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Selectivity of chemical sensors based on micro-cantilevers coated with thin polymer films

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Abstract

In an effort to impart selectivity to chemical sensors based on micro-machined silicon cantilevers, thin films of polymeric chromatographic stationary phases (SP-2340 and OV-25) were applied to one side of the cantilever surface using a spin coating procedure. These coatings influenced the response of the micro-cantilever to vapor phase test analytes of varying chemical compositions. For the SP-2340 coated micro-cantilevers, the effects of both polymeric film thickness and cantilever structure thickness on response characteristics were investigated. Sensitivity improved as both film thickness and cantilever leg thickness were decreased. The selectivity, as indicated by differences in relative responses to the test analytes, were different for the two phases which differed significantly in polarity. The SP-2340 coated cantilevers exhibited response characteristics that are fairly similar to that expected for adsorption of the test analytes onto silica. Responses are shown to be proportional to analyte concentration. Response characteristics are shown to be consistent with predictions based on a gas chromatographic stationary phase classification scheme. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Micro-cantilevers; Chemical sensors; Thin polymer films

1. Introduction

Recently, there has been an increasing interest in the development of miniature chemical sensors using micro-cantilevers (MCs) such as those commonly used in scanning force microscopy (SFM). MCs have been used in the past for a variety of physical and chemical applications. For example, bimaterial MCs have been used as miniature physical sensors for infrared radiation detection and temperature measurements [1–5] and as chemical sensors [6–15].

MC chemical sensors have the potential to offer a considerable increase in chemical sensitivity; over two orders of magnitude compared to other currently available chemical sensors [16,17] such as quartz crystal microbalances (QCM) [18,19], surface acoustic wave (SAW) [20–22], acoustic plate mode (APM) devices [22], chemiresitors [16,17], and flexural plate wave oscillators (FPW) [22].

A distinct advantage of the MC chemical sensors is the relatively small size of the sensing element; MCs used in the present work have an active area ($\sim 10^{-5}$ cm^2) that is about five orders of magnitude

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smaller than that of SAW devices; QCM and FPW devices (that have an active sensor area on the order of $\sim cm^2$). The MCs, along with SAW, QCM and FPW devices fall under the general category of mass load transducers or gravimetric sensors [16] and achieve sensing by monitoring the adsorption processes on the sensing element that result in mass changes. However, for any practical chemical sensing application, a chemically selective layer must be deposited on the gravimetric sensor which can also provide a reversible binding of the analyte to the coated surface and real-time monitoring. Differential response to multiple chemicals (selectivity) is a common problem with gravimetric sensing devices and attempts to overcome this shortcoming have focused on coating the surface of individual sensing elements with analyte-selective films to provide the specificity for the target species [18,23–25].

In order to enhance the sensitivity of an MC sensor for a particular analyte, a variety of coatings have been applied to the cantilever surfaces. Phosphoric acid and bovine skin gelatin have been applied to the tips of cantilevers to detect humidity [7]. Gold-coated cantilevers were used to detect Hg vapor [8] and alkanethiols [11]. A polymethylmethacrylate coating was used to detect short-chain primary alcohols [13]. Polydimethylsiloxane was applied to the tips of cantilevers to adsorb volatile organic compounds [12]. These sensors have proven to be very sensitive with mass resolution down to the pg range. However, the selectivity of these sensors has not been thoroughly investigated.

In the present studies, we investigated Si-MCs coated with thin films of polymeric chromatographic stationary phases (SP-2340 and OV-25). The Si-MCs were similar to those commonly used in SFM. We investigated how a thin polar polymer coating of poly(bis-cyanopropylsiloxane) (SP-2340) affects both the sensitivity and selectivity of a microcantilever to analytes that vary greatly in their mode of interaction with coated and uncoated sides of an MC. The effects of film thickness and leg thickness to sensitivity and selectivity are investigated. An MC with a relatively nonpolar coating of poly(phenyl-methyl dimethylsiloxane) (OV-25) was compared to the polar coating. We also used focused ion beam milling to modify the existing MCs and optimize their geometric characteristics.

