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MICROSCALE TEMPERATURE MEASUREMENT BY SCANNING THERMAL MICROSCOPY

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Abstract

Scanning thermal microscopy (SThM) can measure thermal image with a nano-scale spatial resolution. However, there remains an issue in quantitative temperature measurement. We proposed an active temperature measurement method that provides a real temperature image by compensating a variation in contact thermal conductance. Performance of the active method was examined by a multi-function cantilever made with micro-fabrication process. Response test of the cantilever showed about 50 Hz cut off frequency for both passive and active method. Temperature measurement test indicated that sensitivity of heat flow detection was not enough to measure real temperature regardless of the thermal contact conductance. Imaging test demonstrated that the active method takes temperature image closer to real temperature distribution than the passive method.

Keywords: active method, scanning thermal microcopy, temperature measurement, thermal feedback

Introduction

With remarkable progress of nano-technology, microscale thermal measurements with nano-scale spatial resolution have gained importance in various fields. In the microelectronics industry, minimum feature size of electric devices and lines in current ICs have become smaller than 200 nm. Miniaturization and integration of devices and circuits leads to high-density heat dissipation and resultant high operating temperatures. Thus, thermal diagnosis is important for checking soundness and operating conditions of devices. However, conventional optical micro-thermometry such as infrared radiation thermometry [1], the micro-Raman method [2], and reflectance thermometry with focused laser [3] are no longer available for nano-scale objectives because their spatial resolution is limited by diffraction of the light used. Needs for a nano-scale thermal measurement method also arise from the fields of material sciences, chemicals and bioengineering. Nano-scale thermometry will allow direct thermal observation of a properties profile of materials with nano-structure such as ceramics and composite materials, and heterogeneous chemical reaction on catalysis, biological tissues, and cells.

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Currently, SThM exists as a novel thermometry having spatial resolution smaller than light wavelength by adding a thermal detection function to scanning probe microscopes (SPM) with atomic scale resolution. After the first SThM [4] demonstrated topographic and thermal imaging of working electric devices using a tiny thermocouple cantilever in the early ’90s, various SThM have been developed. The SThM with resistance temperature device (RTD) of a Wollaston wire allows temperature and thermal conductivity distribution measurement by using RTD as both a sensor and heater [5]. Thermal bending of a bimorph cantilever in contact with an AC biased sample was used for thermal imaging on the atomic force microscope (AFM) [6]. Also, high vertical spatial resolution of the AFM enables detection of slight thermal expansion of a biased sample. It was proposed as a scanning Joule expansion microscope [7]. Moreover, micro-reflectance thermometry based on the scanning near field optical microscope (SNOM) was reported [8]. The thermocouple method on the AFM seems like a promising one among these various microscale thermometries because of its wide applicability, smallness of junction size, non-exothermic feature, and its capabilities for quantitative temperature measurement as described later. Therefore, we will discuss the thermocouple type of SThM.

Up to now, we have shown that SThM can detect thermal information of a sample surface by the cantilever through a small contact region of about 10 nm scale under vacuum conditions, and measure temperature and thermal properties distribution at the sub-micron scale [9, 10]. The key to improving SThM is to fabricate a small temperature sensor near the contacting tip. A thermocouple with sub-micron scale junction on the tip allows thermal measurement with spatial resolution of less than 30 nm and thermal response of more than 1 kHz [11, 12]. Research on SThM has aimed for nano-scale spatial resolution; so there is the prospect that thermal measurement with spatial resolution of 10 nm will be achieved by making a small thermal cantilever with the micro-fabrication technique.

In contrast to this success for spatial resolution, there remains an important issue in quantitative temperature measurement. It is well known that many artifacts related to topological conditions are included in SThM thermal images. They occur because the SThM does not measure sample temperature, but the heat transfer rate between the cantilever and the sample. Because the heat transfer rate is proportional not only to temperature difference but also to a contact heat conductance, the measured value depends on such various factors as properties, surface shape, contact force, adsorption layer, and so on. It is very difficult to derive real temperature and thermal properties from measured values by an ordinary calibration process.

Therefore, we have proposed and studied a new active method to realize quantitative temperature measurement on the SThM [13]. Target performance of the active method is set as temperature accuracy of 1 K as well as spatial resolution of less than 100 nm and response time of 10 ms. In this paper, we report the principle of the active method for real temperature measurement with sub-micron resolution, a thermal feedback system, a design and fabrication of a multi-function thermal cantilever to realize the method, and test results on the prototype system.

