Batch Fabricated Dual Cantilever Resistive Probe for Scanning Thermal Microscopy

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Abstract

In this study dual cantilever resistive probes for scanning thermal microscopy (SThM) have been batch fabricated. In the dual probe, one is used as local heater and a second one nearby detects the thermal diffusivity at a microscopic scale. Various types of dual probes have been fabricated in one batch to allow experimental determination of the optimal sensor type for the measurement. Thermal scans with the dual cantilever probes have been performed in atmosphere, and contrast in thermal imaging indicating the difference of thermal conductivity is shown. Test of these probes under vacuum indicates strong thermal coupling through air between the two probes in the dual cantilever probes.

Keywords: Microfabrication, dual cantilever, scanning thermal microscopy (SThM)

1. Introduction

With the remarkable recent progress in materials science and nanotechnology, there has been growing interest in thermal property measurement at submicrometer scale. Scanning thermal microscopy (SThM) [1-3] was developed to probe thermal properties with high spatial resolution, in which a sharp temperature-sensing tip is brought in close proximity to a sample solid surface. By scanning a heated tip across the sample surface a spatial map of the thermal properties of the sample may be mapped out as localized heat transfer between the tip and sample surface changes the tip temperature. Many theoretical and instrumental studies have been devoted to the measurement of temperature and conductivity [4-13]. However, the results obtained depend strongly on the nature of the tip-surface contact, which is poorly defined [3, 10]. The use of a heater fabricated on the surface may be used to obtain the information on local lateral thermal conductivity with SThM, but this requires the sample to be modified. In order to permit this type of measurement without the need to fabricate a device structure on as-grown materials a dual cantilever SThM probe is proposed. One of the probes is used as local heater and a second one nearby detects the resultant temperature rise at the microscopic scale. In this study, various types of dual silicon nitride cantilever resistive probe have been fabricated in order to allow experimental determination of the optimal sensor type for the measurement. The probes have been tested and thermal scanning is demonstrated.

2. Batch fabrication of dual cantilever resistive probes

The batch fabrication process for dual cantilever resistive probes was developed based on that used for the fabrication of single cantilever resistive SThM probes [14]. The fabrication process is outlined in Figure 1. The fabrication starts with a 3 inch <100> oriented 380 μm thick n-type silicon wafer with 40 nm thick thermal silicon oxide film and 60 nm low pressure chemical vapor deposited (LPCVD) silicon nitride film on top, as shown in Figure 1 (a). Firstly, the back side of wafer was bulk micromachined to form a silicon membrane, as shown in Figure 1 (b). This process defines the body of the SThM probe die. In this step the oxide and nitride mask layer was patterned by photolithography and subsequent dry etch in C_2F_6. Then the wafer was anisotropically etched in 7M KOH solution at 105˚C for 70 minutes which gave an etch depth of 300 μm.

