Microcalorimetry applications of a surface micromachined bolometer-type thermal probe

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(Received 1 June 2000; accepted 27 July 2000)

This article describes a surface micromachined scanning thermal probe that uses polyimide as the structural material and an embedded thin film metal resistor as the sensing element. The typical dimensions of a fabricated probe are 350 μm in length, 50 μm in width, and 3–10 μm in thickness. The resistor and the scanning tip are formed by sputter-deposited films of nickel and tungsten, which provide temperature coefficient of resistance of 2963 ppm/K. The probe is used to map surface and subsurface spatial variations in the thermal conductivity of a test sample. It is also used as a spatially localized microcalorimeter to measure the glass transition temperature of photoresists: the values obtained for Shipley 1813 and UV6 are 118±1 °C and 137±1 °C, respectively. These are in close agreement with results obtained by other methods that utilize larger samples. © *2000 American Vacuum Society*. [S0734-211X(00)01206-3]

I. INTRODUCTION

A number of scanning thermal probes have been developed in the past decade for mapping spatial variations in surface temperature or thermal properties of samples. The transducing elements for these devices have included thermocouples, Schottky diodes, bolometer-type resistance change, or bimorphs.[1–9] A bolometer-type sensing element, which maps temperature by fractional changes in the electrical resistance, can provide certain advantages for microcalorimetry applications. In particular, the resistor can be used to supply heat if adequate current is passed through it. Since the tip temperature is ultimately influenced by the heat flow between the tip and the sample, variations in thermal conductivity across the sample can be mapped by this probe. If the heat is supplied by a periodic signal, local variations in thermal capacity can also be measured. In essence, since the probe tip serves as a point source of heat as well as a temperature sensor, the device can be used as a spatially localized microcalorimeter.[10–12]

Although lithography-based micromachining techniques have been successfully used for fabricating scanning thermal probes in the past,[5–8] they have typically relied on the removal of the scanning probe from its host substrate or the dissolution of a portion of the substrate in order to provide the necessary clearance for the scanning tip. A fabrication process based on surface micromachining that circumvents these procedures was recently developed.[9] It exploits the mechanical flexibility of polyimide to implement an assembly technique that eliminates the need for probe removal or wafer dissolution. An additional benefit of polyimide is that it offers a very high degree of thermal isolation: its thermal conductivity is 0.147 W/mK, in contrast to 141.2 W/mK for silicon. Moreover, since this process has a small thermal budget, it will eventually permit the thermal probes to be postprocessed onto integrated circuit chips. This article describes a polyimide probe that uses an embedded thin film metal resistor as the sensing element. The sensing resistor and the scanning tip are both formed by a deposited thin film of metal. The probe has been used to map surface and subsurface spatial variations in the thermal conductivity of a test sample. It has also been used to measure the glass transition temperature of photoresists. Section II presents the structure and operation of the probe, Sec. III describes the fabrication sequence, and Sec. IV describes experimental results that have been obtained.

II. STRUCTURE AND OPERATION

The structure of the bolometer-type polyimide thermal probe is illustrated in Fig. 1. A cantilever with a thin film metal resistor sandwiched between two layers of polyimide extends past the edge of the substrate. The scanning tip is formed with the same metal that forms the resistor. It is molded into a notch etched in the substrate and initially points downward. After processing, it is released from the substrate and the probe shank is folded over so that the tip points upward and also has the necessary clearance from the substrate to permit alignment to the sample. Typical dimensions of the probes used in this effort are 350 μm in length, 50 μm in width, and 3–10 μm in thickness. Figure 1 shows two probes with their leads connected in series. One probe is used as a reference so that a differential measurement can be made.

The bolometer-type thermal probe can be used for sensing variations in the thermal conductivity of a sample surface. A current is passed through the probe resistor to cause joule heating at the tip. Variations in heat flow between the tip and the sample, influenced by local variations in the thermal conductivity of the sample, are measured by monitoring the probe resistance and then used to construct the scanned image. This mode of operation can be executed with constant

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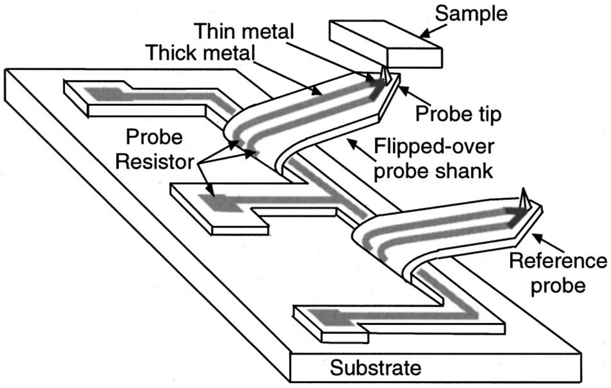