Both the resonance frequency and bending of MCs have been found to vary reproducibly and sensitively as a consequence of adsorption of analytes on their surfaces [7–9,26]. In the present studies, we utilized stress-induced changes in the bending of Si-MCs [7–10,26–32] coated with selective chemical layers. When a specific analyte is adsorbed on the surface coating, an additional differential surface stress ($\Delta s = s_c - s_{si}$, where s_c and s_{si} are the stresses on the coated and uncoated Si surfaces) is induced due to the additional mass and change in the spring constant. This in turn results in changes in the resonance frequency and bending of the microcantilever that can be measured very sensitively using optical, piezoresistive or capacitive detection means [26] but in the present work we used an optical detection technique. The bending, z, depends linearly on differential surface stress, Δs and is given by [5,33]

$$z = \frac{3l}{t_1 + t_2}\left[\frac{1 + (t_1/t_2)}{3(1 + t_1 + t_2)^2 + (1 + t_1E_1/t_2E_2) \times (t_1^2/t_2^2 + t_2E_2/t_1E_1)}\right] \times \frac{\Delta s}{E^*} \quad (1)$$

where t_1 and t_2 are the thickness of the coating and MC substrate, l is the MC length, E_1 and E_2 are the Young's moduli of the coating and MC, and $E^*[= E_1E_2/(E_1 + E_2)]$ is the effective Young's modulus of the coated MC.

2. Experimental

Thin films of gas chromatography stationary phases (SP-2340 and OV-25, Supelco Co., Bellefonte, PA, USA) were deposited on micro-machined, V-shaped, silicon cantilevers (Park Scientific Instruments, Sunnyvale, CA, USA). The 600 nm thick silicon cantilevers had a 120 μm height, 90 μm base, and legs with a width of 26 μm. As supplied, the MCs had a 50 nm coating of gold on one side. In some cases, the gold coating was removed using aqua regia (75% HNO_3, 25% HCl) or the MC was cleaned with piranha solution (75% H_2SO_4, 25% H_2O_2). Thin films were cast on the MCs using a spin-coating procedure. SP-2340 dissolved in acetone (350 μl) was deposited

on a MC spinning on a Teflon mount at 400 rpm during a 10 s time window. After spinning at 400 rpm for 10 s, the spinning rate was increased at 30,000 rpm/s to a final spin rate of 4000 rpm and maintained at that spinning rate for 2 min. After the spin coating process, coated MCs were placed in an oven at 70°C for at least 5 h. Control of film thickness was achieved by varying the concentration of the SP-2340 solution. SP-2340 solutions ranged from 0.03 to 3 wt.% resulting in films from 50 to 500 nm thick. Films of OV-25 were cast in the same manner except this polymer was dissolved in methylene chloride.

Film thickness was measured by profilometry (Dektak 8000, Veeco/Sloan Technology, Santa Barbara, CA, USA) on films cast under the conditions described above on silicon wafers. The flexibility of the MCs prevented us from dragging the profilometer stylus across the MC surface. Therefore, film thickness measured on the wafer was used as an estimate of the thickness on the MC. The validity of this estimation of thickness was confirmed for one spin coated MC using changes in the resonance frequency of the coated cantilevers. The mounted MC was excited near its resonance frequency (35 kHz) by a piezoelectric speaker. Detection of the resonance amplitude was accomplished with a lock-in amplifier as the excitation frequency was swept across the resonance frequency range. The difference in the resonance frequency between the coated (f_i) and uncoated (f_f) MC is related to the change in the mass of the resonating structure (Δm), assuming the spring constant (k) remains constant, through

$$\Delta m = \frac{k}{0.72\,\pi^2}\left(\frac{1}{f_f^2} - \frac{1}{f_i^2}\right) \tag{2}$$

The thickness of the layer can then be calculated from the density of the phase and the geometric area of the coated surface. Thickness determined in this fashion agreed for a MC coated with a 0.6% solution of SP-2340 was ~150 nm whereas the value from a plot of thickness obtained by profilimetry versus % SP-2340 was ~140 nm.

Because both sides of the cantilever were coated during the film-casting process, the polymeric phase was removed from one side of the cantilever legs with a focused ion beam (FIB) mill (FIB 200, FEI Co., Hillsboro, OR, USA). The FIB was used to remove unwanted polymeric phase and also to thin the cantilever legs. Typical conditions for the FIB were a 600 nm aperture resulting in a beam current of approximately 11,500 pA and a dwell time of 1 ms with 25% overlap. The depth of ion etching was calibrated by determining the time required to etch a square hole completely through a 600 nm thick silicon cantilever. The depth of etching over a specified time can be determined by considering the difference in areas of the calibration hole and the sample. Fig. 1 shows a MC in which the thin film was removed from one side of one of the legs. Before the cantilever was used as a sensor, the thin film was removed from the same side of the second leg.

In an effort to enhance the sensitivity of the cantilever to gaseous analytes, the entire surface of one side of the cantilever was coated with polymer rather than only the tip. In this work, the deflection of the cantilever was used to detect the presence of absorbed analytes. By coating the entire surface of the cantilever legs, absorption-induced differential stress (i.e. $\Delta s_c - \Delta s_{si}$) is enhanced [9].

