A Comparison of Zeolites and Polymers used as sensitive layers in microcantilever-based gas sensors

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Zeolites as sensing layer

The specificity, adsorption and catalytic properties of zeolites, besides the possibility of its pores to host different ions, atoms, molecules and clusters have opened up numerous opportunities of zeolites as advanced nanomaterials, applied in micro-sensor, micro-separators and micro-reactors.

There are several examples where zeolites have been used as a sensitive layer over a cantilever to detect humidity [1] and certain gases like Freon [2]. In this work the use of other zeolitic materials more selective to a specific analyte has been proposed. To obtain this selective material, cation exchanges have been carried on. Increased selectivity to polar molecules using Co-Beta, zeolite with BEA structure exchanged with cobalt, has been verified [3] and it is used as a sensitive layer in this research. This type of zeolite accommodates hydrophilic molecules and big sized molecules in its nanostructure due to its excellent properties of pore size and polar behavior (specific sensors to detect explosive materials, nitrotoluene for instance).

The resonance frequency of microcantilevers is measured and the adsorbed mass of the sensed-gas is then calculated. The cantilevers are fabricated using SOI silicon wafers and Deep Reactive Ion Etching (DRIE) by IMS Laboratory [4]. Zeolites are directly deposited on the surface of the cantilever. An electromagnetic actuation is used to excite the movement and the deflection is measured with integrated piezo-resistive circuit.

In this work, a comparison of zeolites and polymers as sensing layers has been studied. Moreover the selectivity of Co-Beta zeolite to ethanol and nitrotoluene has been performed.

Zeolite versus polymers

Adsorption of ethanol has been tested using different sensing coating layers on the same type of cantilever based sensors. Table 1 summarizes the characteristics of these devices. In particular three standard classical polymers have been tested: PEUT, PECH and PIB. The results obtained with the sensitive layer of Co-Beta zeolites are shown as well.

When ethanol is sensed, the sensitivity normalized to the mass of sensing layer is for zeolite at least 3 times higher (in some cases more than 30 times) than polymers. In figure 1, the variation of the adsorbed ethanol per amount of sensitive layer is shown. Zeolite sensitivity is up to 25 times higher than the values obtained with PEUT polymer. The response time of the sensor was similar when zeolite was used as the sensitive layer instead of a polymer.

To analyze the versatiliy of zeolites as sensing layer, the selectivity of this material to detect nitro-derivates (explosives) has been studied. Adsorption measurements for ethanol and nitrotoluene have been carried out with a sensitive coating of 4.38 µg of zeolite Co-Beta, Figure 2 shows the obtained adsorption curves. In these results the sensitivity ratio obtained of nitrotoluene/etanol is 80, showing that zeolite Co-Beta has good selectivity for nitrotoluen.

Zeolites are shown to be good candidates as sensitive layers for gas detection. When compared to polymers, they offer better stability to vibrations and temperature, thus increasing the sensor lifetime. They will permit to locally heat the sensor allowing a quick absorption-desorption cycle. Therefore a very fast and re-usable sensor is obtained. Further more it has been shown that the normalized sensitivity is higher compared with several polymers. Finally, their specific selectivity is demonstrated and found to be good candidates as sensing material for explosives detection.

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Figures:

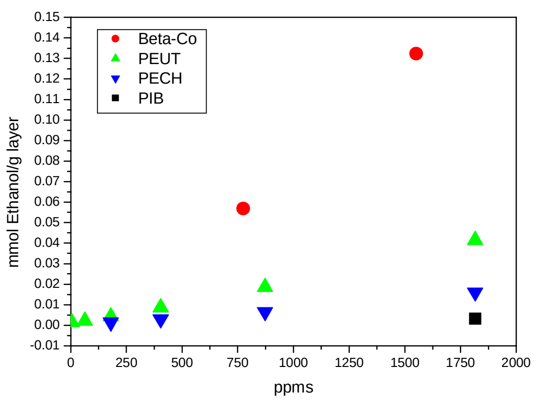

Figure 1: Comparison of ethanol adsorption in the four sensors.

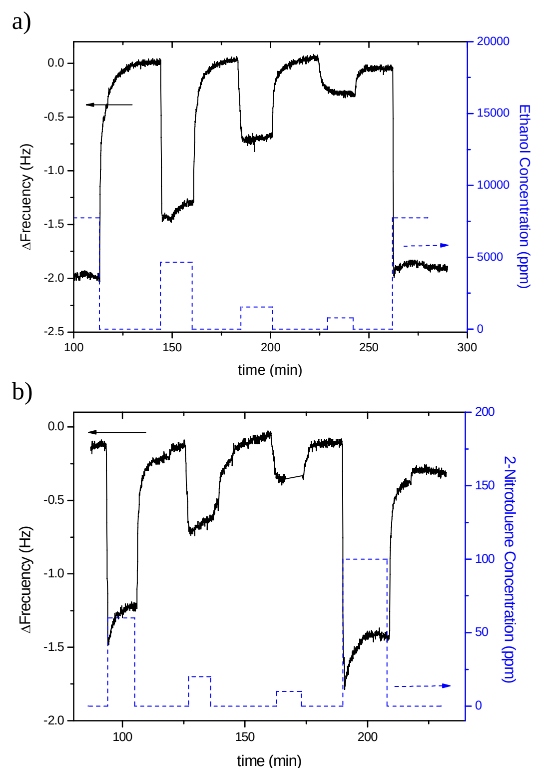

Figure 2. Response of the silicon microcantilever with 4.38 µg Co-Beta. a) Between 7000 ppm and 700 ppm of ethanol. b) Between 100 ppm and 10 ppm of 2-nitrotoluene

Table 1. Characteristics and properties of cantilevers and layers in ethanol atmosphere.

	Sensor A	**Sensor B**	**Sensor C**	**Sensor D**
Layer	PEUT	PECH	PIB	Beta-Co
Layer-mass	36.5µg	43.5µg	25.3µg	4.38µg
Resonance Frequency.	3061.7Hz	3099.5Hz	4786.2Hz	20289Hz
Quality factor	938	756	1027	375
Sensitivity [mHz/ppm]	0.144	0.026	0.011	0.343
Detection limit	95ppm	147ppm	526ppm	116ppm