

Analytical STEM applying a superconductor transition-edge sensor type microcalorimeter EDS for x-ray nanoanalysis

Hara, T.¹, Tanaka, K.², Maehata, K.³, Mitsuda, K.⁴, Yamanaka, Y.⁵, Hidaka, M.⁶ and Nakamura, K.²

¹ National Institute for Materials Science, Japan, ² Hitachi High-Technologies Corporation, Japan, ³ Kyushu University, Japan, ⁴ Japan Aerospace Exploration Agency, Japan, ⁵ Taiyo Nippon Sanso Corporation, Japan, ⁶ National Institute of Advanced Industrial Science and Technology, Japan

Microstructure characterization is essentially important to develop new materials. Electron microscopes, such as scanning transmission electron microscope (STEM), plays an important role for such purpose. A function of compositional analysis in nano-scale, in addition to microstructure morphological observation, is very important for current microstructure characterization. In order to analyze the composition in electron microscopes, x-ray spectroscopy is now widely applied. In order to realize precise x-ray analysis in electron microscopes, we have been trying to apply a transition-edge sensor (TES) microcalorimeter detector for x-ray spectrometry with a STEM. This detector has high energy resolution, such as lower than 10eV, while that of the SSD is around 125eV. With using this detector, it is expected that most of all elements can be measured without any peak overlaps, it leads high accuracy and sensitivity. To ensure high spatial resolution, less than 1nm, we adopt the TES detector onto a STEM.

Our target specs of the developed x-ray detector are as follows: i) x-ray energy resolution: <10eV, ii) detecting x-ray energy range: 0.5-10keV or wider, iii) x-ray counting rate: >5kcps. Applying this x-ray detector with a STEM whose accelerating voltage of 200kV, we will be able to realize the elemental mapping with a spatial resolution of the x-ray map less than 10nm. In order to realize the specs listed above, we developed a new detector system. The characteristic points of the detector are as follows: i) detector device is 64 pixel TES to increase countrate more than 5kcps, ii) a polycapillary x-ray lens is applied to increase detecting solid-angle, iii) compact Liq.He free cooling system has been developed for high stability. Newly developed cooling system, based on a dilution refrigerator, can keep low temperature more than half a year, so that we can measure anytime with the same condition. We have installed this detector onto a STEM (Hitachi HD-2700B) and developed the new analytical STEM. By this system, we succeeded to obtain x-ray spectra with high energy-resolution, less than 10eV, for the range from 0.5-15keV. In this report, I will briefly review the recent progress of development on the analytical STEM applied with the multipixel TES detector system with some applications.

This work is supported by 'Development of advanced measurement and analysis systems, JST-SENTAN', Japan Science and Technology Agency.