectrometer system mounted on a transmission electron microscope", Cryogenics,(2014), in press.

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Fig. 1: Figure 1. Transition-Edge Sensor type microcalorimeter EDS mounted on a TEM.

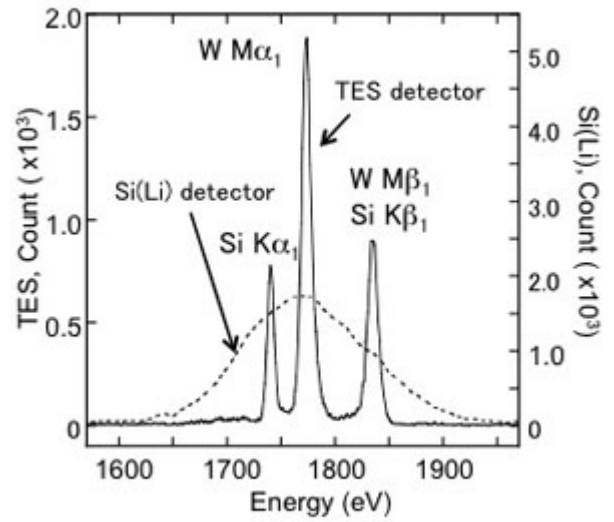


Fig. 2: Figure 2. Comparison between the TES (solid line) and the SSD(Si(Li)) (dotted line).

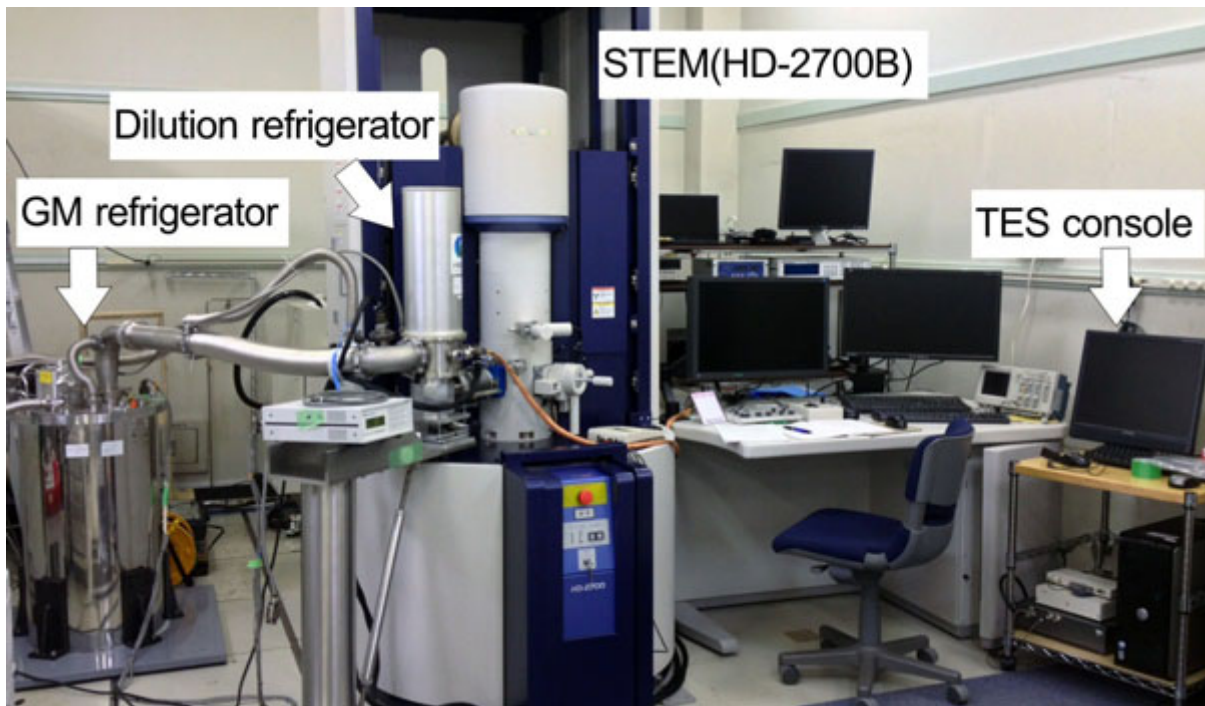


Fig. 3: Figure 1. Transition-Edge Sensor type microcalorimeter EDS mounted on a TEM.