MCs were mounted in an optical system similar to that used for atomic force microscopy. The physical arrangement of the optical setup is shown in Fig. 2. A 5 mW diode laser (Coherent Laser Corp., Auburn, CA, USA) operating at 635 nm was spatially filtered and focused onto the triangular pad at the tip of the cantilever using a video microscope to visualize the process. The reflected beam was focused using a bi-convex lens onto a quad-cell, position-sensitive detector built in house [34]. Deflection of the cantilever was measured using the output of the position-sensitive detector that corresponded to vertical beam deflection. The amplified voltage from the position-sensitive detector was monitored using a lock-in amplifier (Stanford Research Systems, Sunnyvale, CA, USA).

A flow cell was mounted over the cantilever to allow flow of gaseous analytes across the MC surfaces. A constant flow of ultra high purity nitrogen of 1.5 ml/min. was achieved using a digital mass flow controller (MKS Instruments, Inc., Andover, MA, USA). Nitrogen flow was directed first through a fixed loop sample injection valve (Model 5020, Rheodyne, Inc., Cotati, CA, USA) that had a volume of 1.0 ml and then through the MC flow cell. This configuration allowed reproducible injections of gaseous analytes under controlled nitrogen flow. The volume

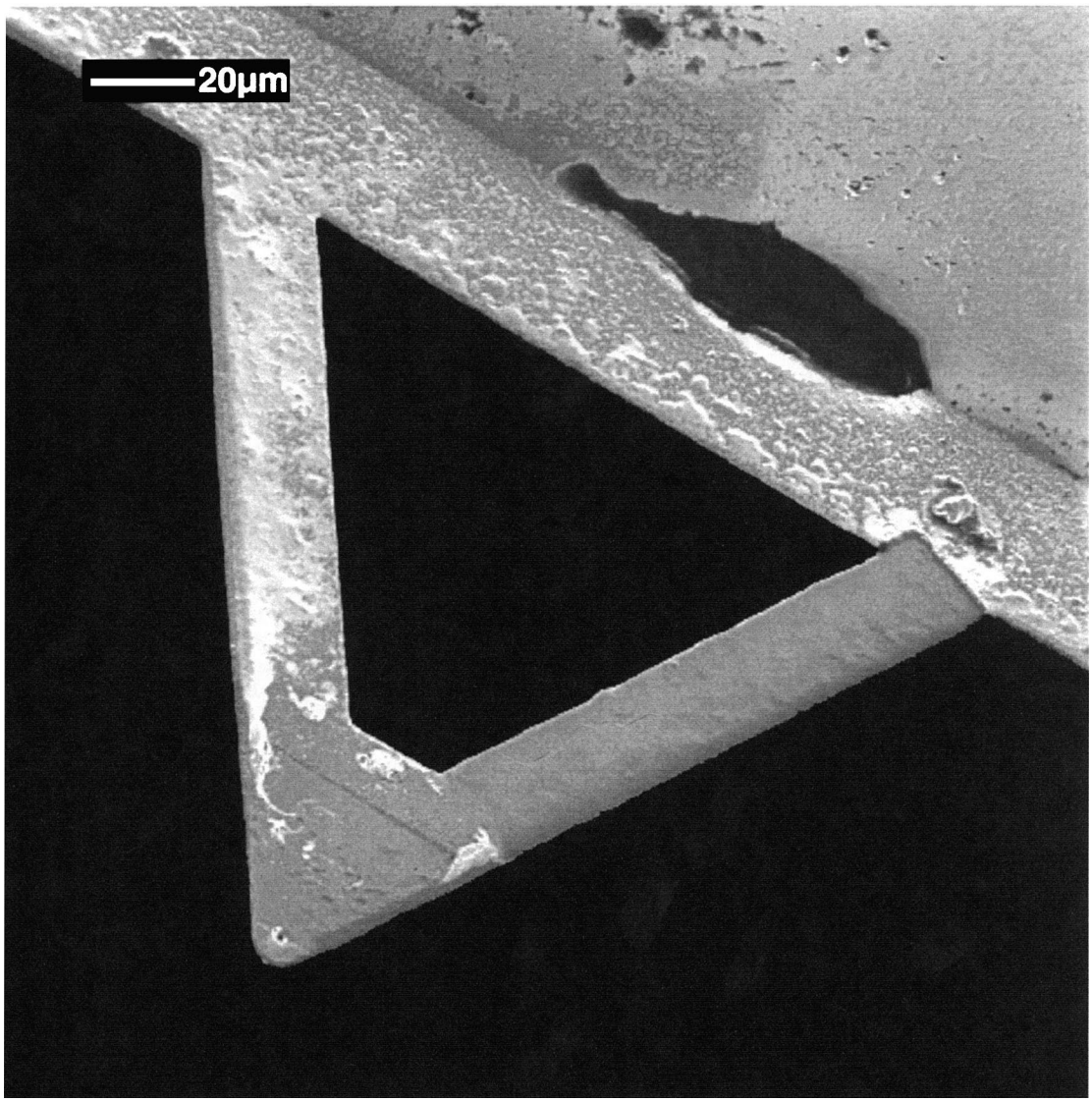

Fig. 1. V-shaped cantilever with a thin film of SP-2340 on one of the legs. A FIB mill was used to remove SP-2340 from the other leg. A film of SP-2340 coats the entire surface of the legs on the opposite side of this cantilever.

of the flow cell containing the MC is approximately 3 ml, consequently significant dilution of the injected analyte vapor is experienced.