Next, the top side of the wafer was also subjected to bulk micromachining, as shown in Figure 1 (c). This level of processing defines the surface relief of the SThM tips and also topographic alignment marks for subsequent electron-beam lithography (EBL). Photolithography was performed with photolithographic front-to-back alignment to the previously defined membrane level. Again dry etch was used to pattern the oxide and nitride mask layer. Anisotropic etching of silicon was performed in KOH-IPA (consisting of 4 parts of 7 M KOH and 1 part of IPA) solution at 55˚C for 60 minutes for better surface finishing which is important as the silicon nitride cantilever will later be formed on top of this (100) etched surface. At this step, the alignment markers for EBL, consisting of square {111} terminated etch pits on (100) mesas, were formed. At the same time long bars with {111} slope walls, which are shown in Figure 2 (a), were formed. This differs from the process used to define single SThM probes [14]. For a single probe, the exact position of the tip relative to the cantilever is not critical. Accordingly the tip may be defined on top of a curved surface (“pyramid”) formed by a number of fast etching {311} planes. A silicon nitride film will later be deposited on top. This has the advantage that the Si_3N_4 used to form the tip is deposited onto a curved surface giving shape stiffness. The film on one pyramid wall will be fabricated into a single probe tip, as shown in Figure 3 (a). However, in the case of dual cantilever probes the pyramid walls are no longer large enough for fabrication of two tips. In addition, the roughness of the pyramid is such as to make it impossible to fabricate two tips with well-defined separation and height. Therefore, in the present study long silicon bars with wide, flat {111} etch stop planes were designed and fabricated. Figure 3 (b) schematically shows that two probe tips are formed on one side wall of the long bar, and Figure 2 (b) shows the real case. The long bars make it possible to fabricate dual cantilever probes with various tip-to-tip spacing so as to allow the realization of a range of cantilever designs. Next, the remaining oxide and nitride mask on both sides of wafer was stripped away by means of a 4:1 mixture of water : 48% unbuffered HF solution, as shown in Figure 1 (d). Then 400 nm thick of low stress LPCVD silicon nitride film was deposited on both sides of wafer, as shown in Figure 1 (e). In the following step (shown in Figure 1 (f)) the film was fabricated into dual cantilevers with sharp tips. The fabrication was performed by lithography and subsequent pattern-transferred by dry etch. The very sharp tips of the probes were defined by EBL and transferred by lift-off into a metal mask consisting of 75nm of NiCr. The remainders of the cantilevers which require only low resolution were then defined by photolithography, and photo resist mask was formed. Both masks were transferred simultaneously into the silicon nitride using reactive ion etching in CHF_3 and O_2. Figure 2 (c) shows an SEM image of the dual cantilevers, in which the arrows indicate where the alignment errors occurred between the two steps of lithography. These errors have no effect on the function of the device. Before stripping the dry etch mask, the silicon nitride film at the back side of wafer was etched away by reactive ion etch in gas of SF_6, as shown in Figure 1 (g). Next a miniaturized temperature sensing resistor consisting of a 40nm thick Pd film was fabricated by EBL and lift-off, as shown in Figure 1 (h). Afterwards EBL and lift-off were employed to fabricate large pads with 10 nm thick NiCr and 145 nm thick Au, as shown in Figure 1 (i). Finally, as shown in Figure 1 (j), the probes were released by removing the silicon membrane in an anisotropic etch solution consisting of 4 parts of 7M KOH and one part of IPA at 80°C. Figure 2 (d) shows a SEM image of a dual cantilever resistive probe fabricated through the batch fabrication process.

Dual cantilever resistive probes with various tip-to-tip spacing, from 300 nm up to 10 μm, were fabricated. Figure 4 (a) and (b) show the SEM images of the dual cantilever resistive probe with tip-to-tip spacing of 300 nm and 2 μm, respectively.

3. Thermal scanning with a dual cantilever resistive probe

A Digital Instruments Dimension 3000 AFM system was employed to test the dual cantilever resistive probes. The experimental set-up for thermal scan with dual cantilever probes is schematically shown in Figure 5. One of the dual probes used as a heater was directly excited by the oscillator of a lock-in amplifier. The other probe used as a thermal sensor formed one arm of a Wheatstone bridge. The bridge was transformer coupled in order to isolate the sensor probe from electrical interference from the heater probe [15]. The bridge output was amplified, demodulated and passed to the lock-in amplifier where the second harmonic signal was extracted: A current in heater probe at angular frequency ω heats the sample at 2ω and produces the temperature oscillation at 2ω, resulting in the resistance oscillation in the second probe, the thermal sensor, at 2ω. Therefore, the second harmonic signal of bridge output extracted by the lock-in amplifier is taken as the thermal signal. The thermal signals were applied to the AFM scanning system via a Digital Instruments Signal Access Module III (SAMIII). The AFM was in contact mode and constant probe-sample interactive force was maintained during scanning. With this setup, topographic and thermal scanning images were obtained simultaneously.

