

# Study of X-ray response of a TES X-ray microcalorimeter for STEM

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**Abstract**—We have been developing Energy dispersive X-ray Spectroscopy performed on a scanning transmission electron microscope utilizing TES X-ray microcalorimeter. The requirement is an energy resolution of 10 eV from 0.5 to 15 keV with a count rate of 20 k cps. We designed to meet the requirements and fabricated TES array of  $8 \times 8$  pixels using in-house fabrication process. The TES X-ray microcalorimeter has 40 microseconds pulse decay time. That means the TES array of  $8 \times 8$  pixel will achieve the 50 k cps counting rate. We measured energy resolution at several x-ray energy using radio isotope ( $^{55}\text{Fe}$ ,  $^{41}\text{Ca}$ ) and secondary X-rays from secondary target (Ti, Cu). We found the energy resolutions were about 10 eV in an energy range of 3 - 8 keV. Thus we consider that the device meets the requirements.

## I. INTRODUCTION

X-ray energy dispersive spectroscopy (EDS) performed with scanning transmission electron microscope (STEM) is widely used for compositional analysis in nano scale. EDS systems use the electron beam to generate a characteristic X-ray of elements.

Si-solid-state detectors are commonly used detectors for EDS. However, typical energy resolution of  $\sim 130$  eV is often inadequate to resolve closely adjacent emission lines. Also the line sensitivity and quantitative analysis of line intensities are limited by the energy resolution. In order to improve the energy resolution, utilization of TES micro-calorimeters as an EDS detector was proposed and tested [1]. We achieved an energy resolution of 8.6 eV with a single TES microcalorimeter mounted on a STEM-EDS. We obtained the element distribution on a material as a  $64 \times 64$  divided map [2]. It required  $\sim 1$  hour to map the material by electron beam scanning because the maximum count rate of TES was limited to be less than 100 cps. Due to this low counting statistic, signal to noise of the image was not quite good.

We have started up a new project, the STEM-TES-EDS, which enables to achieve detailed element mapping, high sensitivity of elements and high signal-to-noise ratio by expanding maximum count rate of TES microcalorimeter EDS detector. The requirement of this system for a TES

detector is a count rate of 20 k cps, a wide energy band of 0.5 - 15 keV and an energy resolution of  $< 10$  eV.

## II. TES MICROCALORIMETER FOR STEM-EDS

The most challenging point of this project is to achieve the count rate of 20 kcps. The maximum counting rate of a single TES X-ray microcalorimeter is limited by a typical signal decay time to be about 300 cps. We then adopted an  $8 \times 8$  sensor array which is operated by parallel read-out electronics, and work as a large single sensor.

The sensor size, heat capacitance, thermal conductance, transition temperature were designed to achieve these requirements. We selected transition temperature as 200 mK by Ti/Au bilayer with a thickness of 40/90 nm. The TES detector consists of a  $180 \times 180 \mu\text{m}^2$  and  $120 \times 120 \mu\text{m}^2$  Au absorber with a thickness of 2.5  $\mu\text{m}$ . The thickness of Al electrodes and  $\text{SiO}_2/\text{SiN}_x$  membrane are respectively 120 nm and 0.5  $\mu\text{m}/1 \mu\text{m}$ . The membrane size is 300  $\mu\text{m}$ .

We fabricated the TES X-ray microcalorimeter array using in-house fabrication process. We show a microscope photograph of single pixel TES X-ray microcalorimeter that we fabricated (Fig.1).

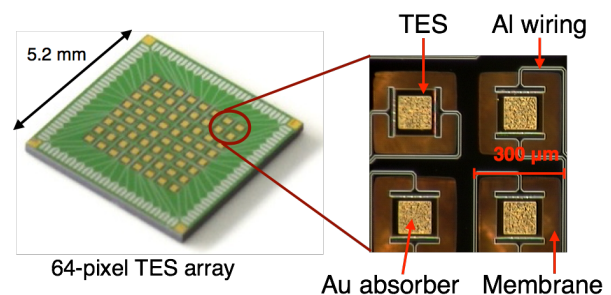


Fig.1(Left) TES array of  $8 \times 8$  pixels that we fabricated, (Right) Microscope photograph of the single pixel TES X-ray microcalorimeter from top

### III. PERFORMANCE OF THE TES X-RAY MICROCALORIMETER

We measured the RT and IV curve of TES sensor, and performed X-ray irradiation test. We found that transition temperature is about 200 mK and the transition curve of 7 pixels is almost uniform. Energy resolution for X-ray at  $^{55}\text{Fe}$  (5.9 keV),  $^{41}\text{Ca}$  (3.3 keV) are measured as to be 7.26 eV and 7.30 eV respectively (Fig.2). The signal decay time was 40  $\mu\text{s}$  (Fig.2). With this decay time, we expected to achieve 50 k cps by the  $8 \times 8$  array.

