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2006-09-01

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Original Publication Citation

Ashwini R. Kadam, G. P. Nordin, and Michael A. George, "Comparison of microcantilever Hg sensing behavior with thermal higher order modes for as-deposited sputtered and thermally evaporated Au films," *J. Vac. Soc. Am. B* 24(5) pp. 2271-2275 (26)

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Nordin, Gregory P.; George, Michael A.; and Kadam, Ashwini R., "Comparison of microcantilever Hg sensing behavior with thermal higher order modes for as-deposited sputtered and thermally evaporated Au films" (2006). *Faculty Publications*. 978.
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Comparison of microcantilever Hg sensing behavior with thermal higher order modes for as-deposited sputtered and thermally evaporated Au films

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(Received 12 April 2006; accepted 1 August 2006; published 18 September 2006)

As-deposited sputtered and thermally evaporated Au films are studied for Hg sensing with rectangular Si microcantilevers. Microcantilever deflection and frequency shifts in the first four thermally induced flexural modes are measured for each type of Au film as a function of Hg exposure. All thermally induced higher order modes show shifts in their frequencies for Hg exposures when the Au is sputter deposited whereas typically only the third and fourth order modes show frequency shifts when the Au is thermally evaporated. A technique is employed in order to vary Au coverage along a microcantilever surface. The shifts in the thermal microcantilever modes upon Hg exposure are thus studied as a function of fractional Au coverage along the microcantilever surface. Microcantilevers having various coverages of either sputtered or thermally evaporated Au films show a distinct response to Hg exposure. From both the deflection and spectral shifts it is observed that sputtered films give better response to Hg than thermally evaporated films when the Au is coated along the entire length of the microcantilever. © 2006 American Vacuum Society. [DOI: 10.1116/1.2345204]

I. INTRODUCTION

Microcantilevers have been widely studied for detection of adsorption of analyte specific mass by monitoring the frequency shift of the externally excited fundamental mode.¹⁻³ A number of studies have examined the use of Au-coated microcantilevers for sensing Hg adsorption.⁴⁻⁶ As-deposited thermally evaporated⁴⁻⁶ or sputtered⁷ Au films are employed as the Hg sensing surface. Adsorption of Hg onto the Au film deposited on microcantilever surface results in bending of the microcantilever and shifting its fundamental resonant frequency.⁴⁻⁷

Recently it has been shown that monitoring frequency shifts of higher order thermal microcantilever modes is a viable detection mechanism.⁸ In this article we compare microcantilever Hg sensing behavior with thermal higher order modes for as-deposited sputtered and thermally evaporated films. We investigate both types of Au films for various fractions of microcantilever coverage to determine what coverage gives the largest frequency shift for Hg sensing.

II. EXPERIMENT

The optical reflection technique is employed to study commercially available rectangular Si microcantilevers (Ultrasharp). The experimental setup is shown in Fig. 1. A microcantilever mounted on a glass holder inside an atomic force microscope head (Digital Instruments) is fitted on top of a gas chamber in which a constant N₂ flow is maintained.

A Hg effusion tube (Vici Inc.) with an effusion rate of 32.576 ng/min is placed inside a Dynacalibrator (model 190, Valco Instruments Co. Inc.) and used to deliver precise concentrations of Hg to the gas chamber. The Hg concentration is constantly monitored at the outlet of the gas chamber with a Jarome Hg detector (Arizona Instruments). The output of the position sensitive detector is used to monitor the bending and thermal resonance spectrum⁹ of a microcantilever in the gas chamber.

An HP3588A spectrum analyzer is used to acquire frequency data at a resolution of 0.1 Hz. The experimental modal frequencies for each of the measured modes are given with the associated noise (i.e., the standard deviation of frequency measurement) in our setup. These modes have been verified as the flexural modes of microcantilever vibration.⁸ The standard deviations in Table I are an average of the standard deviations measured for ten different microcantile-

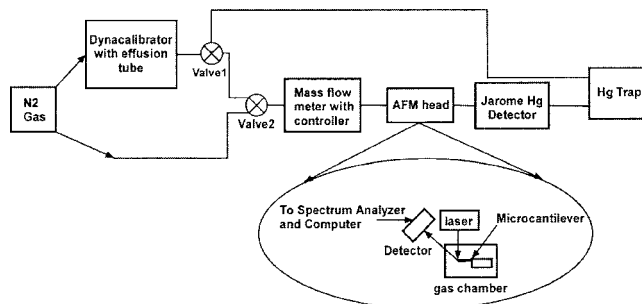


FIG. 1. Experimental setup.

