

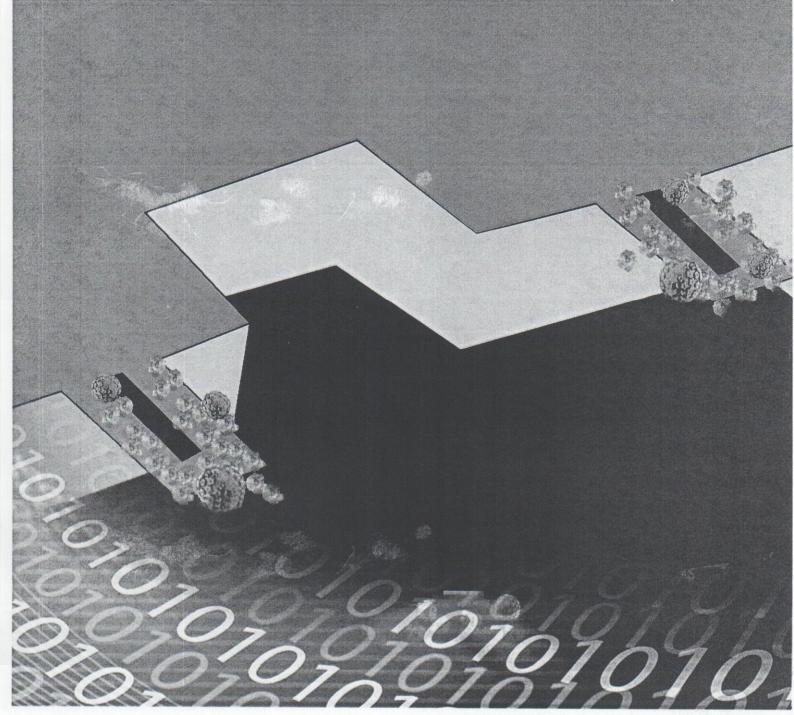


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# Investigation of a sensitive layer of polyaniline as coating for microcantilever as a nanosensor\*

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Abstract - In recent years, research on microelectromechanical systems in sensor devices has grown due to their potential for broad applications, great variety of designs, and simplicity. The objective of this study was to evaluate the use microcantilevers sensors (MCS) with a sensitive layer of conducting polymer (polyaniline doped with HCl) for sensing relative humidity (RH). The MCS surfaces were coated using spin-coating and drop-coating (small volumes deposition using thermoelastic probe TEProbe®) techniques. The deflection of the MCS at changes of RH was monitored by the optical lever principle, using an AFM (atomic force microscope) (Veeco, Dimension V). The MCS coating with the spin-coating technique had a relatively fast response and higher sensitivity (177 nm/% RH) than the drop-coating technique (26 nm/% RH) in a linear RH range (~40%). The excellent sensitivity and reversible nanomechanical bending of the nanosensors submitted at RH cycles may be due to the fast adsorption/desorption and also to the dissociation of water molecules adsorbed on the imine nitrogen center. This dissociation causes a migration of positive charge through the polymer, resulting in a shrinking or swelling of the polyaniline at different RH.

Index Terms - microcantilever sensor, sensitive layer, polyaniline, humidity, sensitivity.

### I. INTRODUCTION

In recent years, research on microelectromechanical systems (MEMS) in sensor devices has grown due to their potential for broad applications, great variety of designs, and simplicity. Microcantilever sensors are one type of MEMS with particularly high sensitivity. The sensor is made by depositing a chemically adsorbent film on the surface of the MCS [1]. When the film adsorbs or binds a target analyte, a differential surface stress develops on the film surface causing a perceptible deflection in the MCS that can be detected mechanically. The responses can be precisely detected using optical methods that are routinely used for AFM imaging technique. These types of sensors can be applied in many areas such as environmental control monitoring, chemical

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vapors [2], detection of humidity [1] and pH; pharmacological research; precision agriculture, biotechnology [3] and agribusiness, among others. The objective of this study was to develop a MCS with a chemically sensitive layer of conducting polymer (polyaniline in the emeraldine oxidation state doped with HCl) for detection of relative humidity (RH).