As a wide-band EDS system, proper understanding of X-ray response is essential to identify emission lines from several elements. We attached a small X-ray generator directly on a test chamber, and irradiated fluorescent lines from Ti (4.5 keV) and Cu (8.0 keV). We could successfully obtain the spectrum Ti and Cu and the energy resolutions (FWHM) were 10 eV and 8.9 eV respectively (Fig. 2). The required energy resolution of 10 eV was proofed in an energy band of 3 - 8 keV.

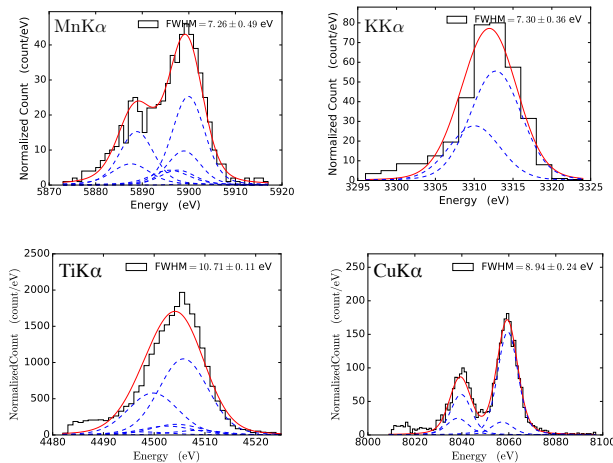


Fig2. The spectrum of MnK $\alpha$ ,KK $\alpha$ ,TiK $\alpha$ ,CuK $\alpha$  that obtained a single pixel TES X-ray microcalorimeter.

At present, we measure the current change through a TES by a SQUID. The relation between the pulse height and energy is represented by a power function ( $\text{PHA} = aE^c$ ). We defined that  $c$  equals 1 is to be linear and  $c$  less than 1 is to be non-linear. In typical measurement,  $c$  is to be 0.88. To obtain the best TES response in a wide energy range, improvement of the non-linearity of TES response is preferable. A possible method to revise this nonlinearity by transforming the TES current space into a resistive space is proposed (Bandler et al. 2004). We employed this method by Ti/Cu fluorescent lines. The value of  $c$  has been improved to 0.96.

### IV. CONCLUSION

We designed and fabricated  $8 \times 8$  pixel TES microcalorimeter for STEM-EDS. We found the energy resolutions were 10 eV or less at an energy band of 3 - 8 keV while requirement is 10 eV. We found pulse decay time of a single pixel TES is 40  $\mu\text{s}$  while the requirement is  $< 100 \mu\text{s}$  to obtain 300 cps/pixel. We could fabricate the TES X-ray microcalorimeter to meet the requirements of STEM-EDS and evaluate the TES X-ray microcalorimeter.

### REFERENCES

1. Toru Hara, Keiichi Tanaka, Keisuke Maehata, Kazuhisa Mitsuda, Noriko Y. Yamasaki, Mitsuaki Ohsaki, Katsuaki Watanabe, Xiuzhen Yu, Takuji Ito and Yoshihiro Yamanaka, "Microcalorimeter-type energy dispersive X-ray spectrometer for a transmission electron microscope" *Journal of Electron Microscopy* 59, no.1, pp.17-26, (2010).
2. K. Maehata, T. Hara, K. Mitsuda, M. Hidaka, K. Tanaka, and Y. Yamanaka, "A Transition Edge Sensor Microcalorimeter System for Energy Dispersive Spectroscopy Performed on a Scanning-Transmission Electron Microscope" *J Low Temp Phys* (2015), submitted for publication.
3. Haruka Muramatsu, In-house fabrication of TES X-ray microcalorimeter Arrays and Study of their X-ray response, Master thesis, University of Tokyo (2016), unpublished