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TABLE I. Modal frequencies and the associated measurement noise.

Mode	Experimentally measured frequency (Hz)	Observed standard deviation (Hz)
1	12 766	0.9
2	78 991	2.8
3	225 990	5.0
4	446 460	7.1

vers. The mode shapes of first four flexural modes along the microcantilever cross section are shown in Fig. 2.¹⁰

For sputter deposition of 5 nm Cr and 50 nm Au on one surface of a microcantilever, a Denton Discovery 18 sputtering system is used in which the base pressure is 5×10^{-6} Torr. For thermal evaporation of 5 nm Cr and 50 nm Au on one surface of a microcantilever a CVC thermal evaporator (CVC Products, Inc.) is used in which the base pressure is 2×10^{-6} Torr. The microcantilevers are transferred to the gas chamber following Au deposition and are studied for their Hg response.

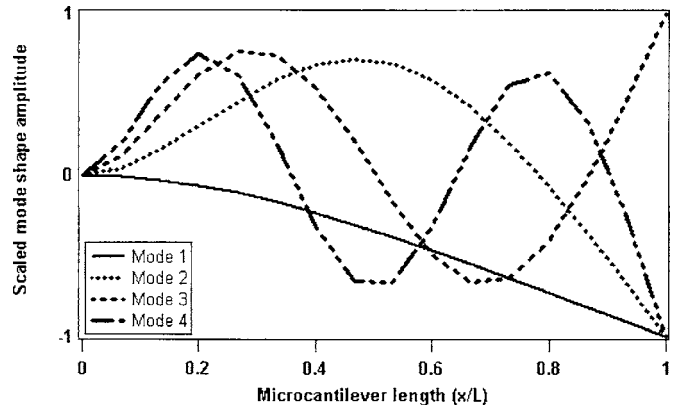


Fig. 2. Normalized mode shape amplitude as a function of the scaled microcantilever length (x/L) for the first four modes.

III. RESULTS AND DISCUSSION

Microcantilevers with both sputtered and thermally evaporated Au films deposited along entire length of one surface (i.e., 100% Au coverage) are exposed to four 100 s, 14 ppb Hg exposures with measured results as shown in Fig. 3. In Figs. 3(a) and 3(b) the microcantilever deflection and

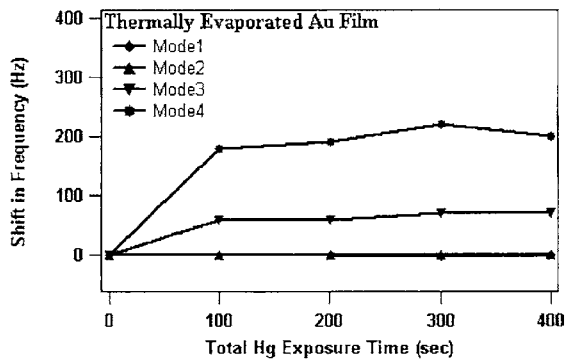
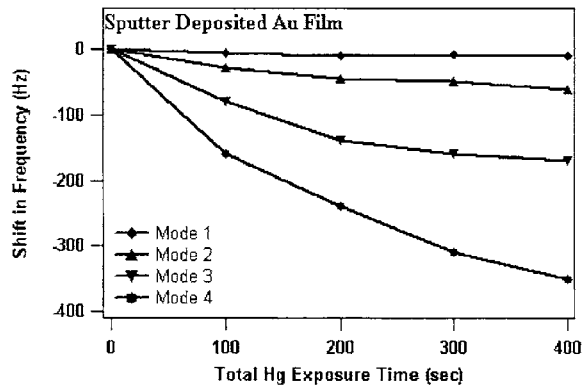
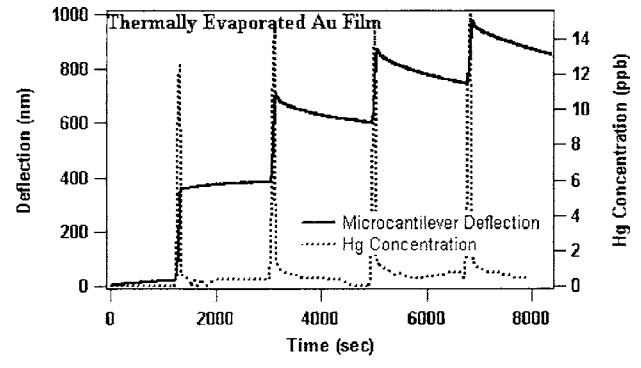
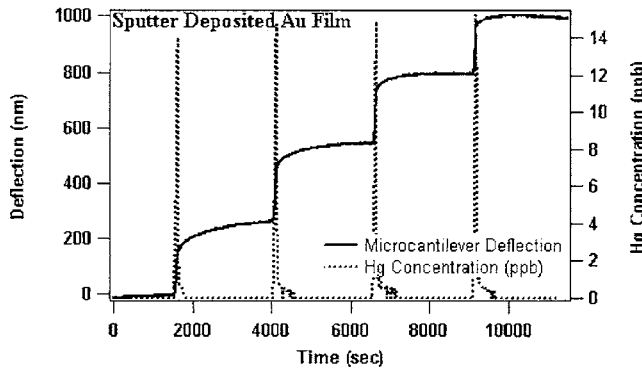