A dual probe with tip-to-tip spacing of 300nm was scanned on a glass substrate with 50 nm thick gold lines on top. Figure 6 (a) and (b) show topographic and thermal images of the sample, respectively. The topographic scan shows clear contrast without double imaging which may happen for double tips using single cantilever, indicating that the laser beam used to detect the cantilever deflection can be fully aligned on one of the dual probe although the two probes are very close. Note that the two cantilevers are substantially elastically uncoupled since the cantilevers are divided all the way down to the silicon substrate. Figure 6 (b) shows contrast between gold lines and glass substrate, which is expected due to their very different thermal conductivities. However, the contrast is not strong although the thermal conductivity of gold is much larger than that of glass. Dual cantilever probes with spacing of 2 μm and 10 μm have also been scanned, and led to even weaker thermal contrast. The weak thermal contrast was suspected to be dominated by direct thermal conduction between the probes through the air.

4. Dual cantilever resistive probes in vacuum

In order to examine the role of air the dual cantilever probes were tested under vacuum. A similar experimental setup as that shown in Figure 5 was employed with a dual cantilever probe mounted in a vacuum chamber but without either coupling transformers or AFM scanning system. No sample was present. The heater probe was directly excited by the oscillator of a lock-in amplifier. After amplification the second harmonic signal of the bridge output from the sensor probe was extracted by the lock-in amplifier. The second harmonic signal was measured as a function of chamber pressure. Taking the measured signal in the atmosphere as a level of 100% for the corresponding probe, Figure 7 shows the signal level as a function of pressure for dual cantilever probes with tip-to-tip spacing of 2 μm and 10 μm. The signal level decreases as the pressure decreases from 1000 mbar to 1 mbar, and the signal is effectively zero as the chamber pressure is reduced below 1 mbar. The results indicate that there is strong thermal coupling through air between the two probes in the dual cantilever probes and the coupling disappears as the chamber pressure is lower than a certain level. At pressure of 1 mbar the mean free path of air is estimated to be about 10^{-4} m, much longer than spacing of 2 μm and 10 μm so that the results appear reasonable. The coupling may be dominant in air so that the effect of conduction through the sample will appear to be minor, resulting in the weak contrast in Figure 6 (b).

Several experimental studies [16, 17] have indicated that the gas conduction plays an important role for heat transfer from a microcantilever heater. The present results have again confirmed it. A recent thermal scan by SThM in vacuum with single cantilever probe has achieved high resolution due to the elimination of both air conduction and conduction through the water meniscus [18]. We therefore believe that the sensitivity can be greatly improved if thermal scan with dual cantilever probes is performed in vacuum. Currently our experimental setup for thermal scan in vacuum chamber is being constructed.

5. Conclusions

Various types of dual cantilever resistive probes for SThM have been fabricated. The batch fabrication process is based on that used to fabricate single cantilever resistive probes. The main difference is the formation of long bars with {111} walls which provide enough space to accommodate two tips with various tip-to-tip spacing down to 300nm. The fabricated probes have been scanned successfully in atmosphere, and thermal contrast indicating the difference of thermal conductivity is shown although the contrast is weak. Test of these probes in vacuum suggests strong thermal coupling through air between the two probes in dual cantilever probes, resulting in weak contrast in thermal imaging in atmosphere.

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Figure captions:

Figure 1 Schematic diagrams of whole batch fabrication process for dual cantilever resistive probes, in which a cross section of a silicon wafer is shown. (a) Silicon wafer with silicon oxide and silicon nitride film on both sides; (b) Back side micromachining to form membrane; (c) Front side micromachining to form long bars with {111} walls; (d) The silicon oxide and silicon nitride mask stripping; (e) 400 nm thick of low stress LPCVD silicon nitride film deposition; (f) Cantilevers defining; (g) Back side silicon nitride film stripping; (h) Pd thin film resistors fabrication; (i) Au connector pads formation; (j) Probes releasing.

