STEM-based thermal analytical microscopy

kawamoto, N.[1], Kakefuda, Y.[1], Yamada, I.[2], Tsujii, N.[1], Mitome, M.[1], Bando, Y.[1], Mori, T.[1,3] and Golberg, D.[1,4]

[1] International Center for Materials Nanoarchitectonics (MANA), National Institute for Materials Science (NIMS), Japan, [2] Yamada R&D Support Enterprise, Japan, [3] University of Tsukuba, Japan, [4] Queensland University of Technology (QUT), Japan

It becomes timely important to perform simultaneous analysis of nanostructures and their physical properties, such as electrical, mechanical, magnetic, optical, and thermal, in a scanning transmission electron microscope (STEM). For example, *in situ* transmission electron microscopy (TEM) combined with a probing technique using a dedicated TEM holder with piezodriven mechanics has revealed electrical properties of free-standing individual carbon nanotubes [1], mechanical [2] and optical dynamic phenomena in various nanostructures [3,4]. Besides, it is of prime interest to measure thermal properties and to observe thermo-induced phenomena in nanoscale materials, e.g. heat-sink composites, thermoelectric materials, thermal diodes, and so on. Especially, it is highly desirable to develop an advanced technique for simultaneous observation of a given structure, *e.g.* its lattice defects, grain boundaries, and impurities which induce phonon scattering, and measuring its thermal transport properties.

In this study we report on the development of STEM-based thermal analytical microscopy (STAM) (Fig. 1) by combining nanoscale temperature measurements using the world-smallest thermocouple assembled in TEM with a scanning heat input from a focused electron beam using the STEM mode in a 300 keV JEM-3100FEF microscope. Non-magnetic constantan and chromel nanoprobes [5,6] for assembling a nanoscale thermocouple were prepared by electrochemical etching. The diameter of the assembled thermocouple could be controlled to be below 10 nm. As a model material we prepared a thinned TEM specimen made of $CuFeS_2$ by using a focused ion beam. The specimen thickness was controlled to be uniform at about 300 nm. This allows us to input a sufficient heat in it under a convergent electron beam, 20 nm in diameter, using the STEM mode.

In order to measure the local temperature of a TEM specimen, the nanoscale thermocouple was attached to its edge, as shown in a high-angle annular dark field (HAADF)-STEM image of Fig. 2. A STAM image during thermal analysis was obtained by using an electron beam-induced heat scan at a constant current. STAM image shows a two-dimensional temperature distribution map constructed by measuring generated thermoelectromotive force acquired at point M where the thermocouple has been attached. In the presentation, we will analyze the thermal transport within the sample and the data acquired under high spatial and temperature resolutions.

References:
[1] Z. L. Wang et al., J. Phys. Chem. Sol. 61 (2000) 1025.
[2] D. M. Tang et al., Nature Commun. 5 (2014) 3631.
[3] C. Zhang et al., Nanotechnology 26 (2015) 154001.
[4] O. Cretu et al., Appl. Phys. Lett. 110 (2017) 111904.
[5] N. Kawamoto et al., Nanotechnology 22 (2011) 485707.
[6] N. Kawamoto et al., Nanotechnology 26 (2015) 465705.

Acknowledgement:
This work was supported by JSPS KAKENHI Grant Number JP 26820287, 23760626. It was also supported by JST CREST Grant Number JPMJCR15Q6, Japan. D.G. is particularly grateful to the Australian Research Council (ARC) for granting a Laureate Fellowship (Project FL160100089).

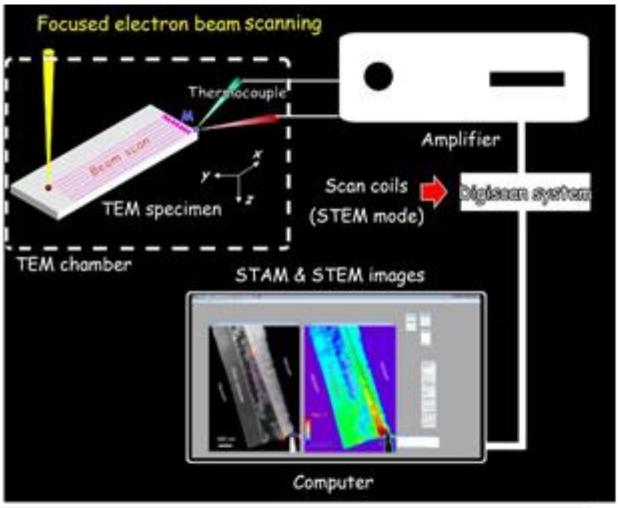

Fig. 1: Schematic diagram of the STEM-based thermal analytical microscopy (STAM).

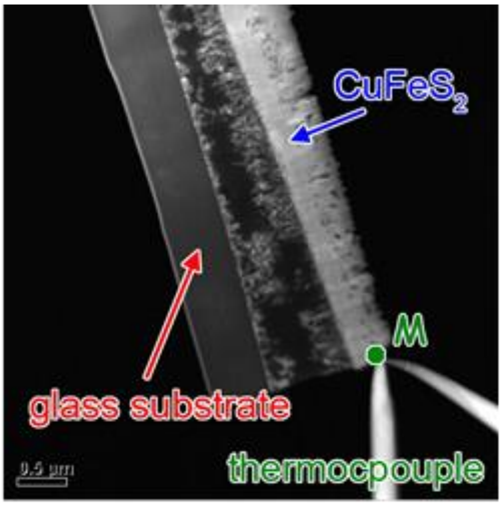

Fig.2: HAADF-STEM image of a cross-sectional specimen consisting of $CuFeS_2$/impurity substrate/glass substrate with a thermocouple attached to the edge of $CuFeS_2$ portion at M.