Active temperature measurement method and experimental setup

Principle of the active method

Figure 1 shows the principle of the active method for real temperature measurement on SThM. The method uses a multi-functional cantilever having a differential thermocouple, an electric heater, and another thermocouple. A steady state is supposed to simplify discussion. In the conventional passive method, sensor temperature near the cantilever tip is decided by balance of thermal resistance among the sample and cantilever system. On the other hand, cantilever temperature is actively varied in the active method by detecting heat flow along the cantilever and feeding power proportional to it to the cantilever. Feedback control with sufficient gain keeps the cantilever the same temperature as the sample contact point, then cantilever temperature is measured by another thermocouple on the middle of the cantilever. From a methodological point of view, the passive one is put into the category of a 'deflection method', where output of a sensor affected by a sample is directly measured; whereas the active method uses the concept of the 'null method' in which sensor potential is measured under the zero-flux condition. In principle, the active method can measure real temperature as long as contact thermal conductance has finite value, no matter how it varies.

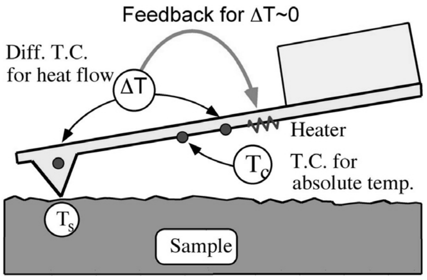

Fig. 1 Principle of the active temperature measurement method on SThM

Cantilever design and fabrication

To realize the active method, it is necessary to fabricate the cantilever with three functions and thermal feedback circuit. We had already performed preliminary experiments of the method with a hand-made multi-functional cantilever of about 1 mm length, confirming that the method yields quantitative temperature for high thermal conductive materials [14]. Since large feedback gain often leads to system divergence, the prototype system could not provide real temperature for low conductive samples. Thus, we had also conducted a performance simulation of the feedback method with a simple discrete thermal model and designed a cantilever which would allow real temperature measurement within 1 K error even in a low thermal contact conductance case. This time, a micro-cantilever equipped with required functions was made according to design by means of micro-fabrication technology for practical use of the active method.

Figure 2 shows an optical microscopic image of the multi-functional thermal cantilever in the fabrication stage before depositing the gold film on its reverse. Nickel and chromium metal lines comprise the differential thermocouple, electric heater, and another thermocouple in about 150 μm region on the SiO_2 cantilever of 260 μm length. First and third junctions lined from the end are used as the differential thermocouple and the second one is used for detecting cantilever temperature. Although heating and cooling functions are required for complete linear feedback control, Joule heating and cooling by conduction through the cantilever body are used for simplicity. Moreover, a cantilever for the AFM usually has a sharp stylus tip to make nanometer scale interaction with sample surface. However, our cantilever is a type non-stylus type because of difficulty in three dimensional fabrication process; its end directly contacts the sample.

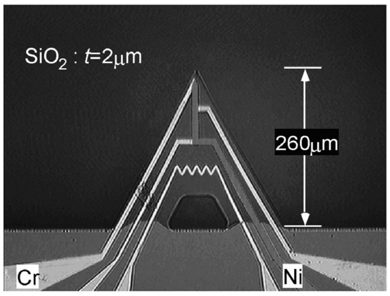

Fig. 2 Multi-function micro thermal cantilever

The cantilever was made with an ordinary micro-fabrication process as shown in Fig. 3. The fabrication sequence begins with a 200 μm thick silicon (100) wafer coated with 2 μm thick thermally oxidized layer. The wafer is diced to 15×15 mm chips. The SiO_2 layer is patterned and etched with buffered hydrofluoric acid (BHF) at room temperature to form a triangular cantilever shape. Lines of 1.0 μm thick Cr and 0.5 μm thick Ni are fabricated on the SiO_2 layer by sputter deposition and the lift-off method. The cantilever is then coated with a 1.0 μm thick SiO_2 film by sputter deposition, which protects metal lines following Si etching and prevents an electrical short circuit during measurement of a biased sample. After the Si substrate is ground to 50 μm thickness from the backside with a dicing saw, here is an extraordinary process, residual Si substrate is etched away with TMAH solution at 80°C over about half an hour. Finally, a 1.3 μm thick gold film is deposited on the cantilever backside. It acts as a laser reflector in AFM operation and also compensates thermal stress for prevention of a cantilever thermal bending.