FIG. 1. Schematic of the bolometer-type polyimide thermal probe and a companion probe that may serve as a reference.

input current, or with the tip maintained at a constant temperature using a feedback circuit to control the heating. The probe may also be utilized as a spatially localized calorimeter. Rather than heating an entire sample, such as in a conventional differential scanning calorimeter, only the region in the vicinity of the tip is heated. A property change in the sample material that occurs as the probe temperature is ramped up will generally affect the temperature change per unit input power.[10] This is presented as a change in slope of a plot of the probe resistance versus input power. This method is used to determine the glass transition temperature (T_g) of photoresists, as described later.

III. FABRICATION

The fabrication sequence illustrated in Fig. 2 requires six masking steps. First, a pyramidal notch that serves as the tip mold is created by anisotropic wet etching of a (100) oriented Si substrate. Next, a 2 μm thick sacrificial layer of Ti is deposited and patterned. The first of two polyimide layers is then applied, but cleared from the field regions and the notch. The resistor and its leads are formed in the following two masking steps. For the purpose of this effort a 4000 Å thick sputter deposited film of Ni/W was used. The choice of metals is limited to those that are not attacked by buffered HF, since this acid is used to etch away the sacrificial layer at the end of the process. The metal segments along the probe shank are thicker than at the tip to reduce the resistance of the lead transfer. The tip diameter is 2000 Å, and can be reduced by introducing a thermal oxidation step following the anisotropic wet etch in the fabrication sequence. (This oxidation step would occur between the formation of the notch and the deposition of the sacrificial Ti.) The second polyimide is deposited and patterned after the metal. Finally, the sacrificial layer that extends along a portion of the probe shank is etched away, partially releasing the probe from the substrate. The probe is manually flipped over and restrained with an integrated polyimide clip or with a dab of epoxy. In the future, ultrasonic actuation[13] or stress mismatch between the two layers of polyimide[14] may permit this step to be automated. Figure 3 shows an optical micrograph of an unreleased probe, and Fig. 4 shows scanning electron micrographs of released probes after the flip-over assembly step.

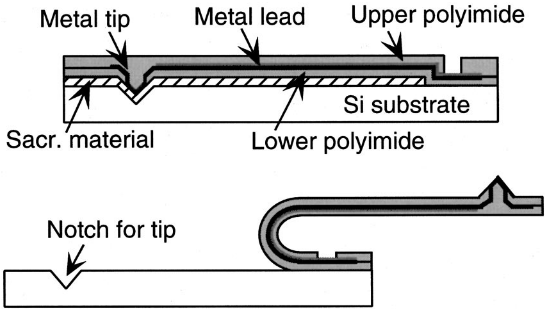

FIG. 2. Schematic of a device cross section illustrating the six-mask fabrication sequence.

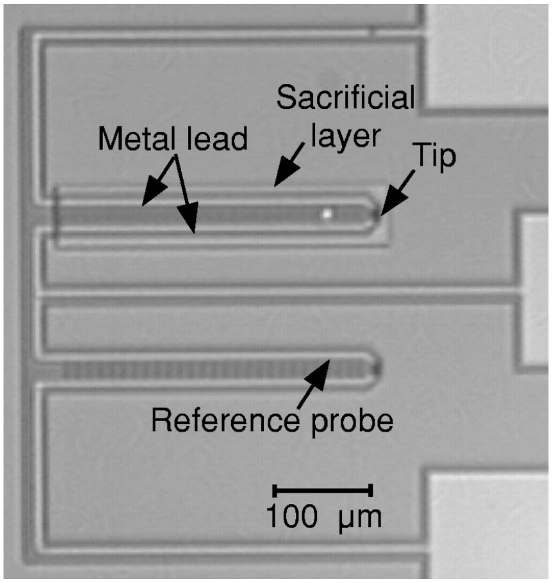

FIG. 3. Optical micrograph of an unreleased thermal probe.

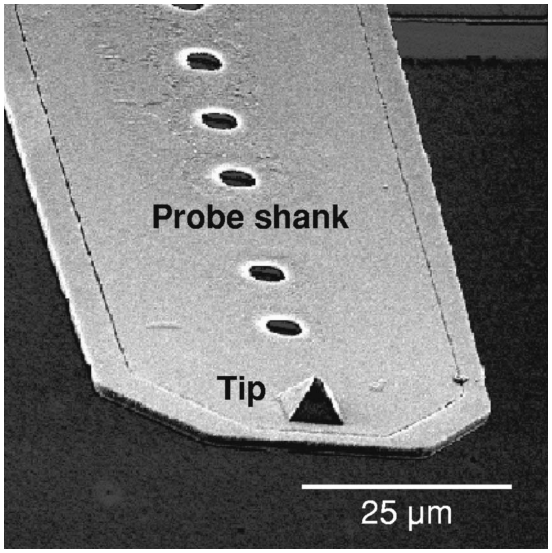

FIG. 4. SEM image of a scanning probe tip after the flip-over assembly step has been completed.