FIG. 3. (a) Microcantilever deflection and (c) frequency shift in the thermal modes after Hg exposures for sputter deposited Au. (b) and (d) are the same for thermally evaporated Au. The trends and features in each of these graphs were repeatedly observed for many microcantilevers (>10) subject to the same experimental conditions.

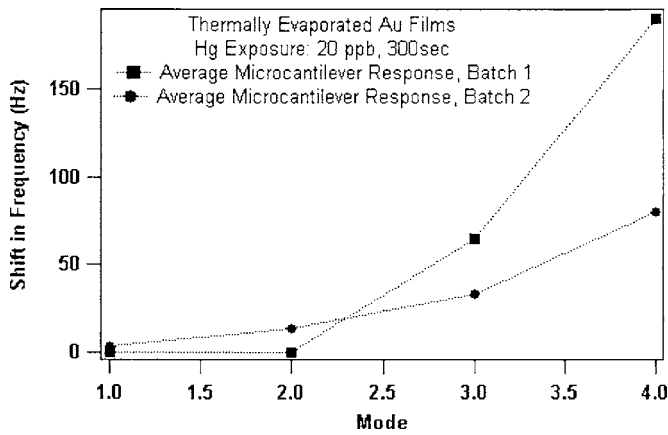


FIG. 4. Average shift in the thermally induced flexural modes observed for three different microcantilevers from Batch1 (rectangles) and Batch2 (circles) after 20 ppb Hg exposure for 300 s. Note the consistency in shifts between microcantilevers within a batch, but not between different batches. To make the differences between two batches easily visible the data points are connected with lines.

the Hg concentration as monitored by a Jarome Hg detector are plotted as a function of time for sputtered and evaporated Au films, respectively. Note that for sputtered films the deflection response of the microcantilever always stabilizes after the Hg is turned off whereas for thermally evaporated films it shows an exponential decay after the Hg exposure is stopped (except after the first exposure). The deflection response in both cases indicates that the microcantilever bends away from the Au surface and, hence, the resultant surface stress is tensile in nature.¹¹

The shifts observed in the modal frequencies of the first four thermally induced modes as a function of total Hg exposure time is plotted in Figs. 3(c) and 3(d) for sputtered and thermally evaporated films, respectively. The spectral shifts are negative for sputtered films and positive for thermally evaporated films. Moreover, all the studied modes except the fundamental mode (mode 1) show a response to Hg when the sensing Au film is sputter deposited. In contrast, when using thermally evaporated films the first higher order mode (mode 2) as well as the fundamental mode (mode 1) show no Hg response. The deflection and spectral response observed upon Hg exposure for both the sputtered and thermally evaporated Au films (Fig. 3) has been repeatedly observed for more than ten microcantilevers under the same experimental conditions.

