IN-SITU MONITORING OF MICRO-CHEMICAL VAPOR DEPOSITION (μ -CVD): EXPERIMENTAL RESULTS AND SPICE MODELING

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ABSTRACT

Two new methods of monitoring the thickness of metallic films *as deposited* on microfabricated bridge structures *via* micro-chemical vapor deposition (μ -CVD) are described. These methods require no additional fabrication to produce four-point resistance-based thickness probes. Instead, the relatively large increase in (a) the heat conductance and (b) the heat capacity of the microbridge/metal system as a result of deposited metal are detected as variations in the heating power provided to the microbridge by the control circuitry.

Experimental results demonstrating these effects are given for a microbridge controlled with a novel constant-resistance circuit. The heat-transfer equations for the microbridge/metal system and the circuit model for the control circuit are then solved using SPICE. This coupled model predicts within 10% the measured thickness of μ -CVD platinum.

INTRODUCTION

Recently, μ -CVD of metals on micromachined devices has been reported [1,2,3,4]. In this technique the self heating of microhotplates and microbridges is used to thermally decompose precursors directly on their surface (Figures 1 and 2). The high degree of thermal isolation of these structures confines deposition only to their active regions, and is therefore highly selective. In the absence of this technique, one additional deposition and patterning step would be required in a conventional microfabrication sequence to fabricate such a film [5], and coverage of the underside of suspended structures would be difficult to achieve. Applications of μ -CVD include noble-metal catalyst deposition for calorimetric gas sensors [3] and metal deposition for conductometric gas sensors [1,2,4].

In-situ thickness monitoring *via* measurement of the thin-film resistance of μ -CVD metal on microhotplates has been demonstrated [1,2]; for this purpose, four-point probes were fabricated. We show that the sensitive heat-transfer properties of microheater platforms such as microhotplates and microbridges can be utilized instead. Given their relatively small values prior to metal deposition, the (a) heat conductance and (b) heat capacity of the microheater/metal system increase significantly with deposited metal quantity and can be detected *in situ* with appropriate control circuitry. While (a) alters the steady-state heating power requirements of the system, (b) introduces a delay in its transient response to a heating pulse.

These effects are demonstrated experimentally for μ -CVD of Pt on CMOS-compatible, silicon-nitride-encapsulated, polysilicon microbridges. A constant-resistance control circuit used for microbridge heating during μ -CVD is presented (Figure 3). This circuit provides both steady-state (CW) and pulsed resistance set points so that both heat conductance and capacity changes can be probed. It should be stressed that the techniques described here are applicable to microhotplates as well, and that no additional

microfabrication is required, in contrast to four-point resistance probes. Furthermore, *in-situ* monitoring of the μ -CVD of *dielectrics* should also be achievable in a pulsed-bias mode, since this technique only requires changes in heat capacity.

An electro-thermal model of the microbridge/metal system is coupled with the circuit model of the control circuit and solved in SPICE. Boundary conditions, implementations of temperature-variable resistors, and the conductivity and heat capacity of the deposited metal are described. Simulations predict metal thickness within 10% of measured values for both CW and pulsed heating bias.

Figure 1. Scanning electron micrograph of two meandered microbridges. The lower bridge appears brighter due to a smooth layer of Pt approximately 0.1 μm in thickness, which was deposited by μ -CVD from the precursor $\text{Pt}(\text{acac})_3$.

Figure 2. Focused ion beam cross section of a silicon-nitride-encapsulated, polysilicon microbridge with a rough coating of μ -CVD platinum. The microbridge is 2 μm thick and 10 μm wide.

THEORY

As the thickness of metal deposited on a microbridge increases, so does its ability to conduct heat along the device. This means that the power, $P(t)$, required to maintain the bridge/metal system at constant resistance, R , increases with deposition time. The relative power provided to the bridge can be written

$$P(t)/P_o = 1 + P_m(t)/P_o, \quad (1)$$

where P_m is the increasing power conducted by the catalyst and P_o is the power required prior deposition. By the chain rule, the rate of increase of P_m with metal thickness, z_m , at constant resistance is

$$\left. \frac{dP_m}{dt} \right|_R = \left. \frac{dP_m}{dz_m} \right|_R \cdot \left. \frac{dz_m}{dt} \right|_R. \quad (2)$$

The growth rate of the film is then

$$\left. \frac{dz_m}{dt} \right|_R = \left. \frac{dP_m}{dt} \right|_R \cdot \left(\left. \frac{dP_m}{dz_m} \right|_R \right)^{-1}. \quad (3)$$

The composite thermal conductivity K of the microbridge with deposited metal is

$$K(z_m) = K_b + \left[\frac{(2z + w)z_m}{wz} \right] \cdot K_m \cdot f, \quad (4)$$

where K_b , z and w are the conductivity, thickness, and width, respectively of the bridge. The factor f accounts for the fact that the deposited film has less than bulk density (Figure 2), so its actual conductance is less than that implied by the bulk conductivity K_m of the metal. Finally, the term in brackets is the ratio of cross-sectional area of the deposited film, to that of the bridge, as shown in Figure 4.

