Visualizing nanoscale heat pathways

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1. Introduction

With respect to the primary energy sources, like oil, gas, and coal, that mankind consumes, only one third is effectively used, while two thirds are being lost in some form, of which the majority is waste heat. Therefore, we need to find advanced ways to control thermal energy. An important route toward achieving this ambitious goal is the ability to characterize thermal phenomena at various length scales, preferably down to the nano-dimensions. This is particularly important for in-homogeneous substances, such as heat-sink materials (typically composed of a resin and sub-micrometer ceramic fillers [\textsuperscript{1}]), nanocomposite thermoelectric materials [\textsuperscript{2}], etc. for example, to design an efficient heat-sink material it is critical to have dynamical knowledge on where the heat transport is impeded, on which kind of interfaces it is slowed and so on. Although energy saving by thermal control is very important, it is still particularly difficult to perform precise thermal measurements as compared to evaluation of other physical properties.

Several methods have been invented for testing thermal conductivity of low-dimensional materials, e.g. nanostructured thermomaterials [\textsuperscript{3\textendash}7]. Recently, a combination of nanoscale heating source, e.g. electron beam and resistance thermometers, has also been proposed [\textsuperscript{8,9}]. However, a fully quantitative method, which is compatible with high temperature resolution, and high spatial and thermal probe positional resolutions, has still not been realized, although there is an extremely high demand to better evaluate and understand heat transport within nanostructures and the role of defects in them, e.g. interfaces, grain boundaries, impurities and so forth.

Transmission electron microscopy (TEM) is capable of observing micro- and nanostructures with unprecedentedly high-spatial resolution, reaching 50 pm these days. In recent years, the remarkable development of in situ methods for simultaneous access to electrical [\textsuperscript{10}], mechanical [\textsuperscript{11}] and optical dynamic phenomena [\textsuperscript{12,13}], using micropores attached to individual low-dimensional materials, in relation to their detailed atomic and defect structures has been accomplished. Over the last decade, unique techniques for temperature measurements in spatially localized areas were designed as a potential technology for...
thermal conductivity studies in TEM [14,15]. However, in order to reveal a clear relationship between nanostructure peculiarities, e.g. lattice defects and impurities, and phonon transport, and to in situ observe phonon and carrier behaviors in a desired spot/region of a material, a new highly responsive method is required. In addition, although a scanning thermal microscopy for two-dimensional thermal imaging of a composite material surface using a microfabricated thermocouple has been reported [16,17], only the thermal conductivity of particle surfaces, and only qualitatively, can be evaluated [18]. Furthermore, such method is not suitable for investigating thermal flows inside and/or between particles which are non-uniformly dispersed. It also suffers from a continuous change in contact thermal resistance while scanning.

Herein we report on an original method for characterizing thermal behavior at the nanoscale, while analyzing desired locations within an inhomogeneous heat-sink material as the model sample. Thermo-electromotive force, as a function of temperature changes during non-contact nanoscale thermal scanning over the sample (under convergent electron beam irradiation in a scanning transmission electron microscopy (STEM) mode), is measured employing a nano-thermocouple fixed to the specimen. This method, namely, STEM-based thermal analytical microscopy (STAM), is able to obtain detailed information in the form of two-dimensional thermal distribution maps revealing heat conduction in the fillers embedded in a composite material and/or between them. The temperature distribution and nanoscale heat pathways could also be visualized. The prime advantage of the developed technique is its thermal map resolution, up to ~20 nm, which allows for the first time observation of a specific nanoscale thermal “neck” in a composite heat-sink material. The advantage of this method is that it would easily be applied to any material (if the sample of interest can be prepared using a focused ion beam (FIB)).