Headspace above analyte solutions was developed in 40 ml headspace vials fitted with Teflon septa. To remove air and water vapor from the samples, the vial headspace was purged with nitrogen prior to development of analyte headspace. This was necessary to insure that the analyte was the only difference between the flowing nitrogen stream and the injected headspace sample. Sampling and subsequent injection into the fixed loop injector was accomplished using a gas-tight syringe. For preparation of different analyte vapor concentrations, a measured volume of headspace was diluted in nitrogen.

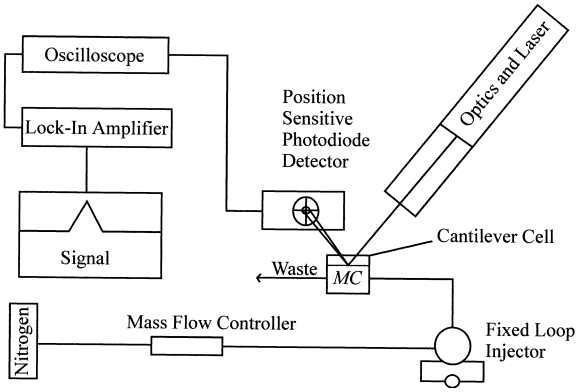

Fig. 2. Depiction of optical and analyte delivery systems.

3. Results and discussion

A primary goal of this work was to enhance response and impart selectivity to MC chemical detection systems by coating cantilevers with thin films of gas chromatographic stationary phases. In addition, studies of the effects of film and cantilever thickness on sensor performance were also conducted. The thin films chosen for this work were selected to create a sensor that would be capable of distinguishing analytes in a general sense based on polarity. The following analytes, all monitored in the vapor phase, and potential modes of molecular interaction with the films were employed: pentane (very non-polar compound, dispersive interactions only), toluene (aromatic system with high polarizability), aniline (aromatic weakly basic compound with H-bonding capabilities), ethanol (very weakly acidic compound with strong H-bonded characteristics), methylene chloride (modest dipole moment compound with weak H-bonding acceptor characteristics), and H_2O. Sensors were evaluated based on responses to this set of test analytes.

Fig. 3 compares the response of three different Si-MCs to the test analytes. The three cantilevers were the same shape and dimensions but their surfaces were modified in different manners. One of the cantilevers (Fig. 3, Trace A) was treated with aqua regia to remove the thin gold layer, leaving nearly identical Si surfaces on both sides of the MC. Because deflection depends upon the difference in the analyte-induced

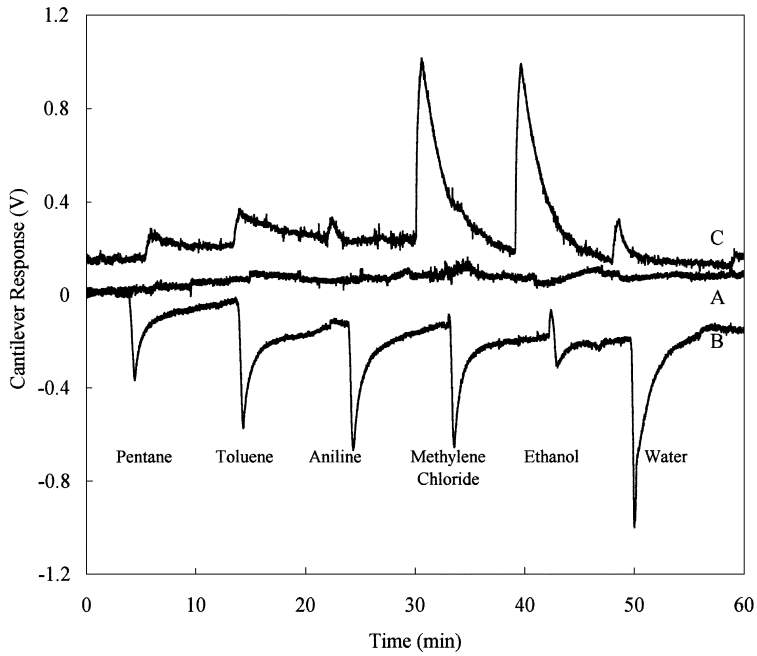

Fig. 3. Deflection responses of coated and uncoated MCs upon exposure to gaseous analytes. Trace A: Aqua regia-treated Si-MC. Trace B: Si-MC with gold removed by a FIB. Trace C: SP-2340 coated Si-MC with gold removed by a FIB.

stress on either side of the MC legs, this particular cantilever shows little response to the analytes.

