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Vacuum Deposited Polyimide Layers as Humidity Sensors*

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Abstract. The polyimide layers are prepared by thermal vacuum co-evaporation of the precursors pyromellitic dianhydride (PMDA) and 4,4'-oxydianiline (ODA) and subsequent thermal treatment up to 250 or 300°C.

In this paper it is investigated the humidity sensing properties of polyimide layers (with thickness of 50 and 630 nm) by measuring the electrical resistance R [Ω] and the capacitance C [pF] as a function of the relative humidity RH [%]. The layers show very good characteristics as humidity sensors operating at room temperature – the electrical resistance changes by more than 4 decades and the capacitance – by 40% in the range of 20–100% RH.

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1 Introduction

Humidity sensors have been widely used in many areas of applications such as electrical appliances, consumer products [1], industrial process control [2], or scientific and medical equipment [3-5]. The principles of operation of these sensors are mainly based on resistive or capacitive characteristics of moisture sensitive materials such as ceramics, porous silicon and polymers [6]. Sensors of different type (based on change in dimensions of the sensing material, resistive, capacitive, cantilever sensors) use polyimide thin layer for water adsorption and desorption [7-9]. Polyimide exhibits excellent thermal stability, a low dielectric constant and low equilibrium moisture content. It is resistant to irradiation, mechanically strong and chemically stable in the presence of most common contaminants. Also, polyimide processing is fully compatible with standard electronic processing procedures, an important consideration for cost control.

The aim of this investigation is to study the properties of thin polyimide layers prepared by vacuum evaporation technique as resistive or capacitive humidity

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sensors working at room temperature. Following the response kinetics information about the sensing mechanism is obtained.

2 Experimental

The polyimide layers were formed on planetary rotating (30 rpm) substrates by vacuum co-deposition of ODA and PMDA (ratio 1:1) from two independent thermally heated Knudsen-type sources [11]. The thickness of the layers were 50 and 630 nm. After deposition the layers were thermally treated for 1 hour at 170°C and subsequently 1 hour at 250°C or 1 hour at 300°C. The surface of polyimide layers is smooth – without noticeable peculiar defects or granular structure [12]. Moreover, IR spectroscopy revealed, that the annealed layers are fully imidized and do not differ in composition [14].

For electrical characterization of the resistivity sensors interdigitated Au electrodes were vacuum deposited through masks on the top of the polyimide (electrode width 0.8 mm, distance between electrodes 1 mm). The samples were placed in holders having two Au pressure contacts. The construction of the capacitive sensor includes a substrate carrier, on the top of which, a sensitive polyimide layer is deposited. The technological sequence includes the following processes: semiconductor (silicon) substrate was covered with layer of SiO_2 with thickness of 0.3–0.4 μm by thermal oxidation. On the top of this structure an aluminum layer (with thickness 0.5–0.6 μm) was applied by thermal evaporation in vacuum. The aluminum layer was structured by photolithographic technique.

Sensor response to humidity, i.e. the change in electrical resistance (R [Ω]) or capacitance (C [pF]), was studied at room temperature varying the relative humidity (RH [%]) continuously or stepwise. The behaviour under gradually increased or decreased RH in the range 20–100% (R as a function of RH) was followed in a test chamber equipped with temperature and RH controllers. For studying the sensor response kinetics under step-like change in RH the sensor was put alternatively in small closed vessels with saturated solutions of $(NH_4)_2SO_4$ and NaOH (maintaining constant RH values of 82 and 6%, respectively) and the electrical resistance was monitored as a function of time. The measurement of R was performed with a multi-channel ohmmeter. The control of the ohmmeter, the acquisition and processing of the data were computerized. The computer programs were created using LabView software. The capacitance was measured with Agilent 34405A. The recovery time of the capacitive sensors was measured by introducing of dry air inside the camera, through dehydration filter, starting from 100% RH and decreasing the values to 10% RH.

3 Results and Discussion

Variation of the sensor output (in Ohms) as a function of RH [%] at room temperature, monitored under continuous rise or drop of RH, is shown in Figure 1. The response curve reveals a close exponential relationship between the sensor resistance and RH spanning over 4 decades of resistance when the relative environmental humidity varies in the range of 10–100% and is linearized by taking the logarithm of the resistance.