2. Experimental setup

2.1. Setup for nanoscale temperature measurement in TEM

Fig. 1a shows an optical image of the tip of a twin-probe scanning tunneling microscopy (STM)-TEM holder [19] manufactured by “NanoFactory Instruments AB”. As shown in Fig. 1a, the W base supporting a thinned TEM specimen is fixed with a conductive Ag paste to the frame portion of the holder. Two individually piezo-driven nanoprobes made of constantan and chromel alloys are merged into a nanoscale thermocouple for local temperature measurement, as shown in the schematic view of Fig. 1b. These ultimately thin metal probes were prepared by electrochemical etching. They were then fixed into holes of the Cu movable jigs, as depicted in Fig. 1a. The diameters of original chromel and constantan wires were 200 µm. The tip diameters of both probes after electrochemical etching were controlled to become only several nanometers. The tip portions of probes were joined under accurate nanomanipulations inside TEM using piezo-driven motors of the twin-probe holder, thus creating the nanothermocouple as shown in a high-resolution transmission electron microscope (HRTEM) image of Fig. 1c. After electrical current and e-beam treatments of the joint, the electrical internal resistance between two probes became as low as 1 kΩ, or even less. After applying a current up to 100 nA the wire connection shows pure Ohmic behavior. Hence, it is confirmed that the junction is made of perfectly physically bonded metal surfaces where the electromotive force is generated under an instantaneous temperature change. The reference temperature for a thermocouple calibration was the temperature of the junctions R1 and R2 on the Cu jigs holding constantan and chromel probes, as shown in Fig. 1b. The temperatures of R1 and R2 are usually kept at about 298 K (room temperature). A typical thermocouple made of the pair of chromel and constantan probes shows the Seebeck coefficient of 61 µV/K at room temperature. As seen in Fig. 1a and b the points R1 and R2 are separated by a distance of ~3 mm, or more, from the junction point M in Fig. 1b and c. Considering the heat capacity of both metallic wires (having a diameter of 200 µm) and large heat capacity of the Cu jigs, supporting sapphire balls and metal rods of the holder, the reference temperatures at R1 and R2 should not be affected by a temperature increase (within at least several 10 K) with respect to the junction point M, which has a small heat capacity (less than 1/2000 of the portions R1 and R2). In fact, our past experiments [20] have confirmed that the thermocouple temperature returns to a steady state within at least 100 ms, or less, after spot heating by a focused electron beam. It was also theoretically established that the TEM specimen of dimensions similar to ours would return to in a steady state within 10−6 s or less after electron beam irradiation [21].

2.2. TEM specimen preparation for thermal measurement

A heat-sink composite material was prepared by curing alumina fillers (with dimensions ranging from 30 nm to 10 µm) in a thermosetting epoxy resin. The epoxy, having low thermal conductivity, as an adhesive, enters the gaps between tightly connected fillers. A thinned TEM specimen for thermal measurements was prepared by using a FIB system (JEM-9320FIB, JEOL) with an accelerating voltage of 30 kV. The specimen with dimensions of 5 µm × 10 µm × 250 nm was cut from the bulk composite and placed onto the W base using an epoxy resin with low thermal conductivity, as an adhesive, enters the gaps between tightly connected fillers. A thinned TEM specimen for thermal measurements was prepared by using a FIB system (JEM-9320FIB, JEOL) with an accelerating voltage of 30 kV. The specimen with dimensions of 5 µm × 10 µm × 250 nm was cut from the bulk composite and placed onto the W base using an epoxy resin with high thermal insulation. A scanning ion microscopy (SIM) image (Fig. 1d) shows a cross-sectional view of the prepared sample of uniform thickness, 250 nm. The sample fixed onto the W base (painted in red) and a thermal insulating epoxy layer (indicated in blue) are illustrated in Fig. 1d.
2.3. Conditions for STAM analysis

High-resolution microscopes (300 kV JEM-3100FEF and 200 kV JEM-2800, JEOL) were used for microstructural observations in TEM and STEM modes. STAM measurements and electron energy loss spectroscopy (EELS) tests were carried out using the JEM-3100FEF instrument. In order to carry out the nanoscale thermal conduction measurement in TEM, as illustrated in Fig. 1b, the position of the heat input was alternated by using a scanning convergent electron beam, 20 nm in diameter, having a current of ~10 nA. The dissipated power \( P_o \) of the electron beam can be expressed as follows [21]:

\[
P_o = \frac{\pi r_j^2 j \Delta E}{\epsilon \Lambda} t
\]

Where \( r_j \) is the radius of the electron beam, \( j \) is the current density, \( \epsilon \) is the elementary charge, \( \Delta E \) is the plasmon energy loss, \( \Lambda \) is the electron mean free path, \( t \) is the thickness of specimen. In the case of this study, \( P_o \) could be estimated to be \( 4.2 \times 10^{-7} \) W from the values of \( \Delta E = 24 \) eV, and \( \Lambda = 143 \) nm [22] for Al\(_2\)O\(_3\). In order to obtain a STAM image with the high S/N ratio and spatial resolution, a large amount of heat input is desirable. The beam current can be increased by changing the size of apertures and the extracted voltages of electrons in the field emission gun. For instance, the size of an electron beam is controllable to a few nm by choosing a small aperture, while, the current of the electron beam becomes 1/10 or less. In such a case, it is difficult to properly analyze a STAM image due to low S/N ratio. Hence, we used the experimental parameters as mentioned above. The temperature was measured at the tip of the thermocouple which was brought into contact with the corner of a TEM sample under heat input scanning. The sample thickness, about 250 nm, has been small enough to reduce the influence of multiple scattering (while considering the mean free path of 300 kV electrons travelling in alumina [22]). Such sample thickness also suffices to effectively generate the heat through interactions of plasmons in a specimen [21]. The electron beam-induced heat passes through the TEM specimen in the direction of the attached metallic thermocouple as a heat-sink. The sample is well thermally insulated due to surrounding high vacuum of \( 10^{-5} \) Pa in the TEM column. The high thermal resistance of the large volume of epoxy (blue region in Fig. 1d) blocks the heat transfer to the W base. Thus, the input heat can be assumed to flow through a specimen toward the attached metallic thermocouple. STAM images as two-dimensional maps were constructed from thermoelctromotive force outputs measured by a voltmeter (7461A, ADCMCT).