The heat capacity of a differential element of the bridge/metal system is the parallel combination of the capacity of the bridge, and the capacity of the metal. For a bridge of length L divided into N elements, the latter term is

$$C_m = f \cdot c_m \cdot (2z + w) \cdot z_m \cdot (L / N), \quad (5)$$

where c_m is the bulk specific heat of the metal. The factor f is the same as in Eq. (4).

Figure 3. Constant-resistance control circuit. A CW set point is shown, but pulsed operation is also possible.

Figure 4. A cross section of the microbridge with deposited metal catalyst. Dimensions of the bridge and assumed average thickness of the catalyst are shown

EXPERIMENTAL DETAILS

The μ -CVD apparatus is described elsewhere [3,6]. During μ -CVD of Pt, the microbridge was heated with a constant-resistance control circuit (Figure 3) and the power provided to the device was monitored. Referring to Figure 3, the resistance and current of the bridge (for the feedback resistor values shown) are

$$R = 60.2 \cdot V_R \quad \text{and} \quad I = V_I / 0.31, \quad (6)$$

and the power is obtained by the relation $P = I^2 R$. For CW biasing, the voltages V_R and V_I of Eq. (6) were recorded with a digital multimeter; for pulsed biasing, an 8-bit digitizing oscilloscope was used. Note that the polysilicon bridge material is electrically insulated from the deposited metal by a 0.25 μ m layer of LPCVD silicon nitride.

Figure 5. Relative power provided to a microbridge by the control circuit in a CW bias-mode μ -CVD experiment. P_o is the power required prior to metal deposition. Depositions for 12 min (filled triangles), and 15 min (open squares) are shown, along with a linear fit to the latter data. For both depositions, the bridge resistance was maintained at $1.4R_o$, where R_o is its room-temperature resistance.

EXPERIMENTAL RESULTS AND DISCUSSION

The relative power provided by the control circuit to maintain constant (CW) resistance during Pt deposition is shown in Figure 5. Two depositions of 12 and 15 min total time on two separate devices are shown. These data can be described by Eq. (1). Initially, the power does not increase due to the fact that although Pt is being deposited, the film is not yet continuous and carries no additional heat to the bridge ends. With increasing deposition time, however, the Pt nucleation sites connect and the film begins to conduct heat; this change is evident at 0.75 min into the deposition. Subsequently, growth is nearly linear in time and the slope is precisely that given by the left-hand side of Eq. (2).

Following deposition, the device with a 12 min deposition was cross-sectioned in four places along its length using a focused ion beam (FIB). A metal thickness distribution was thereby

obtained, and from this an average metal thickness of 0.67 μm was derived. One such FIB cross section is shown in Figure 2.

Deposited Pt significantly increases the heat capacity of the microbridge/metal system, introducing a measurable delay in the transient response of the system to a heating pulse. The two curves shown at the top of Figure 6 are the measured response of a microbridge to a square heating pulse (a) before and (b) after a 12 min CW deposition. (Note that this deposition was performed at a bridge resistance of 1.4 times the room-temperature value for the bridge, the same conditions under which the CW depositions of Figure 5 were performed.) The system's response *after* metal deposition is delayed with respect to that *before*. Furthermore, in the steady state, the power required after deposition is greater than that before; this is the same effect as that already described for the CW depositions above.

SIMULATION DETAILS

The basic electro-thermal model of a microbridge is based on the work of Mastrangelo [7], where the temperature of an element of the bridge is determined by Joule heat gain, and losses due to (a) conduction along the bridge, (b) conduction through the air gap from the hot bridge to the underlying substrate, (c) convection, and (d) radiation. The length of an element is L/N for a bridge of length L divided into N elements.

Using electrical analogues of thermal parameters, components representing the various heat-loss mechanisms and Joule heating gains were implemented in PSPICE™, as diagrammed in Figure 7. Voltage-controlled current sources (VCCS) were used to make the magnitude of convective and radiative losses and Joule heat gains in a given element dependent on the temperature (voltage) of the node. In this way, complex functional dependencies, such as the fourth-order temperature dependence of radiation, were easily implemented. Secondly, temperature-variable thermal and electrical resistors were constructed from a series combination of a voltage-controlled voltage source and an ammeter (a 0-V independent source in PSPICE™). This is possible since, by Ohm's Law, resistance and voltage are proportional. The output of a VCVS for a given element (the element resistance) is directly proportional to the heating current into that element, as measured by the ammeter. The thermal resistance of an element is also inversely proportional to the thermal conductivity defined in Eq. (4).

Mastrangelo's model assumed that the temperature of the silicon substrate below the device remains constant, regardless of device temperature. In fact, since the substrate is not an ideal heat sink, the temperature in the vicinity of the microbridge increases as the microbridge is heated. This effect is greatest at the microbridge ends, as most of the heat generated within the device is conducted along its length and deposited at its extremities. This leads to localized heating at the ends of a microbridge and increased resistance to heat flow there. By analogy to electrical spreading resistance [8], this effect has been called "thermal spreading resistance", and can be modeled in SPICE by placing additional thermal resistance between the bridge ends and the substrate [5,6].