The second MC was first cleaned in piranha solution. Then the gold layer was removed from the surface of one side of the legs via FIB milling. The resulting MC had legs that were composed solely of silicon, but the surfaces of the legs were prepared differently. The fact that significant bending occurs upon exposure to chemical vapors (Fig. 3, Trace B) indicates that the stress induced on the legs by analyte adsorption is different on either side of this cantilever. Assuming that the stress is expansive in nature, the direction of the bending (as indicated by the direction of the signal) indicates that the expansive stress on the FIB modified side is greater than that on the untreated side. It is well known that silicon can develop an overlayer of silicon oxide when exposed to air [35]. It is possible that FIB treatment alters the chemical nature and roughness of the silicon surface.

The third MC was cleaned with piranha solution, and then spin coated to produce a 150 nm thick film of SP-2340. SP-2340 is a very polar gas chromatographic stationary phase (100% cyano) with an average McReynolds constant, ΔI_{ave}, of 736 (by contrast OV-25, a 25% phenyl and 75% methyl phase, (see below) has an ΔI_{ave} of only 235). Any coating that ended up on the bottom of the cantilever surface was removed using FIB milling (see Fig. 1). The thin layer of gold was also removed from the bottom of the cantilever surface at this time. With the exception of the thin stationary phase coating, the resulting MC was identical to the uncoated cantilever that produced Trace B in Fig. 3. The response of this coated cantilever to the test analytes is shown as Trace C in the figure. The presence of the thin film caused the MC to deflect in the opposite direction of the uncoated MC when exposed to the same chemical vapors. In order for the signal to change direction, the polymeric phase must first counter the stress on the side cleaned by the ion beam, then exert an even greater stress to cause it to bend in the opposite direction. The direction of cantilever deflection indicates that the expansive stress induced by absorption of vapors on the coated side is greater than adsorption on the uncoated side. The change in deflection direction resulting from the presence of the thin polymeric coating shows that we have been able to dramatically modify response characteristics via the coating procedure.

Both coated and uncoated MCs (Fig. 3, Traces B and C) bend shortly after injection of analyte. After a sharp increase, the signal begins to decrease slowly owing to desorption of analyte from the cantilever surface under continuous nitrogen flow. The return of the MC to its original position after exposure is important because it shows that the absorption is reversible and allows the sensor to be reused. It should be possible to alter the dynamics of the MC response via ambient or resistively-induced temperature changes.

The responses of each MC sensor to analytes of different polarity and molecular characteristics are quite different. Because the gaseous analytes used in this study have different vapor pressures, Table 1 presents the responses normalized to the known room temperature, 1 atm, and vapor pressures of the analytes. Since the injected analyte headspace vapors were developed in a fixed volume container for which the vapor pressure increases beyond 1 atm, this procedure should not be taken as a strict normalization to the relative concentrations of the analytes in their respective headspaces. However, we feel this approach

Table 1
Response characteristics of MC normalized to vapor pressure

Analyte	Normalized response factor (maximum signal/vapor pressure) V/atm			Selectivity factor (response relative to pentane)	
	Uncoated	SP-2340 coated	Δ	Uncoated	SP-2340 coated
Pentane	−0.54	0.15	0.69	1	1
Toluene	−15	4.6	20	27	31
Aniline	−690	120	810	1300	820
Methylene chloride	−1.0	1.8	2.8	1.8	12
Ethanol	−1.6	11	13	3.0	75
Water	−26	5.5	31	48	37

is adequate for initial illustrations of coating-induced MC response and selectivity effects. The selectivity factors presented in Table 1 are all relative to pentane and calculated by dividing each response factor by the response factor of pentane. Because the magnitude of the signal depends on many variables present in the optical arrangement, it is difficult to compare absolute signal magnitude between two separate MCs. The signals in this work are reported in voltage output of the detector. In general, this has not been converted to actual cantilever displacement since the focus of this work was selectivity not sensitivity. Moreover, the volts to displacement conversion would vary as the optical alignment (e.g. distance between MC and position sensitive detector) was slightly altered during the course of these experiments. However, for a typical optical configuration (not optimized for detectability) the conversion is roughly 80 nm/V based on a relationship presented in an earlier report [10]. The high frequency noise that appears in the traces in Figs. 3, 6, 7, and 8 could be diminished greatly with filtering. The baseline drifts in those traces that exhibit time frames comparable to the transient signals, hence limit detection of injected analyte bands, correspond to approximately 2 nm deflections (e.g. see small negative baseline disturbance at 47 min in Trace B in Fig. 3).