3. Results and discussion

White contrast areas in a high-angle annular dark-field (HAADF)-STEM image (Fig. 2a) denote small alumina fillers \( A \) and \( B \), with diameters of 1.5 \( \mu \)m and several hundred nm, in addition to the large fillers \( I \), \( 2 \), and \( 3 \), with diameters of \( \sim 10 \) \( \mu \)m. Also, numerous smaller fillers, with diameters of several tens of nanometers, are seen contacting each other. They are uniformly dispersed in a thermo-resistive resin (dark gray contrast, Fig. 2a). Fig. 2b is a bright field (BF)-TEM image of the region indicated by the blue-dashed frame in Fig. 2a. The tip of the thermocouple is brought into contact at the temperature measuring point \( M \) (lower left corner in Fig. 2b). The aspect ratio of HAADF-STEM image (Fig. 2a) is different from that of BF-TEM image (Fig. 2b) due to sample tilting after physical contacting of the thermocouple to the specimen. From standard morphological observations based on ordinary TEM and STEM imaging, it is impossible to judge how heat flows propagate within or between the fillers. As discussed above, it is worth noting that some methods for evaluating thermal transport have indeed been proposed [15,18], but they have difficulties in visualizing complex heat flow pathways in a composite material due to limitations with respect to spatial and temperature resolutions, positioning accuracy, and controlling the location of heat inputs/outputs. Fig. 2c shows an enlarged HAADF-STEM image of the red dashed frame in Fig. 2a. In addition to medium size fillers, such as those marked as \( A \), \( B \), and \( C \), a multitude of fillers with smaller diameters (several tens nm), as the one denoted as \( D \), can be seen being sandwiched between large fillers \( I \) and \( 2 \). In order to investigate the complicated heat conduction paths from filler \( 2 \) to filler \( I \) in Fig. 2a, it is necessary to acquire thermal information within only several tens nm or even smaller areas.

Fig. 3a is a HAADF-STEM image of the area surrounded by a yellow dashed-dotted frame in Fig. 2a. The main field of the image is taken by filler \( 1 \). The point \( M \), at which the tip was attached to the sample, is also visible. The blackest contrast areas are vacuum regions. The white dashed line in Fig. 3a indicates a grain boundary. The grain \( a \) of the filler \( 1 \) is considered to be a single crystal based on \( Z \) contrast imaging and electron diffraction analysis. In order to obtain a STAM image of the same area, shown in Fig. 3a, the heat injection position was alternated under convergent 20 nm electron beam scanning (at approximately 50 nm intervals) with the irradiation time of 1 s at each point. This irradiation time of the electron beam was long enough to reach the steady heat conduction state, as discussed in Section 2.1. The time of the thermocouple is brought into contact at the temperature measurement point \( M \) in a superimposed HAADF-STEM image in Fig. 3b. The purple color corresponds to the maximum temperature increase at the point \( M \) in a reference HAADF-STEM image in Fig. 3b. The maximum value of the color bar in Fig. 3b corresponds to 33 \( \mu \)K of the measured voltage, whereas the minimum value to 0 V (vacuum region). According to a gradual color change (from purple to black), an overall two-dimensional color map was constructed based on the elevation of temperature at the point \( M \). It should be noted that the amount of heat injected by the electron beam depends on the sample thickness and electron density of the material [21]. In addition, as the heat input goes through the sample, the temperature at \( M \) (and the measured potential) decreases according to the Fourier’s law. In other words, while discussing the thermal conductivity...
based on the proposed method, it is necessary to analyze the temperature gradient in the specimen. And such sample should have a uniform thickness and have no difference in electron density. While acquiring a line profile of the relative sample thickness determined using EELS [23] in the interval X-Y (Fig. 3a and b), a thickness graph was drawn (Fig. 3c). This shows the relationship between a distance from X and contrast intensity corresponding to sample thickness. As seen from the line profile of the relative thickness (Fig. 3c), the sample thickness increases to the maximum value at the point Z located about 1.2 µm from X. The specimen thickness from Z to Y is almost uniform. With respect to the STAM image (Fig. 3b), a line intensity profile of the section X-Y could also be obtained, as depicted in Fig. 3d. The temperature measured at point M gradually increases due to increasing input heat with increasing sample thickness from X to Z. On the other hand, although the measured section from Z to Y is composed of a single crystal grain of uniform thickness, the temperature decreases