### II. CANTILEVER FUNCTIONALIZATION

The MCS was developed using a rectangular silicon microcantilever (350μm x 30μm x 0.5μm, thickness) with spring constant in the range 0.03-0.11N/m. A polyaniline (PANI) is obtained by interfacial polymerization method [4], when the monomer (aniline) was dissolved in dichloroethane and the oxidant (ammonium persulfate) was dissolved in HCl (1M) dopant acid solution.

The surface of the microcantilever was coated with polyaniline as a sensitive layer using *spin-coating* and *drop-coating* thermoelastic techniques. In the *spin-coating* technique the films were cast on the surface of the microcantilever using a spinner. After spinning of 500 rpm for 8.0 s, 3.0 µl of PANI solution was deposited in the microcantilever surface, the spinning rate was increased at 1000 rpm for 10 s and finally 3000 rpm maintained at spinning rate for 1min. By the *drop-coating* technique [5], PANI solution was placed into a capillary tube (thermoelastic probe®) and then the solution was compressed depositing a drop of 1.5 nL on the microcantilever surface. Both coated surfaces were dried and kept in a vacuum desiccator.

# III. SURFACE CHARACTERIZATION

Surface morphology and average roughness of coated and uncoated MCS were characterized with AFM, using a pyramidal silicon nitride tip attached to a cantilever with a

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spring constant of 0.03 N/m, in the tapping mode over a  $10\mu m \times 10\mu m$  scan area.

The topographic images showed a smooth morphology in the uncoated silicon MCS, with 2.02 (± 0.30) nm of roughness Rms. In the other hand, the coated MCS had a granular and uniform morphology, characteristic of the PANI film, showing a roughness of 152.0 (± 9.70) nm and 79.43 (± 7.80) nm on the coated surfaces by *drop-coating* or the *spin-coating* techniques, respectively (Fig.1).

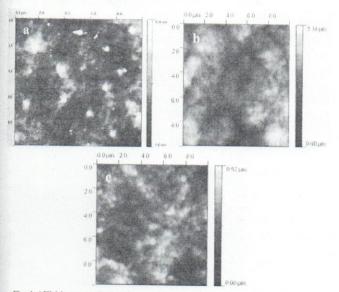


Fig. 1 AFM images of the microcantilever surface a) uncoated, b) coated by drop-coating and c) coated by spin-coating.

#### III. RESPONSE AT HUMIDITY

The bending response of the MCS was measured by monitoring the position of a laser beam reflected onto a four-quadrant photodiode of the atomic force microscope (AFM, Vecco Dimension V). The deflection measurement of the microcantilever sensor at humidity was carried in a closed chamber. The control of RH (%) in the chamber was obtained by passing dry nitrogen flow (flow mass controllers -Analog) at fixed total flow rate of 200 mL/min in a gas bubbler tube containing water.

The static deflection was a result of the absorption/desorption of water molecules into the sensitive layer of conducting polymer in both the organization and conformation of the polymer chains. The MCS coated with the *spin-coating* technique had a relatively fast response and higher sensitivity (177 nm/%RH) than the *drop-coating* technique (26 nm/%RH) in a linear RH (%) range (~40%) (Fig.2). Thus, the sensitivity obtained with the MCS coated with *spin-coating* technique was ~7 times higher than that obtained with other technique.

The uncoated MCS did not show a visible sensitivity to the RH (%), in this way it was found that the polyaniline is an excellent sensitive layer for detecting RH (%). In addition, the

roughness of the film structure also allows for easy vapor diffusion into and out of the sensitive layer, becoming the sensor reversible.

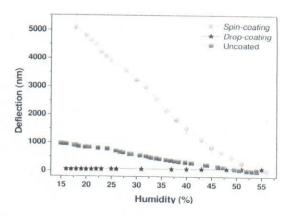


Fig. 2 The deflection vs. humidity of the uncoated MCS and coated MCS with polyaniline sensitive layer by the *spin-coating* and *drop-coating* techniques.

# IV. CONCLUSIONS

The results showed that MCS coated with polyaniline have high sensitivity at RH (%). It was possible observe a sensitivity ~7 times higher of the coated MCS with *spin-coating* compared with the *drop-coating* technique. The influence of the different coating technique has to be considered to developed new sensors. These MCS have a great potential for application, which could have important commercial applications.

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