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Fabrication of scanning thermal microscope probe with ultra-thin oxide tip and demonstration of its enhanced performance

Heebum Chae, Gwangseok Hwang, Ohmyong Kwon *

Korea University, School of Mechanical Engineering, Seoul 136-701, South Korea

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ABSTRACT

With the vigorous development of new nanodevices and nanomaterials, improvements in the quantitation and resolution of the measurement of nanoscale energy transport/conversion phenomena have become increasingly important. Although several new advanced methods for scanning thermal microscopy (SThM) have been developed to meet these needs, such methods require a drastic enhancement of SThM probe performance. In this study, by taking advantage of the characteristics of micromechanical structures where their mechanical stability is maintained even when the film that composes the structures becomes extremely thin, we develop a new design of SThM probe whose tip is made of ultra-thin SiO_2 film ($\sim$100 nm), fabricate the SThM probes, and demonstrate experimentally that the tip radius, thermal time constant, and thermal sensitivity of the probe are all improved. We expect the development of new high-performance SThM probes, along with the advanced measurement methods, to allow the measurement of temperature and thermal properties with higher spatial resolution and quantitative accuracy, ultimately making essential contributions to diverse areas of science and engineering related to the nanoscale energy transport/conversion phenomena.

1. Introduction

Scanning thermal microscope (SThM) is a type of scanning probe microscope (SPM) that maps the temperature and/or thermal property using a sharp SThM probe that has a temperature sensor at the tip [1–3]. SThM has been applied in such diverse areas as the analysis of the energy transfer/conversion in nanoelectronic, nanophotonic, and nanothermoelectric materials, as well as for devices such as carbon nanotube, nanowire, graphene, molybdenum disulfide, and nanotransistor, LEDs [4–11]. In recent years, with the vigorous development of new nanodevices and nanomaterials, improvements in the quantitation and resolution of the measurement of nanoscale energy transfer/conversion have become increasingly important [12]. To meet these needs, a double-scan technique, which can prevent distortion caused by heat transfer through the air, and ultra-high vacuum scanning thermal microscopy have been developed [13,14]. Furthermore, null-point scanning thermal microscope (NP SThM) has also been developed, which can solve the three major problems of conventional SThM: (i) distortion of the measured signal caused by heat transfer through the air, (ii) the unknown and variable value of the tip-sample thermal contact resistance, and (iii) perturbation of the sample temperature caused by heat flux through the tip-sample thermal contact [15].

However, these newly developed advanced SThM techniques require a drastic enhancement of SThM probe performance because they measure local temperatures/thermal properties from the extremely small heat flow between the apex of the tip of the SThM probe and the sample surface. Although a few SThM probes have been batch-fabricated before, perhaps the first SThM probes designed and batch-fabricated through a systematic optimization of the thermal design are those by Shi et al. [16]. The tip of the probe is fabricated by isotropic wet etching of 8 μm thick, low-temperature oxide film, and the probe's cantilever is made of low-stress silicon nitride. Then, in order to enhance thermal sensitivity, Kim et al. fabricated the entire body of the SThM probes (both cantilever and tip) from SiO_2 and increased the tip's height up to 12 μm [17]. They enabled the double-scan technique with full SiO_2 probe. Later, Chung et al. attempted to enable NP SThM with the same type of probe fabricated by Kim et al. but, because the probe's thermal sensitivity was not sufficiently high, the temperature profile obtained by NP SThM was quite noisy [15]. Recently, Hwang et al. optimized the design parameters, such as tip height, tip half angle, cantilever length, position of the thermocouple junction, and thickness of the metal films, that constitute the thermocouple through a rigorous theory of quantitative measurement, and successfully enabled low-noise NP SThM [18].

Hwang et al. explained that the intrinsic performance

* Corresponding author.
E-mail address: omkwon@korea.ac.kr (O. Kwon).

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parameters of the SThM probe are thermal sensitivity (S_{probe}), noise of the temperature sensor (ΔT_n), and tip radius (R_{tip}) [14]. Because the spatial resolution of the SThM probe depends not only on the intrinsic parameters determined by the design and fabrication process of the SThM probe (S_{probe}, ΔT_n, R_{tip}), but also on the extrinsic variables irrelevant to the performance of the probe, such as the temperature gradient of the sample and interfacial heat transfer coefficient between the tip's apex and sample surface, it is not appropriate to express the performance of the SThM probe simply by its spatial resolution. Although Hwang et al. did not consider the thermal time constant of SThM probe, this constant is another important factor in the performance of SThM probe.

In this study, we develop a new design and batch-fabrication process for the SThM probe in order to improve its thermal sensitivity, tip radius, and thermal time constant simultaneously. Such development is performed by exploiting the characteristics of micromechanical structures where their mechanical stability is still maintained even when the film that composes the structures becomes extremely thin. We also demonstrate experimentally the improved performance parameters of the newly developed SThM probe.

2. Thermal design of SThM probe

Although Hwang et al. did not consider the SThM probe's thermal response speed, improving the thermal response speed of the SThM probe is important. If we improve the thermal response speed of the SThM probe by reducing its thermal capacitance, we can increase the probe's scan speed when profiling spatially varying temperature fields. Furthermore, an improved response speed would allow tracking time-varying temperatures at particular locations more accurately, thus extending the applicability of scanning thermal wave microscopy (STWM).