Table 1 shows a modest correlation between response factors for SP-2340 coated and uncoated MC sensors. This may reflect the similarity between the adsorption strengths of the analytes onto a silicon oxide surface and absorption into a thin coating of the highly polar SP-2340 phase. The deviation in this trend for water is also consistent with the large adsorption strength of H_2O on silica. The Δ values in the table indicate the overall stress due to interaction of an analyte with the polymeric phase. Aside from selectivity effects, it is clear that the polymeric phase enhances the overall response to the analytes; in all cases the analyte induced expansion on the polymeric side overcomes the negative uncoated response.

The thickness of the film also influenced the response of the MC. Fig. 4 shows the signal-to-noise levels of silicon cantilevers in which the gold was removed from one side of the legs by ion bombardment. Each of the cantilevers used in this diagram were prepared in the same fashion except for the thickness of the SP-2340 film. All of the MCs had legs that were

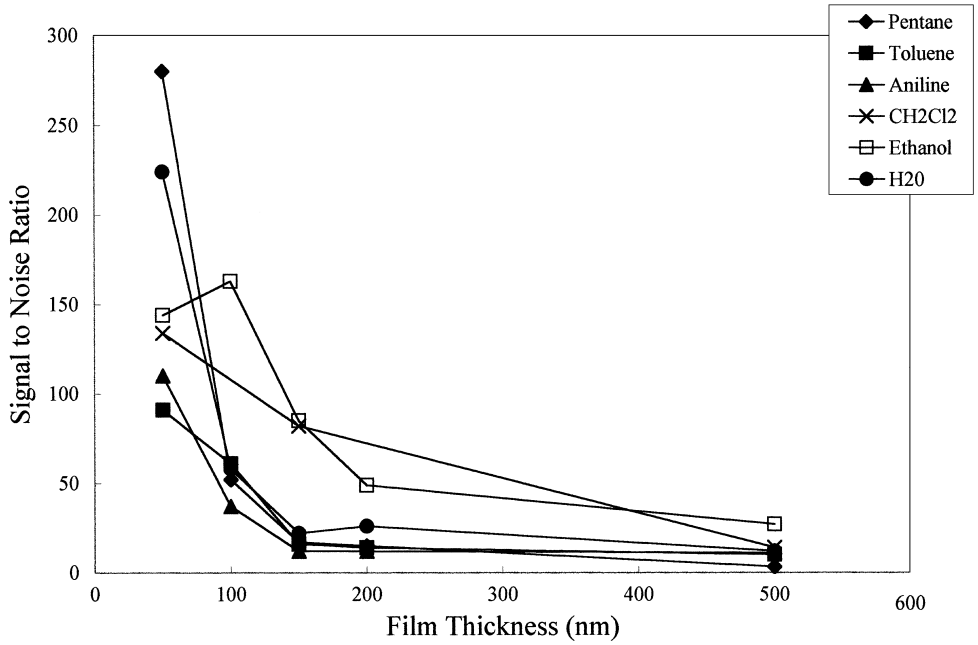

Fig. 4. Effect of SP-2340 film thickness on signal-to-noise ratio for pentane, toluene, aniline, methylene chloride, ethanol and water.

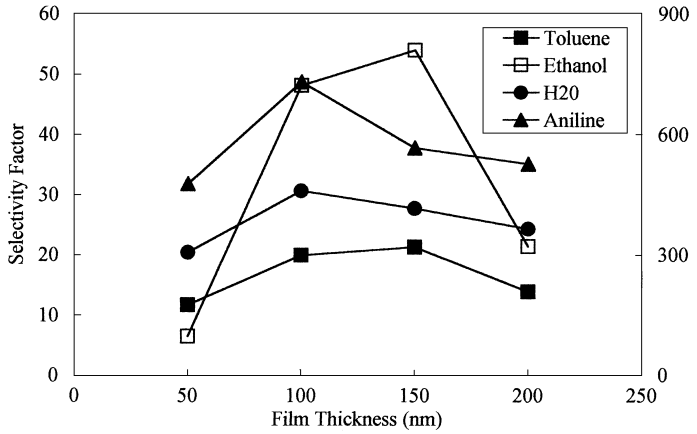

Fig. 5. Effect of SP-2340 film thickness on the selectivity factor of the coated sensors. Selectivity factor was determined relative to pentane.