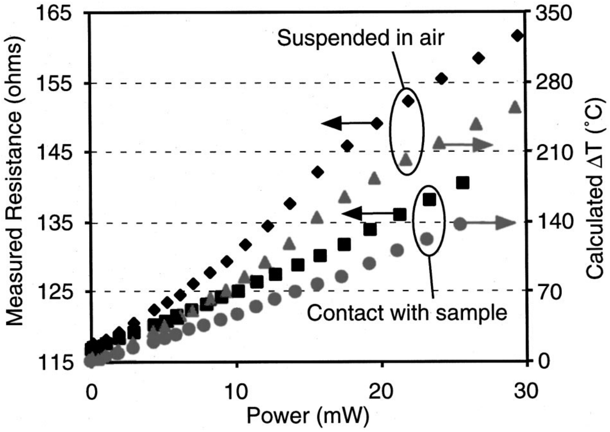

FIG. 5. Variation of probe resistance and calculated temperature as the input power is increased. When the probe is in contact with a sample, its temperature change per unit input power is smaller than when it is simply suspended in air.

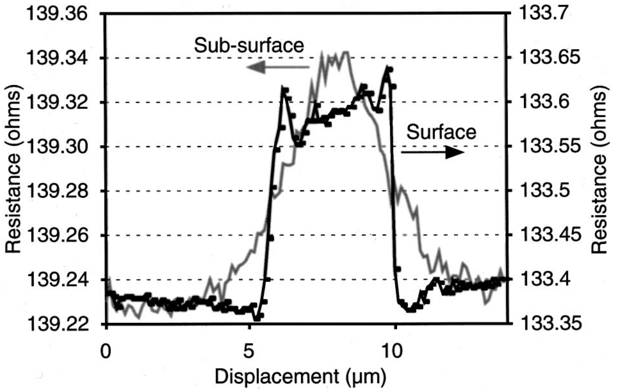

FIG. 6. Linear scan across a 3400 Å thick 5 μm wide metal line patterned on a glass substrate, along with a scan across a similar sample covered by a 1 μm thick layer of baked photoresist, demonstrate the ability to map the contrast in thermal conductivity in superficial and subsurface layers.

IV. MEASUREMENT RESULTS

The sensitivity of a bolometer-type thermal probe is directly related to the temperature coefficient of resistance (TCR) of the sensing resistor. The measured TCR of the Ni/W thin film that constitutes the resistor was 2963 ppm/K. This information permits the tip temperature to be calculated from the resistance change. Note that the resistance measurements for this and all other experiments reported here were performed using the four-probe technique by which the impact of contact resistance at the probe pads is eliminated.

Figure 5 shows the resistance change in a fabricated polyimide probe as the input power is increased. Using the measured TCR, the temperature change at the probe tip with 25 mW input power reaches 225 °C when the probe is suspended in air and the tip is not in contact with any sample. When the tip is brought into contact with a glass substrate, the conductive heat loss reduces the temperature change to 133 °C for the same input power. The electrical resistance of the probe can therefore be used to monitor the proximity of the tip to the sample. The probe used in these experiments was 350 μm long, 50 μm wide, and 3 μm thick. It had calculated spring constant of about 6.5×10^{-2} N/m, which is relatively low, and conducive to scanning soft materials. The estimated contact force due to the deflection of the polyimide cantilever is $<10^{-9}$ N. This probe was used to scan a 3400 Å thick, 5 μm wide metal line patterned on a glass substrate in order to test its ability to detect a contrast in thermal conductivity. The experiments were performed with the tip in contact with the sample. The tip was positioned with a micromanipulator, while the scanning motion was obtained from a piezoelectric bimorph. A linear scan that was obtained while supplying a constant current of 12 mA is shown in Fig. 6. A sharp change in probe resistance is evident as the tip traverses the metal line. The scan was repeated with the sample surface covered by a 1 μm thick layer of baked photoresist. The result is superimposed on the original scan in Fig. 6, and demonstrates that subsurface mapping can be performed. The maximum resistance change in the absence of the photoresist was 0.19%, whereas in the subsurface measurement in the presence of the photoresist it was 0.09%. A two-dimensional plot of the thermal conductivity contrast (taken without the photoresist layer) is shown in Fig. 7. The inset shows an optical micrograph of the scanned sample. The two linear segments of metal that run parallel to each other are 550 Å thick, 10 μm wide, and 20 μm apart. The substrate is 500 μm thick No. 7740 glass. The spatial resolution of the scanned image is limited by scanning apparatus that was available.