Figure 2 SEM images of (a) a long silicon bar with {111} walls; (b) dual tips defined on a {111} wall; (c) dual cantilevers defined; and (d) a dual cantilever resistive probe fabricated through the batch fabrication process. The arrows in (c) show where the alignment errors between the two steps of lithography occur.

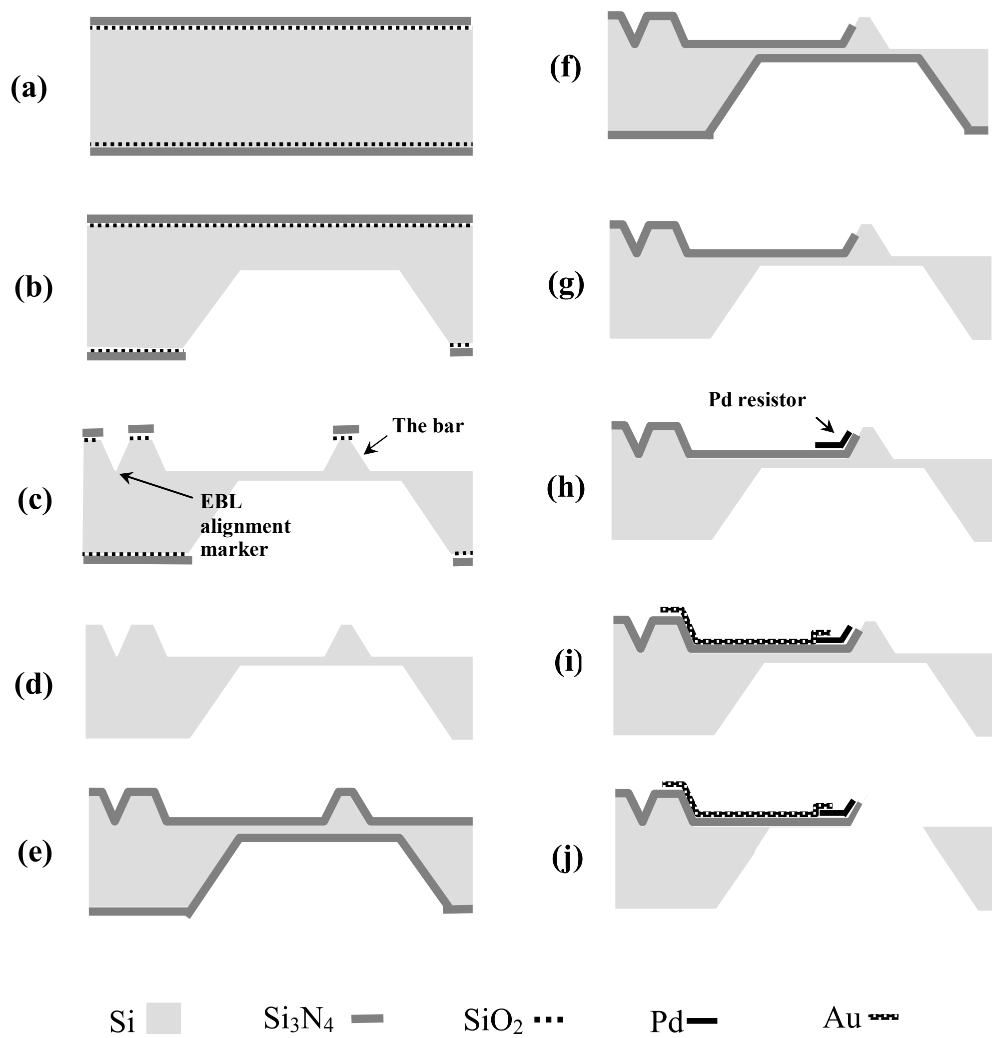
Figure 3 Schematic diagrams shown the fabrication stage for a single cantilever (a) and dual cantilevers (b), respectively.

Figure 4 SEM images of batch fabricated dual cantilever probes with tip-to-tip spacing of (a) 300 nm and (b) 2 μm.

Figure 5 Schematic diagram of experimental setup for thermal scan with a dual cantilever resistive probe.