In order to directly compare frequency shifts between different microcantilevers, it is important to determine the effect of small geometrical differences between commercial microcantilevers. For this purpose three different microcantilevers deposited with thermally evaporated Au films from two different microcantilever batches are studied under identical conditions for Hg exposure. The average shifts observed in their thermally excited modal frequencies are plotted as a function of the mode order as shown in Fig. 4. The important point here is that all microcantilevers show positive frequency shifts and the higher order modes show larger Hg response. The inconsistency seen between the spectral shifts

of microcantilevers from the two different batches is associated with the different microcantilever thicknesses for the two batches since the microcantilever spring constant is a sensitive function of its thickness. The mean microcantilever thickness for Batch1 (rectangles) measured with a scanning electron microscope is $1.44 \mu\text{m}$ whereas that for Batch2 (circles) is $1.32 \mu\text{m}$. Microcantilevers from the same batch (Batch1) are thus used in the studies presented in this article in order to quantitatively compare spectral shifts of the various microcantilever modes.

As discussed in the literature, sensing using the fundamental mode of a microcantilever under external excitation¹⁻³ is ideally conducted with the sensitizing coating deposited at the free end¹²⁻¹⁵ of the microcantilever. We have recently shown that thermally induced higher order modes can be used to detect Hg when Au is deposited at the free end.⁸ In this article we also examine the shifts in thermal microcantilever modes upon Hg exposure when the Au coverage is varied along the entire microcantilever length. This is done in order to experimentally determine what Au coverage gives the largest spectral response for Hg sensing with both sputtered and thermally evaporated Au films. For this purpose, spectral shifts upon Hg exposure of fourteen different microcantilevers from Batch1 are studied as a function of fractional Au coverage.

Seven microcantilevers are deposited with sputtered Au films and the other seven with thermally evaporated Au films. We have reported a simple technique to selectively coat microcantilevers with Au (Ref. 8) in which the Au is wet etched from the microcantilever surface using photoresist as the etch mask which is deposited via a nanopipette. Using this technique, Au on all of the microcantilevers is etched so that each one has a unique Au coverage. An example of various Au coverages resulting from a sequence of Au etch steps carried out on the same microcantilevers is

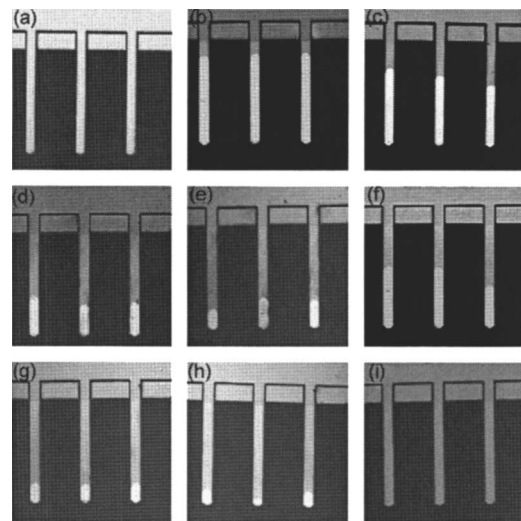


FIG. 5. Optical microscope images of three microcantilevers with various Au coverage achieved by subsequent wet Au etching. (a) 5 nm Cr, 50 nm Au coated microcantilever, (b)–(h) the same microcantilevers after subsequent Au etch steps, and (i) bare Si microcantilevers.

