Development of a 200-pixel TES X-ray microcalorimeter for STEM-EDS system towards microanalysis of astromaterials

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1. Introduction

Why is the Earth water-rich now?
- There are clues in the formation process of our solar system.

Astromaterials have
- Information about the early phase solar system,
- Sub-micro scale structures,
- Various elements such as C, O, 1s, Mg, Fe...

Microanalysis with STEM+EDS
(Astromaterials come to the Earth as meteorites or by way of sample return missions, such as Hayabusa2.)

We have been developing
- 64 pixels TES-EDS and optimized detector head

Abstract

Studies on astromaterials, including sample-return missions, such as HAYABUSA2 and OSIRIS-REx, provide valuable insights into the formation and evolution of our solar system. Astromaterials, which comprise both organic and inorganic materials, can be analyzed at a sub-micrometer scale using energy-dispersive X-ray spectroscopy (EDS) in combination with scanning transmission electron microscopy (STEM). In a previous study, we developed a 64-pixel transition edge sensor (TES) X-ray microcalorimeter array as a detector for the EDS system; the full width at half maximum (FWHM) energy resolution was 7 eV at 6 keV under a few hundred cps [Hayashi et al. 2018]. However, in this system, the counting rate and sensitivity of the low energy band were insufficient, and the counting rate was limited by the number of pixels.

To improve the counting rate, we designed a 240-pixel TES X-ray microcalorimeter array with a mushroom-type absorber. For large absorbers, such as mushroom-type absorbers, the residual resistivity ratio (RRR) is an indicator of thermal diffusion and limits TES performance. We created an electroplating environment and achieved an RRR of approximately 28. We optimized the design of the 240-pixel TES array to cover a 5-mm-diameter focus area of the polycapillary optics in STEM. A micro strip line was used to place the 240-pixel TES on the 5-mm-diameter focus area. We optimized the fabrication process of the mushroom type absorber and the micro strip line. We have been developing the microwave SQUID multiplexer (MWMUX) system in our dilution refrigerator to read out 80-pixel out of the 240-pixel TES microcalorimeter.

2. Transition Edge Sensor X-ray microcalorimeter

- X-ray photon → changing temperature \( \Delta T = E/C \)
- Using changing the resistance of TES for measuring \( \Delta T \)
- Energy resolution \( \Delta E = \frac{C}{\sqrt{1 + 2 \times 10^5}} \) where \( C = \frac{1 \text{ pixel}}{100 \text{ mK}} \)
- \( \Delta T \) = 50 times better than that Semiconductor type detector
- Biased with a constant voltage, a strong electro-thermal feedback (ETF) is obtained, resulting in a >10 times faster response
- Factor of ~10 or more counting rate:
  - Advantage for ground applications, such as material analysis.

3. Developing TES EDS system

- Energy resolution: <10 eV
- Counting rate: <5 kcps
- Energy band: 0.1 - 15 keV

The TES is designed and fabricated by myself using the IC process.

We was able to
- detect an emission line of Cu L, Si K, O Kα...
- detect a self absorption of specimen
- resolve the continuum and the line emissions within the 10 times.

4. Quantitative analysis

- We obtained the spectrum from standard olivine sample.
- Determination of quantitative analysis by model fitting.
- We obtained SiO\(_2\) spectra and calculated Z\(_{Si}/Z\(_{O}\) from O to Si ratio

\[ \frac{Z_{Si}}{Z_{O}} \geq 2.2 \times 0.3 \]

result is consistent with SiO\(_2\) weight.
- (Number-density of SiO\(_2\) ratios Si to O is 2)

However, the system needs a high counting rate.

5. Developing mushroom type absorber

- We optimized the design of the 240-pixel TES array and a mushroom type absorber to cover a 5-mm-diameter.
- For realize the mushroom absorber structure in-house process, we introduced and constructed the electroplating environments.
- We achieved the gold of high conductivity using the electroplating (RDR~25).
- The fabrication process of mushroom type absorber are established.
- We have succeeded in developing a TES array with a mushroom type absorber.
- The absorber size is 260 × 260 \( \mu \)m\(^2\) with a gap of 40 \( \mu \)m.

6. Developing dilution fridges with MWMUX

- Microwave SQUID (MWMUX) system are basic technology for readout large TES array.
- We will then introduce the MWMUX system to our group’s refrigerator.
- In 2023 to 2024, we will construct the analysis methods for quantity study of astromaterials using the 80 pixels TES-EDS system.

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