Figure 6 (a) Topographic and (b) thermal scanning images of a glass sample with 50 nm thick of gold lines on top. The scan was performed by a dual cantilever probe with tip-to-tip spacing of 300 nm.

Figure 7 Signal level as a function of pressure for dual cantilever probes with tip-to-tip spacing of 2 μm and 10 μm.


(a)
(b)
(c)
The bar
EBL
alignment
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(d)
(e)
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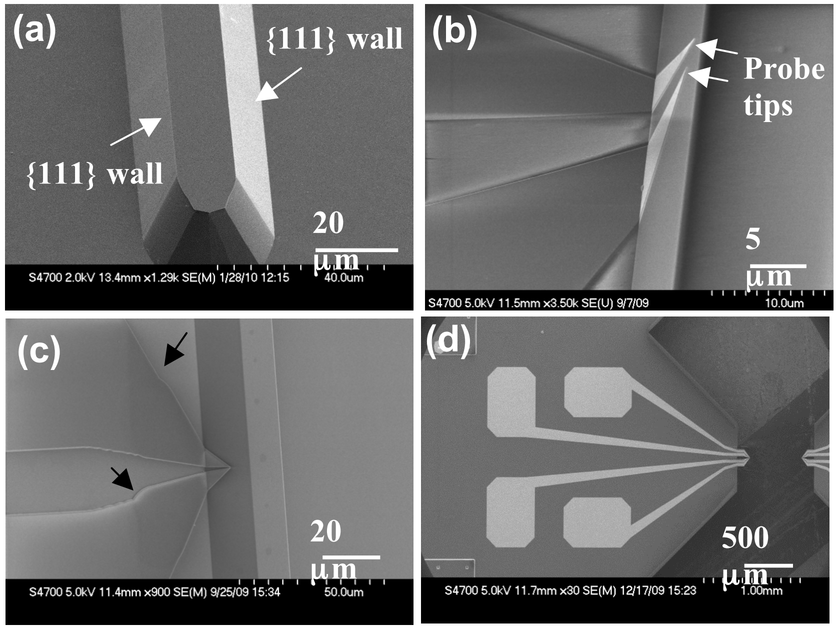
Pd resistor
(h)
(i)
(j)
Si
Si_3N_4
SiO_2
Pd
Au

Figure 1



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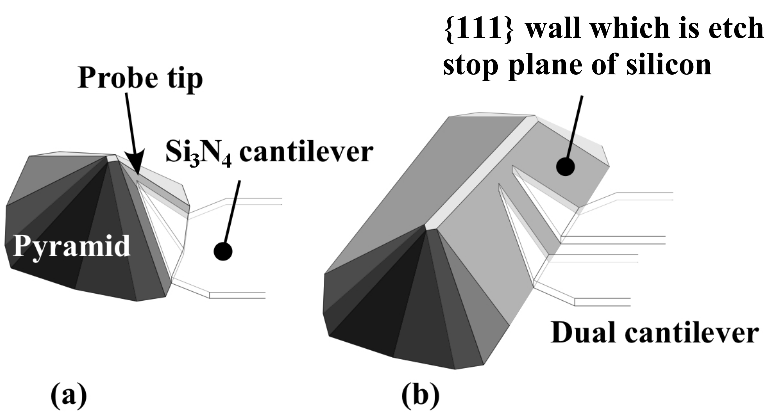