360 nm thick, but the SP-2340 films on one side of the legs ranged from 50 to 500 nm thick. It is apparent that SP-2340 film thickness affects both sensitivity and selectivity. A quantitative analysis of the sensitivity and selectivity are presented in Figs. 4 and 5 , respectively. As film thickness increases, signal-to-noise ratio decreases quite dramatically (see Fig. 4). For ethanol, the signal-to-noise ratio increases then sharply decreases above a 100 nm film. The low signal-to-noise ratio at large film thicknesses could be due to the additional mass on the cantilever legs restricting the bending of the cantilever, effectively altering t_1 and/or E_1 in Eq. (1). Alternately, there may be a 'dilution' of the stress since the mole fraction of analyte in the coated film resulting from injection of a fixed amount decreases as the amount of coating is increased.

The selectivity factors plotted in Fig. 5 are calculated by dividing the response factor of each analyte by the response factor of pentane. The apparent change in selectivity with film thickness is caused by the differential nature of the response. The stress on the uncoated side of the SP-2340 MC is probably constant as film thickness is altered. However, the coated side response is sensitive to film thickness. Hence, as thick-

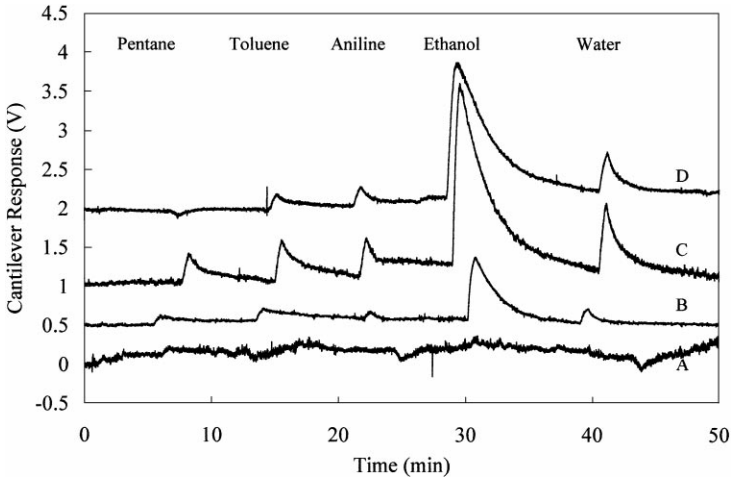

Fig. 6. Effect of cantilever leg thickness on SP-2340 coated MC response. The legs of the same cantilever were thinned incrementally: A. 480 nm, B. 360 nm, C. 300 nm, D. 260 nm.

ness is varied the net response to the analytes varies depending on the constant values of s_{si} and changes in s_c (see Eq. (1)).

Another means to alter the response of the MC sensor is to modify the thickness of the cantilever legs. The commercial cantilevers used had a leg thickness of 600 nm. By FIB milling it is possible to etch the cantilever surface in a very controlled fashion. Fig. 6 depicts the response of a single cantilever sensor with a 150 nm thick film of SP-2340. Each trace shows the response of the cantilever after successive thinning of the legs with the FIB mill. The 480 nm thick legs (Fig. 6, Trace A) show little response when exposed to analyte vapors. The relatively thick legs are not flexible enough to allow adequate bending of the cantilever to provide a reasonable signal-to-noise ratio. As the legs were thinned (Traces B, C and D), signal levels are increased. The differences in the baseline noise shown in Fig. 6 may be a result (at least in part) of differences in optical alignment rather than changes in sensitivity. Curiously, the cantilever with 260 nm thick legs actually bends in opposite directions when exposed to pentane, indicating again that the differential nature of MC response can create unique changes in selectivity. Cantilevers with legs thinner than 260 nm were extremely fragile.

MCs are also capable of providing quantitative information. By diluting analyte headspace with nitrogen in a gas-tight syringe, and passing the diluted vapor across the surface of a SP-2340 coated cantilever, we were able to calibrate the sensor for several analytes as shown in Fig. 7. The response of the cantilever increased with increasing analyte concentration.

By coating the cantilever surfaces with a variety of thin films it may be possible to impart a unique selectivity to individual MC elements in an array. A recent report of this approach involved coating eight MCs in an array with common polymers (e.g. polystyrene, polymethylmethacrylate, etc.) or mixtures of these polymers [14]. When creating such an array it is desirable to use coatings that respond differently for the analytes of interest. A potential advantage of using gas chromatographic phases as coatings, as reported herein, is that there is a wealth of information on the selectivity of these phases. This should facilitate the rational design of arrays containing elements exhibiting orthogonal response characteristics. We have investigated another gas chromatographic stationary phase, OV-25, as a cantilever coating to alter selectivity. Methods of classifying stationary phases often involve measuring the Kovat's indices of selected test compounds on columns prepared using the phase [36]. Characteristic constants are generated by subtracting indices determined for these test compounds using a column prepared with a reference stationary phase such as squalane. McReynolds constants are created in such a classification scheme [36]. The McReynolds constants for SP-2340 and OV-25 phases, expressed as percent of the average value for the test compounds, appear in Table 2. The higher the value (or