Fig. 3. (a) HAADF-STEM image focusing on the large filler 1; i.e. the enlarged area surrounded by the yellow dashed-line frame in Fig. 2(a). (b) STAM image corresponding to the area of (a). The maximum voltage value corresponding to the lower left color bar is 33 μV. The lower left image is a superimposed HAADF-STEM image. (c) Line profile of the sample thickness distribution within the section X-Y (shown in (a)) obtained via EELS analysis. (d) Line profile of the thermoelectromotive force measured at the point M through the section X-Y marked in the STAM image of (b). (e) Line profile of the normalized intensity within the section Z-Y calculated by dividing the line profiles of STAM with the relative sample thickness profile. In the section Z-Y of uniform thickness the temperature gradually decreases as the distance increases from M (according to the Fourier’s law).

Fig. 4. (a) Enlarged BF-TEM image of the area surrounded by the black dashed-line frame in Fig. 2(a). Filler D are in contact with the small filler C and the larger filler 2. (b) STAM image of the same area as in (a). The maximum value of the lower left color bar corresponds to a voltage of 7 μV. (c) Intensity profile for the sample thickness measured under EELS over the section T0-T1 in (a). (d) Line profile of the STAM image of the same section T0-T1 as in (c).
monotonically from Z toward the Y spot. Fig. 3e shows a normalized line intensity profile from Z to Y calculated by dividing the line profiles of the STAM by the relative sample thickness graph. As indicated by the dashed lines (Fig. 3e), the difference between the intensities at Z and Y is clearly apparent as the distance from Z increases. This means that the integrated thermal resistance increases as the distance from the heat input position to the temperature measurement point M increases, according to the Fourier’s law. That is, the analysis of the STAM image based on the Fourier’s law allows for the evaluation of the steady-state thermal conductivity of a TEM specimen.

Fig. 4a is a BF-STEM image obtained by zooming around the filler 2, which is actually the region indicated by a black dashed frame in Fig. 2a. Fig. 4b provides a STAM image of the same area (Fig. 4a). The black color in the filler 2, apparent in its center, indicates that the temperature of the measurement point M, which is far away from that filler, would preferably increase compared to other filler portions. Fig. 4c illustrates a line profile analysis of sample thickness variation from point T₀ to point T₁. A relatively small thickness change is evident. Fig. 4d provides a line profile information of the same section T₀→T₁ with respect to the STAM imaging. In the BF-STEM image (Fig. 4a) a filler C (diameter of 200 nm) and a filler D (diameter of 35 nm) are visible between fillers B and Z (shown in Fig. 2c). On the other hand, filler F is also seen between those, Fig. 4a. When the heat is applied to filler 2, there are two possible routes for the heat conduction between fillers 2 and B: 2→D→C→B or 2→F→E→C→B (as shown in Figs. 2c and 4a). Within the interval T₀→T₁, we mark points T₀ (close to the filler D) and T₁ (close to the filler F). From the line profile thickness modulation (Fig. 4c), it can be considered that the amount of input heat at the point T₀ is almost the same as that at the point T₁. Meanwhile, in the line profile of the STAM image (Fig. 4d), the maximum value is seen near point T₀, in fact, this area looks black in the STAM image (Fig. 4b). This indicates that the heat input in the filler 2 mainly passes through the filler D rather than via filler F. In other words, it can be concluded that the input heat at T₀ diffuses through a route: T₀→T₁→D→C→B→A→I (Figs. 2c and 4a). Thus, the pathways of the heat transfer of intra- and inter-fillers could also be directly visualized by STAM.

To summarize, on the basis of the Fourier’s law we measured thermal conduction in alumina fillers (and between them) embedded into thermally insulating epoxy using newly developed STAM technique and utilizing a nanothermocouple assembled in TEM. STAM images provide us with the two-dimensional information with respect to a nanoscale thermal transport under electron beam-induced heat injection into a microcomposite sample. The proposed STAM method can be applied to not only analyze complex heat flows in heat-sink composites, but also perform quantitative thermal transport measurements [20] and to reveal heat distribution in any nanostructured object. In order to discuss the relationship between the microstructure and the local thermal conductivity in a nanoscale material more quantitatively, it is important to develop an advanced STAM method which can analyze the transient thermal conduction process in addition to the quantitative thermal conductivity measurements based on the Fourier’s law [20]. We are planning to shorten the irradiation time to tens of ms by re-arranging the circuit of the current STAM and measuring the specimens consisting of electron-beam sensitive materials. The improved time resolution of the measurement circuit may be achieved using a pulsed electron beam source. Furthermore, our method has a rare possibility of the nanoscale Seebeck coefficient elucidation during nanoscale thermo-electric transitions in a given composite material.

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