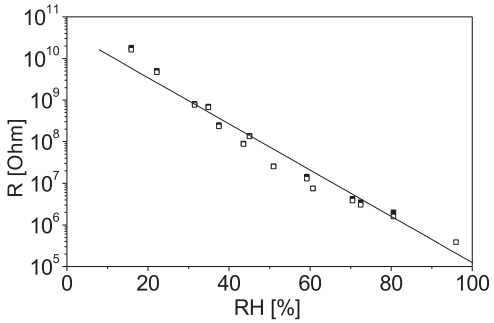

Figure 1: R [Ω] as a function of RH [%] in polyimide layer with $d \approx 50$ nm, thermally treated for 1 h at 170°C + 1 h at 300°C; glass substrate, Au comb-like electrodes on the top of the layer.

Figure 2 presents the results obtained with the same sample under step-like change in RH. The curve shows the sensor response kinetics at sharp drop or rise in relative humidity (6% and 82%). As it can be seen rising the relative humidity from 6 to 82% causes a very fast change in R. The recovery process goes slower, following an exponential dependence. Comparing Figures 1 and 2 it is also seen that both ways of changing the relative humidity lead to equal resistance variation of more than 4 orders of magnitude.

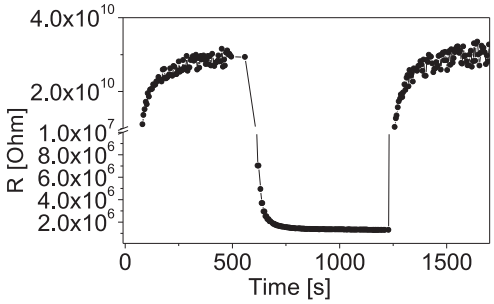

Figure 2: Resistance change with time at 6 and 82% RH of the same polyimide layer as in Figure 1.

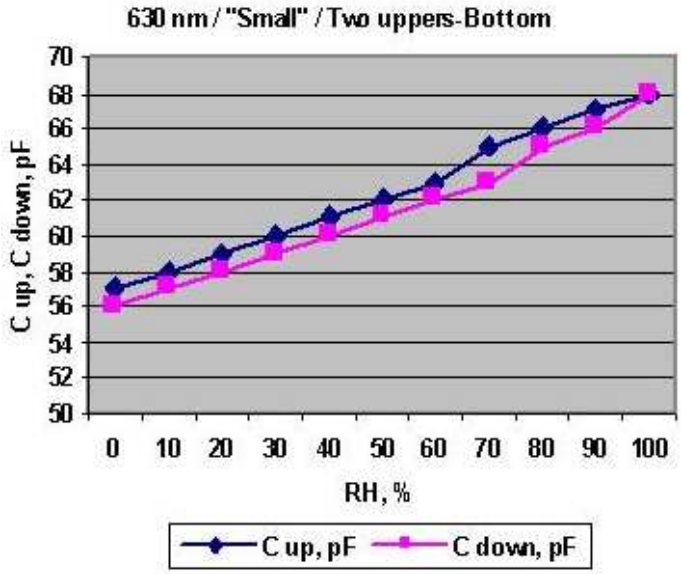

Figure 3: Capacitance change with RH in polyimide layer with $d \approx 630$ nm, thermally treated for 1 h at 170°C + 1 h at 250°C; Si substrate; Al comb-like electrodes on the top of the layer.

The fast humidity response of the polyimide layers could be related to dominating physical adsorption, i.e. only the layer surface is involved in the sorption-desorption process. The recovery process requires more time because it is not forced by heating or blowing through the vessel.

The capacitance was measured between two upper and bottom electrodes. Dependence of the capacitance as a function of relative humidity is shown in Figure 3. The dependence, between the capacitance and RH, in the case of the "volume" structures, is quiet linear. In general, the "volume" capacitance is in the range between 50 and 70 pF, i.e. the capacitance change is around 40 %, comparing the initial point.

The estimation of the hysteresis shows that it is very difficult to formulate some strong dependence. In both directions of humidity change, the values of the capacitance are similar and near.

The recovery time of the structures is between 9 and 12 sec. It is smaller in the cases of thin polyimide layer, which is logical, because of the smaller moisture adsorbing volume.

4 Conclusions

The results presented in this paper show that the thin layers prepared by vacuum co-deposition of ODA and PMDA from independent sources and subsequent thermal treatment reveal promising properties as resistive or capacitive humidity sensors working at room temperature. They meet the requirements for high sensitivity, short response time and low power consumption. The sensing characteristics of the polyimide layers could be attributed mainly to physical adsorption. The preparation method is compatible with the conventional microelectronic technology and allows the development of smart sensors. The obtained results give useful information for future developments of micromechanical humidity sensors for different applications. Together with the improvements of sensor parameters, some innovations could be done – for example, they could be implemented in MEMS devices.

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