STWM is an AC measurement mode of SThM that allows subsurface imaging of micro or microelectronic structures, and measures the thermal conductivity of nanowires or nanofilms by measuring the amplitude and phase lag of the thermal diffusion wave that passes through them. Kwon et al. demonstrated experimentally that decreasing the thermal penetration depth of the thermal diffusion wave by increasing the frequency of the thermal wave could reduce the distortion of the measured signal caused by heat diffusion through the air [19]. In other words, imaging the high-frequency thermal diffusion wave improves the accuracy of STWM. Therefore, in order to extend the applicability of STWM, the thermal response speed of the SThM probe should be further improved.

In general, the response speed of thermal sensors, such as the SThM probe, is represented by thermal time constant τ_{th}, which is proportional to the product of its thermal resistance R_p and thermal capacitance C_{th}, or

$$\tau_{th} = R_p C_{th}. \tag{1}$$

The thermal resistance of SThM probe R_{p} is defined as the temperature rise at the apex of the tip of the SThM probe, where the temperature sensor is located, by the heat that flows through the tip-sample thermal contact Q_{st}, or

$$R_{\mathrm{p}} = \frac{(T_{\mathrm{c}} - T_{\mathrm{nc}})}{Q_{\mathrm{st}}}, \tag{2}$$

where T_c is the temperature measured by the temperature sensor integrated at the end of the probe tip in contact mode, and T_{nc} is the temperature measured by the sensor in the non-thermal contact mode. The non-thermal contact mode is a scanning mode where Q_{st} is zero and Q_{air}, the heat flux through the air gap, is the same as that in the contact mode. Experimentally, T_{nc} can be obtained by linearly extrapolating the two temperature data measured at two different heights above the sample surface with respect to height [13]. T_c is induced by both Q_{st} and Q_{air}, and T_{nc} is generated only by Q_{air}. Therefore, the difference between T_c and T_{nc} corresponds to Q_{st} only. Hwang et al. defined R_{p} as the thermal sensitivity of the SThM probe (S_{probe}) [18].

The recently fabricated SThM probes have greatly improved R_{p} [18]. However, as Eq. (1) indicates, the increase of R_{p} could rather decrease the probe's response speed. If R_{p} were to decrease, the probe's response speed might be improved. However, this is not acceptable because the probe's thermal sensitivity might deteriorate. Hence, in order to improve the response speed of the SThM probe while maintaining the thermal sensitivity high, C_{th} should be minimized.

In a previous study, based on a rigorous theory of quantitative measurement, Hwang et al. optimized the tip's half angle, cantilever length, and thickness of the metal films that compose the thermocouple junction in order to optimize thermal sensitivity, temperature measurement noise of the thermocouple junction, and tip radius [14]. Fig. 1(a) shows the structure of the SThM probe fabricated by Hwang et al. NP SThM01 (TSP Nanoscopy).

In this study, in order to simultaneously reduce the thermal time constant, minimize the tip radius, and maximize the thermal sensitivity of the SThM probe, we modify the design of the SThM probe optimized by Hwang et al. [18], shown in Fig. 1(a), such that the SiO_2 film that composes the probe tip becomes extremely thin, as shown in Fig. 1(b). Such modification is performed by taking advantage of the characteristics of micromechanical structures, where their mechanical stability is maintained even when the film that composes the structures becomes extremely thin.

When the SiO_2 film that composes the probe tip becomes extremely thin ($\sim$100 nm), the heat transfer through the oxide tip is reduced significantly, the thermal insulation of the sensor located at the tip's apex is improved, and the thermal sensitivity of the probe is improved. Furthermore, because the thermal mass of the tip is also reduced drastically, the thermal time constant of the probe is decreased significantly. In addition, because the thin SiO_2 tip is formed by thermally oxidizing a very sharp silicon tip at high temperature ($>$1100 °C) for a much shorter time than is required to produce a thick SiO_2 tip ($\sim$1.5 μm), the sharpness of the silicon tip is maintained, even after thermal oxidation. Hence, the radius of the thin SiO_2 tip is reduced further than that fabricated by Hwang et al.

3. Fabrication and characterization of SThM probe

The fabrication process for the newly designed SThM probe with ultra-thin oxide tip (UTOT01) is developed by modifying the tip formation step of the batch-fabrication process developed by Hwang et al. [18], and it is schematically explained in Fig. 2(a) to (f).

First, a low-stress silicon nitride film is deposited by low-pressure chemical vapor deposition (LPCVD) and patterned into a hard mask for anisotropic wet etching of the silicon tip. The silicon wafer is anisotropically etched in a high-concentration KOH solution until a sharp tip is formed (Fig. 2(a)). Second, a 1.5 μm-thick SiO_2 that composes the cantilever and tip of the probe is grown by wet thermal oxidation (Fig. 2(b)). Third, the SiO_2 the tip is locally wet-etched, and a thin SiO_2 (100 nm) is grown at 1100 °C such that the sharpness of the silicon tip persists even after the oxidation process (Fig. 2(c)) [20]. Fourth, a gold film, which is the first metal layer that composes the nanothermocouple junction, is deposited by electron-beam evaporation and patterned (Fig. 2(d)). Fifth, for the electrical insulation between the two metal films that