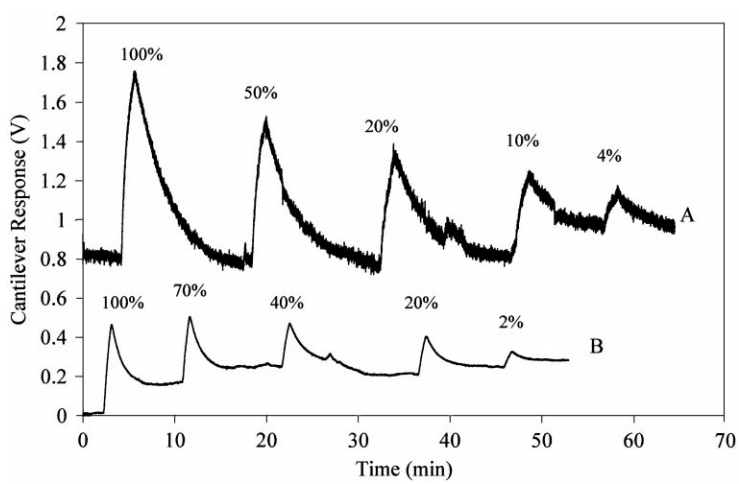

Fig. 7. Quantitative deflection responses of SP-2340 coated MC to gaseous ethanol (Trace A) and aniline (Trace B).

Table 2
Relative McReynolds constants[a]

Phase	Test compounds used in classification				
	Benzene (x')	Butanol (y')	2-Pentanone (z')	Nitropropane (u')	Pyridine (s')
OV-25	76	87	89	130	120
SP-2340	71	103	90	128	108

[a] % of ΔI_{ave} for the phase.

percent in Table 2) the greater the relative absorption strength of the test compound (or compounds with related structural features) for the phase.

Fig. 8 compares the responses of OV-25 and SP-2340 coated MCs to the test analyte vapors used in these studies. Although the response of a coated MC is governed by more than the absorption strength of the coating for the analyte, there are still trends in the responses seen in Fig. 8 that are consistent with the data in Table 2 and worth noting. Based on the possibility of similar molecular interactions, the responses of the test analytes methylene chloride, aniline, and ethanol might be expected to mimic the data presented in the table for 2-pentanone (z'), pyridine (s'), and butanol (y'), respectively. The z' values for the two phases are similar. However, the ratio of s' to z' is considerably larger for OV-25 than SP-2340. Similarly, the ratio of responses for the aromatic base aniline (s' mimic) to the dipole moment compound methylene chloride (z' mimic) is substantially greater for OV-25 than SP-2340. Conversely, the ratio of y' to z' is greater for SP-2340 than OV-25. In agreement, the ratio of responses of the alcohol ethanol (y' mimic) to methylene chloride is greater for SP-2340 than OV-25.

This work shows that depositing thin films of gas chromatographic-type polysiloxane phases on the surfaces of MCs dramatically influences sensitivity and selectivity toward analytes that possess different possible modes of molecular recognition. Moreover, common classification schemes such as McReynolds constants appear to have some value in predicting response characteristics and, hence, may aid in the rational design of MC elements in arrays. Film and MC leg thickness are shown to be important in optimizing MC performance. We believe that coated MCs exhibit

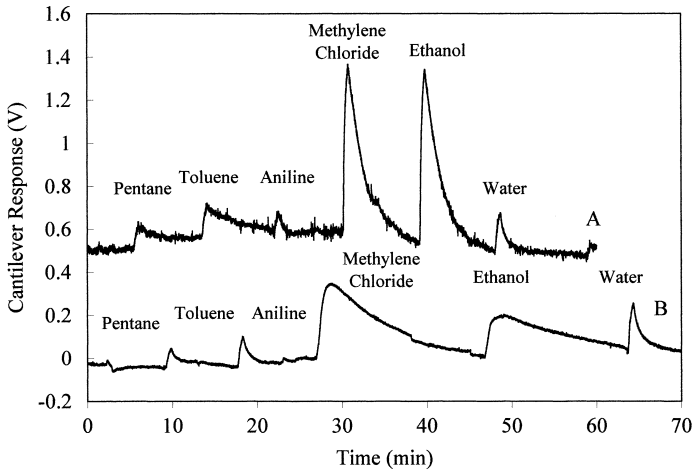

Fig. 8. Deflection responses of SP-2340 coated cantilever (Trace A) compared to OV-25 coated cantilever (Trace B) to vapor phase analytes.

significant potential as tools for qualitative and quantitative analysis.

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