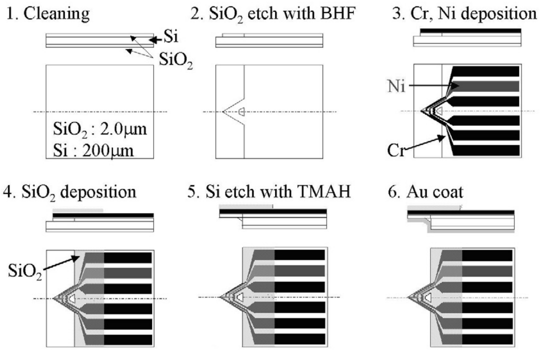

Fig. 3 Cantilever fabrication process

Feedback system

A self-made electric feedback circuit shown in Fig. 4 was used to drive the multi-functional thermal cantilever. In the circuit, a signal of the differential thermocouple is amplified 10^5 times with an instrumentation amplifier (AD624); then passes a square root circuit and a power operational amplifier; then finally power is applied to the heater on the cantilever. The square root circuit is necessary for providing Joule heating proportional to heat flow along the cantilever, in other words for linear feedback. Cantilever temperature is kept equal to that at the contact point on the sample by the feedback system, then temperature of cantilever is measured by another thermocouple. Furthermore, cantilever-base-holder temperature is measured by a standard type T thermocouple because the reference junction of the thermocouple on the cantilever is located on the cantilever base.

Presently, this system does not include a cooling device, so it can measure higher temperature than ambient. It is possible to extend the measurement range to cold samples by applying low temperature bias on the cantilever base with a Peltier cooling element. Actually, the zero point of the active method is shifted to some extent by laser irradiation

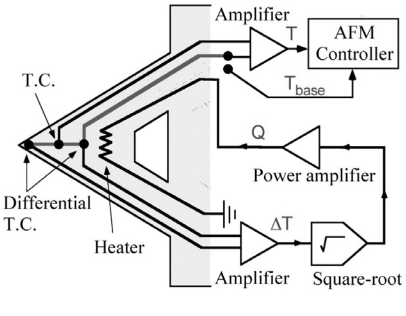

Fig. 4 Block diagram of thermal feedback system

for detecting bending of the cantilever in the AFM. Practically, the active method on AFM will measure over a temperature range higher than ambient by setting base temperature lower than ambient by a few degrees with the Peltier element.

Experiment procedure

Our SThM is constructed by attaching our self-made cantilever to a commercial AFM system (SPA-300 & SPA3700, SII). Under atmospheric pressure conditions, heat conduction through ambient gas plays a dominant role in heat transfer between the cantilever and sample surface. Thus, thermal measurement experiment was conducted under 0.1 Pa order pressure condition by putting AFM into a vacuum chamber to detect thermal information through the small contact region.

We examined this active temperature measurement system from the three standpoints of response speed, sensitivity, and imaging performance. For the response test, thermal response of each junction on the cantilever was measured by a lock-in amplifier for periodic laser heating near the end. In sensitivity examination, some plate samples with different thermal conductivity were prepared. Each sample was set on an insulated plate heater mounted on the piezo-scanner and temperature was measured by the active method while varying sample temperature. For the imaging test, we tried to capture topographic and temperature images of dc biased chromium lines on the Si substrate with the dioxide layer.

Testing and performance of the active method

Response time

Thermal response in both passive and active methods was examined by irradiating a periodic laser near the end of cantilever and detecting thermocouple signals at the heating frequency by a lock-in amplifier. Figure 5 shows thermocouple signal amplitude at the junction near the end and the differential thermocouple signal as a function of heating frequency. For the passive method, frequency characteristics are usually determined by cantilever thermal resistance and thermal capacitance and can be considered one of a first-order system. Response of the end thermocouple without feedback shows a cut off frequency of about 50 Hz, where the signal level is attenuated by 3 dB. This value is similar to what is expected in the previous simulation. The differential thermocouple shows a cut off frequency of about 100 Hz and amplitude of 1/100 of the end thermocouple.

In the active mode case, the differential thermocouple signal diminishes in amplitude and the end thermocouple signal increases. This is because feedback control provides heat proportional to the differential thermocouple signal to the cantilever so as to cancel it out. The active method has the potential to improve thermal response by detecting a rising edge of the differential thermocouple signal, then raising cantilever temperature by electric means. The result, however, shows a similar cut off frequency to the passive method. It can be considered that the active method uses heat-

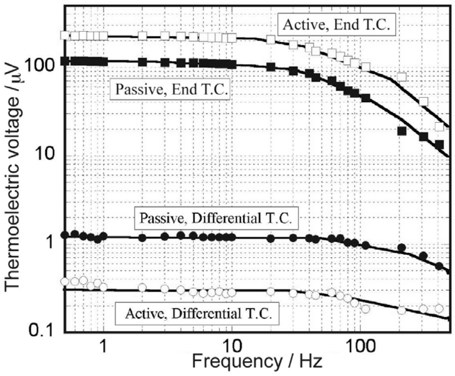

Fig. 5 Thermal response of the multi-function cantilever for periodic laser heating

ing by electricity and cooling by heat conduction through the cantilever, and the slow conduction rate governs system response.