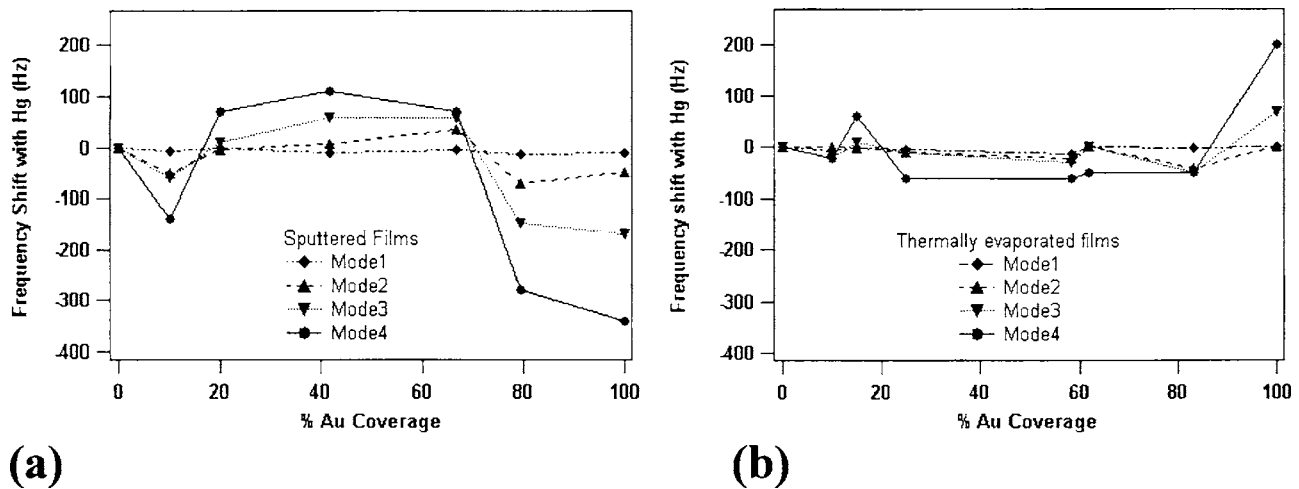


FIG. 6. Frequency shift with Hg exposure vs percent Au coverage for (a) sputtered Au films and (b) thermally evaporated Au films. Each plot shows data taken from seven different microcantilevers.

shown in Fig. 5. In Fig. 5(a) we begin with the entire microcantilever surfaces coated with Au (100% Au coverage). Each succeeding figure shows the result of progressively removing more Au from each microcantilever until there is no Au left as shown in Fig. 5(i) (0% Au coverage). Using this process we can control the fractional Au coverage of an individual microcantilever.

Each of the seven microcantilevers having a unique Au coverage of either sputtered or thermally evaporated Au films was exposed to 22 ppb Hg for 600 s. The thermal spectral data are recorded 1000 s after the Hg exposure is stopped. The measured spectral shifts for the first four thermal modes are shown in Fig. 6 as a function of percent Au coverage.

It is clear from Fig. 6(a) that for microcantilevers with sputter deposited Au films the nature of the higher order modal frequency shifts after Hg adsorption strongly depends upon the fractional Au coverage. All of the thermal modes except for the fundamental mode show some shift with Hg exposure for each fractional Au coverage studied here. However, for microcantilevers with thermally evaporated Au films, all but the fourth order mode [Fig. 6(b)] show only very small spectral shifts. The nature of the spectral response as a function of fractional Au coverage and its implications are currently being investigated by considering a mechanical model. The opposite frequency shifts upon Hg exposure for the two kinds of Au films are believed to be due to the surface morphological differences between the as-deposited films, which we are presently investigating further. However, from Fig. 6 it is clear that microcantilevers with thermally evaporated Au have less shift in the thermal mode frequencies for any fractional coverage as compared to those with sputtered Au, and hence, show less sensitivity to Hg exposure. It is also seen from Fig. 6 that the best sensing response with both sputtered and thermally evaporated films is achieved when the Au coverage is 100%.

IV. CONCLUSIONS

As deposited sputtered and thermally evaporated Au films have been studied for Hg response with microcantilevers. It is seen that although the bending direction of the microcantilevers with the two kinds of Au films is the same, the deflection response for thermally evaporated Au films after Hg exposure tends to decay. Also one needs to use the fourth order mode for studying surface interaction of Hg with thermally evaporated Au films. On the other hand, with sputtered Au films all but the first order thermal mode show reasonable response to Hg. With thermal spectral studies of microcantilevers having various Au coverage it is found that 100% Au coverage gives the best response to Hg sensing for both kinds of Au films. The spectral response of sputtered film is found to be larger than that of the thermally evaporated Au films for all the fractional Au coverages studied here.

ACKNOWLEDGMENTS

This work was supported in part by DARPA Grant No. N66001-01-1-8933, NSF Grant No. ECS-0602261, and NASA EPSCoR Grant No. NCC5-580.

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