Figure 3

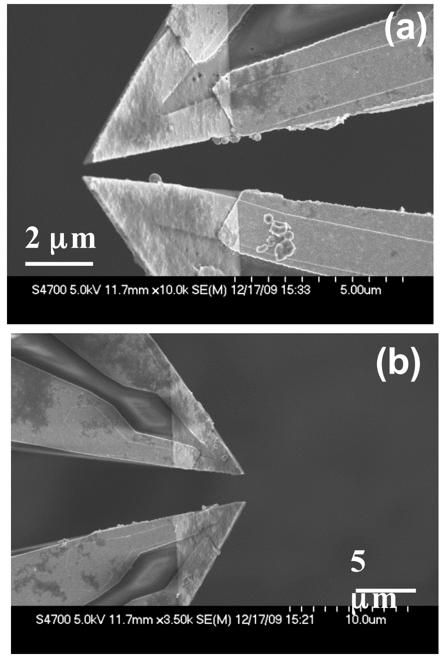

Figure 4

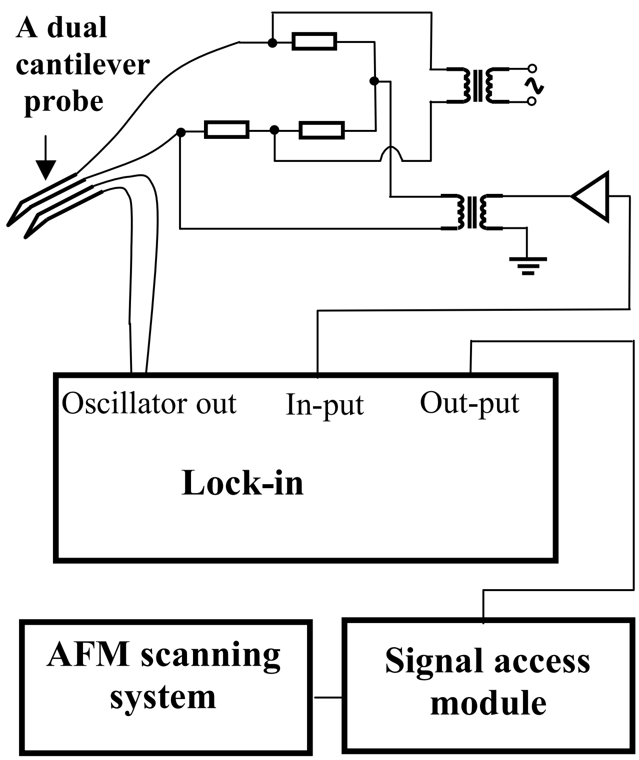

Figure 5

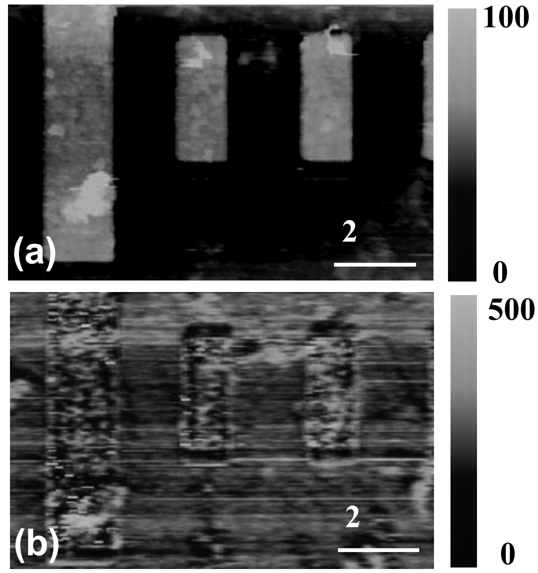

Figure 6

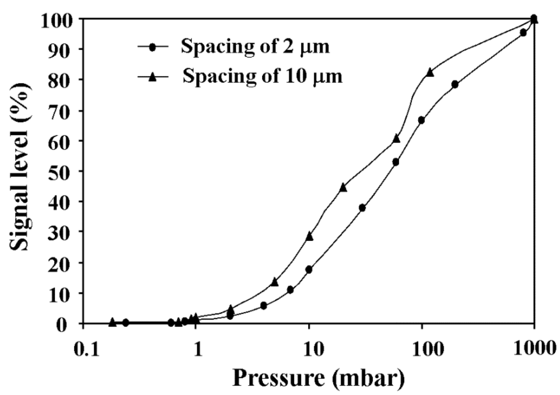

Figure 7