Active method sensitivity

If the active method works properly, real temperature should be measured regardless of contact conditions including sample thermal conductivity, contact force, and so on. In practice, however, there remains some error according to contact thermal conductance in the measurand because cantilever temperature does not perfectly coincide with the sample by feedback control where the finite heat flow rate is detected and finite heat is fed back. We set our error target as less than 1% of real temperature. Figure 5 shows the result of the active temperature measurement test for such samples as gold, chromium, glass, and epoxy resin. Temperature was actively measured as a function of sample temperature by contacting, but not scanning, the cantilever on the sample on SThM under the vacuum condition. Here, thermoelectric power data of corresponding bulk metals was used for converting the thermocouple signal into the measured temperature. The result shows that the measured value varies with conductivity and contact condition, and is more than 30% lower than real temperature at most. Unfortunately, sensitivity of this present system was insufficient to measure real temperature.

Three measures can be suggested for sensitivity improvement: increase of feedback loop gain, reduction of junction size, and use of a thermopile as the heat flow sensor. Increase of gain often leads to feedback system instability, so it does not seem like a promising measure. Making a smaller thermocouple junction at a closer point to the end is most important for detecting a small heat flow rate. Since it is a micro-fabrication technique issue, we cannot put hopes in a difficult fabrication procedure. Practically, we can consider that it is a promising measure to introduce a thermopile structure as the heat flow sensor. There is space on our cantilever for placing a thermopile composed of several differential thermocouples. We plan to increase sensitivity by making a modified cantilever with a thermopile.

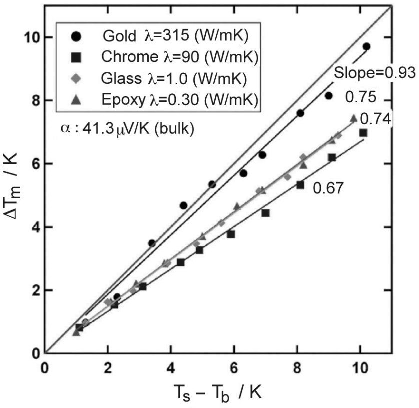

Fig. 6 Active method performance test results

Thermal imaging

Although performance of the active method was not sufficient to measure real temperature under low thermal contact conductance conditions, visualization characteristics of both the passive and active method were also examined for the self-made metal strip sample. Three chromium strips of 25 nm thickness, 5 μm width, and 10 μm pitch were fabricated on the Si substrate with 2 μm oxide layer thickness. During the imaging test, the parallel strips were heated up with DC bias in about 26°C surroundings; it measured about 44°C from electric resistance. Here, thermal images by the passive and active method are called heat flow images and temperature images, respectively, because the passive method measures signal proportional to heat flow along the cantilever.

Topographic and thermal images in Fig. 7 were taken by the passive method with the multi-functional thermal cantilever at a scan rate of 1 Hz. Despite the needle-less cantilever, it can be seen that the strips were measured well in shape. As long as topography is observed at the micrometer scale, there seems to be no big problem or flaw associated with the self-made cantilever. Only mechanical vibrations from a vacuum pump connected with the vacuum chamber resulted in fine stripe noise in the whole topographic image. Regarding the heat flow image, the qualitative temperature profile was measured very clearly. However, impression of the strips in high temperature may come from both actual higher temperature and higher thermal conductivity than the oxide layer on the Si substrate. Maximum measured temperature based on bulk thermoelectric power was 28.5°C and is far from the real strip temperature. It is very difficult to convert the measured value to surface temperature via the ordinary calibration process because the measured value is influenced by various factors such as properties, shape of surface, adsorption layer, contact force, and so on.

Figure 8 images were taken with the active method under the same conditions. First of all, maximum measured temperature is about 38°C and is closer to real temperature of the strips than that of the passive method. We can confirm effectiveness of the active method with real temperature measurement in a microscale.