With measured values of the thermal conductivity, K_b , and heat capacity, C_b , of polysilicon inserted, the SPICE model just described accurately predicts the temperature profile, current-voltage characteristics, and transient response of microbridges without deposited metal [5,6]. To simulate the effects of deposited metal on the steady-state heating characteristics of the bridge/metal system, a thermal conductivity given by Eq. (4) is required. For transient analysis of the system, the elemental heat capacity, C_m , of the deposited metal, given by Eq. (5), is also required and was added in parallel to C_b . Finally, to simulate the coupled response of the system and control circuit, a circuit model for the control circuit of Figure 3 was constructed and connected to the electro-thermal model of the system.

SIMULATION RESULTS

Simulation results for the coupled model consisting of the control circuit and bridge/metal system are described in this section. The growth rate, as defined by Eq. (3), was first predicted. The first factor there is given experimentally by the slope of the line in Figure 5. The second factor, the rate of change of P_m with z_m , is easily determined with the SPICE model, since the composite thermal conductivity described by Eq. (4) introduces the effects of z_m and f on the steady-state behavior of the system. With this in place, one simply selects values of f , R , and z_m and the model calculates the corresponding power. From the 15 min deposition at $R = 1.4R_o$ in Figure 5, $dP_m/dt = 0.82 \text{ mW/min}$. For this same set point and $f = 1/3$, SPICE calculations yield $dP_m/dz_m = 14 \text{ mW}/\mu\text{m}$. From Eq. (3), then, a Pt growth rate of 59 nm/min is obtained. By way of comparison, this rate implies a thickness of 0.7 μm for a 12 min deposition, in agreement with the measured value of 0.67 μm obtained by FIB cross sections.

The transient response of the system to a heating pulse was next simulated, and a comparison with experiment is given in Figure 6. The two curves in each graph of Figure 6 represent the transient response (a) *before*, and (b) *after* a 12 min CW deposition at $1.4R_o$. Note that this was the same deposition used to evaluate the CW results above. One would expect, then, that the value of f obtained here should be close to 1/3. In fact, in the simulation of Figure 6 values of $f = 1/3$ and $z_m = 0.7 \mu\text{m}$ were used for comparison with the CW results. The simulated heating delay (measured at 40 mW) was 0.29 ms, a value 15% smaller than the measured value of 0.35 ms. Using $f = 0.37$, however, yields the correct heating delay of 0.35 ms. This value of f is within 10% of that used in the continuous set-point simulation. Finally, it should be noted that the simulated and measured steady-state heating difference before and after deposition (shown as P_c in Figure 6), agree within 10%.

SUMMARY AND CONCLUSIONS

The relatively large increase in the heat conductance and heat capacity of a microbridge due to $\mu\text{-CVD}$ metal can be detected in real time *as the metal is being deposited* through a variation in the heating power provided to the microbridge/metal system. A novel constant-resistance control circuit was presented that can provide either a continuous or pulsed heating bias to the microbridge during deposition. During steady-state operation, the change in heat conductance with the deposited metal is obtained, and can be used to determine the film thickness *in situ*. On the other hand, during pulsed biasing, the increase in heat capacity with film deposition is extracted and can also be used for *in-situ* determination of the film thickness.

The techniques described here are applicable to *in-situ* monitoring of $\mu\text{-CVD}$ on microhotplates as well, and do not require any additional microfabrication to produce four-point resistance-based thickness probes on the device. Finally, *in-situ* detection of the $\mu\text{-CVD}$ of dielectrics on microheater platforms should also be possible using pulsed biasing, since this technique only requires changes in the heat capacity of the system for detection.

The principal advantage of SPICE modeling over other numerical methods is the ability to simultaneously simulate the electro-thermal behavior of a device and its control/sense electronics. Such a coupled model was presented for a microbridge with deposited metal and its constant resistance control circuit. Simulations predicted within 10% the measured values of the thickness of $\mu\text{-CVD}$ metal in both pulsed- and CW-deposition bias modes.

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Figure 6. The measured (top) and simulated (bottom) response of a 100- μm bridge to a $1.3R_0$ square heating pulse, where R_0 is the room-temperature resistance of the bridge. The power indicated is that required to maintain $1.3R_0$. Time response both "before" and "after" a 0.7 μm thick Pt deposition are shown. Note that heating was initiated at the same time both before and after deposition. Simulation results are for $f = 0.33$.

Figure 7. Elements 'i' and 'i+1' of the bridge in SPICE. The electrical resistors, 'R', are coupled to the thermal model at the points indicated by double arrows. The series combination of a VCVS and 0-V independent source functions as a temperature-dependent resistor. Joule heating (upward-pointing arrow) and losses (downward-pointing arrow) are modeled with VCCSs. The heat capacity of an element is represented by a capacitor.