The polyimide probe was also used to perform spatially localized measurements of T_g of photoresists. Figure 8 shows a plot of the probe resistance (and calculated tip temperature) versus input power for a glass sample covered with Shipley 1813 photoresist, which is used in I-line lithography. The location of the change in slope indicates that the T_g is 118 ± 1 °C. This agrees with a previously published result obtained by an ultrasonic method which also found that the T_g was 118 °C.[15] A similar measurement indicated that T_g is 137 ± 1 °C for the chemically amplified Shipley resist UV6, which is used in deep ultraviolet lithography. This measurement compares well with the value of 143 ± 2.5 °C determined by the gradient of film stress on a full-wafer sample.[16] The consistency of the measurements with expectations in-

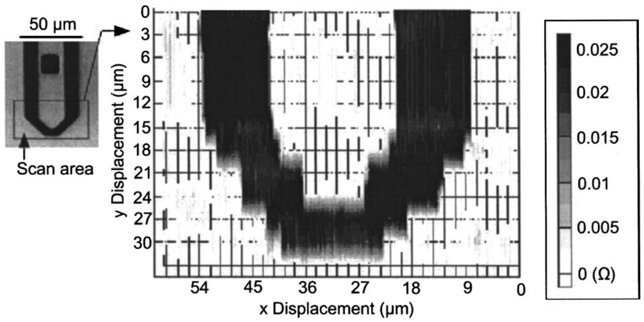

FIG. 7. Two-dimensional image of thermal conductivity contrast obtained from a sample of 550 Å thick metal patterned on a glass substrate.

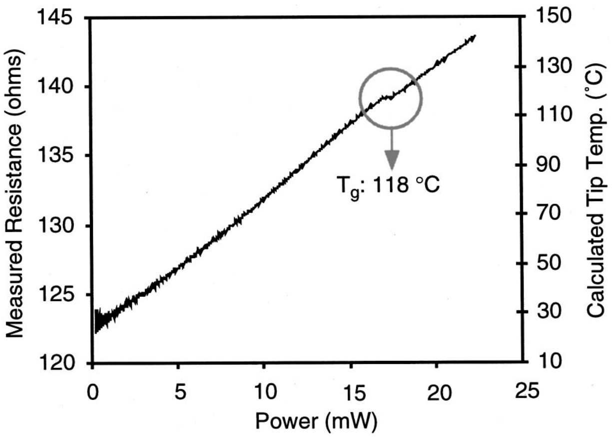

FIG. 8. Measurement of the glass transition temperature (T_g) of photoresists by the polyimide probe shows $T_g = 118 \pm 1$ °C for Shipley 1813 resist.

dicates that the polyimide probe can be a useful tool for photoresist research.

V. CONCLUSION

A scanning thermal probe that utilizes polyimide for the structural material, sputtered metal for the scanning tip and a metal resistor as the transducing element has been presented. Its fabrication sequence involves a six-mask surface micromachining process with a low thermal budget which will permit its eventual integration with on-chip circuitry. The probe is folded out over the edge of the die to provide tip clearance without etching the substrate. Typical probe dimensions for this study were 350 μm in length, 50 μm in width, and 3–10 μm in thickness. A tip diameter of 2000 Å is easily achieved with the existing process; the introduction of a thermal oxidation step in the fabrication sequence can reduce this further.

The operation of the probe has been validated by mapping the contrast in thermal conductivity of a sample with a 550 Å thick metal film patterned on a 500 μm thick glass substrate. Subsurface imaging has been demonstrated by repeating this scan with a 1 μm thick layer of photoresist covering the sample. The T_g of Shipley resists 1813 and UV6 have been determined, and are found to closely match expectations. These results demonstrate the potential usefulness of the polyimide probe as a tool for research in photoresists.

ACKNOWLEDGMENTS

The authors thank Julius Wu for help with mask layout. This work was funded in part by the Semiconductor Research Corporation Contract No. 98-LP-452.005. The Center for Nano Technology, University of Wisconsin–Madison, is supported in part by DARPA/ONR Grant No. N00014-97-1-0460. The Synchrotron Radiation Center of University of Wisconsin–Madison, at which some of the experimental facilities are located, is operated under NSF Award No. DMR-95-31009.

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