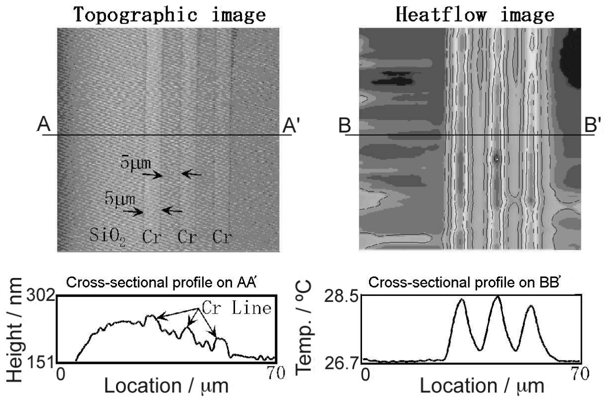

Fig. 7 Thermal imaging of a narrow metal strip by the passive method

However, some problems appear in the images. Strips should measure higher than the substrate, but they measured lower in topography. This is due to thermal bending effects of the cantilever. Because the whole cantilever of a multi-layer structure is heated up to the temperature at the contact point on a sample, thermal bending of the cantilever tends to affect topography in the active method. It is inferred that downward thermal bending made such an artifact in topography that strips in the high temperature state measured lower than the substrate surface. There is room for reducing thermal bending by adjusting gold film thickness on the cantilever back to compensate for imbalance in thermal stress among layers in the cantilever.

Regarding the temperature image, equally heated strips should have symmetric temperature distribution; however the asymmetry profile was measured. The reason for the distortion can be heating of the sample by the cantilever. Since the needle-less cantilever locates very close to the sample and the whole cantilever is heated up to the

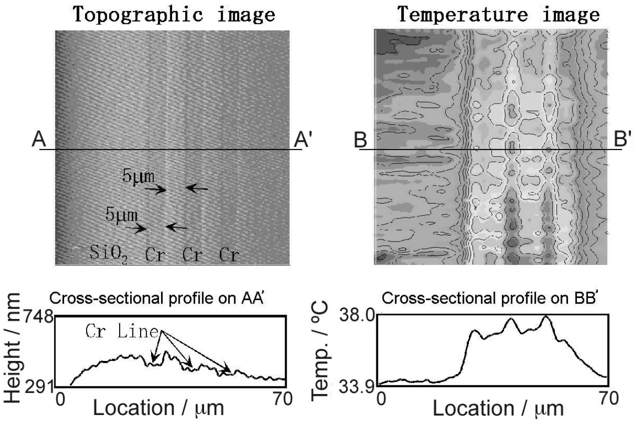

Fig. 8 Thermal imaging of a narrow metal strip by the active method

temperature of the contact point, heat transfer can take place via residual gas from the cantilever to underneath the sample at the moment when the cantilever contacts the hot region. The temperature image was taken by scanning the cantilever from left to right with its end pointing leftward in the image, so the right-hand region of the strip could be heated and was measured as hotter than the original temperature.

For the same reason, it can be inferred that temperature of the region between the strips measured higher than original temperature. Since the active method may induce a temperature raised by itself, it is considered to capture blurred distribution. These issues are caused by the needle-less shape of the cantilever. We expect that a new cantilever with a needle would relieve the issues.

In addition, the temperature image is much noisier than the heat flow image. The cause is unknown, but some factors can be imagined; variation of contact conductance induced by mechanical vibration of vacuum pump, electric noise received from ambient and generated in feedback circuit and so on. Essentially, if active measurement works properly, variation of contact conductance could be compensated by the feedback loop. Now, it can be thought that low sensitivity of heat flow detection and large feedback loop gain make the system unstable. Because reduction of electric noise is not easy, we expect that improvement of sensitivity by the thermopile heat flow sensor solves noise problems, too.

Concluding remarks

To realize the active temperature measurement method that we proposed previously, the multi-functional thermal cantilever was made by the micro-fabrication technique. The cantilever is made of silicon dioxide and has a differential thermocouple, an electric heater, and another thermocouple for detecting heat flow, generating heat proportional to it, and then measuring its own temperature. The cantilever showed about 50 Hz response against laser heating in both the passive and active method. It turns out that test system performance was insufficient to measure real temperature because heat-flow detection sensitivity by the differential thermocouple is insufficient. The system could take a temperature image closer to the real temperature profile than the passive method.

In the future, we will improve the cantilever to solve problems arising from this experimental study, such as thermal bending of cantilever, sample heating by the cantilever, and low sensitivity of heat flow detection.

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