A transition edge sensor microcalorimeter system for the energy dispersive spectroscopy performed on a scanning-transmission electron microscope

Main author:

MAEHATA Keisuke

Co-authors:

Hara Toru, National Institute for Materials Science

Hidaka Mutsuo, National Institute of Advanced Industrial Science and Technology

MAEHATA Keisuke, Kyushu University

Mitsuda Kazuhisa, Japan Aerospace Exploration Agency

Tanaka Keiichi, Hitachi High-Tech Science Corp.

Yamanaka Yoshihiro, Taiyo Nippon Sanso Corp.

Energy-dispersive spectroscopy (EDS) using X-rays performed with electron microscopes plays an important role in a wide range of sciences, including applications in materials science and medical science. EDS coupled with electron microscopy allows quantitative composition analysis within very small local structures by a rather simple operation. Usually Si(Li) semiconductor detectors (SSDs) are used for X-ray detection in the typical EDS systems of electron microscopes. The accuracy of the EDS is limited by the energy resolution of the X-ray detectors. The energy resolution of SSDs has reached the theoretical limit of 120 eV full width at half maximum (FWHM) at 5.9 keV. In addition, recent advanced materials research has indicated that a high energy resolution EDS system is required for use with scanning transmission electron microscopes (STEM). It is therefore necessary to develop X-ray detectors with an energy resolution superior to that of the SSDs for the next generation of EDS performed on the STEM.

We have therefore developed a transition edge sensor (TES) microcalorimeter EDS system operating on a STEM. The TES microcalorimeter was placed outside the STEM column to avoid the influence of the magnetic field generated by the objective lens of the STEM. The solid angle of the EDS system decreases with the inverse square of the length from the STEM specimen to the absorber surface of the TES microcalorimeter. In the TES microcalorimeter EDS system installed on the STEM, an X-ray polycapillary optic was employed to transport the X-rays emitted by the STEM specimen to the TES microcalorimeter placed outside the STEM column. Because it is important to maintain the operating temperature of the TES microcalorimeter with sufficient stability over long periods of time without excessive liquid helium consumption, a compact dry ^{3}He-^{4}He dilution refrigerator (DR) was developed. A remote helium cooling loop system with Gifford-McMahon (GM) cryocooler was employed for precooling the DR with preventing the noise and vibrations. A single pixel TES microcalorimeter EDS system was installed in a STEM for proving tests of our concept. The TES microcalorimeter consists of a Au/Ti bilayer TES and an absorber layer of Au. The transition temperature and the sensitivity α were 172 mK and 180, respectively. An energy resolution was found to be 8.6 eV FWHM at Ir $M_{\alpha1}$ (1979.9 eV), Pt $M_{\alpha1}$ (2050.5 eV) and Ir M_{β} (2053.5 eV) peaks in the X ray energy spectrum for a Ti-Ir-Pt alloy specimen. An element distribution map of Si, Ti and W was obtained with the energy resolution of 9.7 eV FWHM at Si K_{α} (1740 eV) in analytic operation for an electronic device specimen. Although element analysis with the energy resolution superior to that of SSDs was demonstrated in proving tests of the single pixel TES microcalorimeter EDS system installed the STEM, the counting rate of 300 counts per second (cps) was insufficient for a practical application to the STEM. To achieve the counting rate larger than 5000 cps we are now conducting development of a 64 pixel TES microcalorimeter EDS system operating on the STEM.

In this presentation, we report the single pixel TES microcalorimeter EDS system installed in STEM, experimental results of the proving tests and brief introduction of the 64 pixel TES microcalorimeter EDS system.