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# PREPARATION AND HUMIDITY SENSING BEHAVIORS $TiO_2$ THIN FILMS

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**ABSTRACT:** Prototypes of humidity sensors were prepared by depositing metal alkoxide solutions stabilized with the acetylacetone onto $Al_20_3$ substrates with comb-type Au electrodes. The $TiO_2$ films have been prepared by dip-coating method. Heating in air to 400 °C produces continuous films, free of porosity, which appear as layers covering the substrates with no detectable grains. The humidity sensing electrical properties of the films were evaluated using electrochemical impedance spectroscopy (EIS) measurements. It was found that the sensor films an impedance change from $\sim 10^7$ $\Omega$ to $10^3$ $\Omega$ in the humidity range of 0,13 – 97,7 % RH. The reasons of humidity sensitive properties of the nanostructures $TiO_2$ films are discussed.

**KEY WORDS:** humidity sensors; $TiO_2$ films; sol-gel;

## 1. INTRODUCTION

Humidity is a constant environmental factor; therefore its accurate measurement and control are very important. Humidity sensors are generally required in areas including domestic electric appliances, the medical industry, the agricultural industry and the automobile industry. The constructive design of a good humidity sensor is a rather complicated topic, because high performance humidity sensors claim many requirements, including linear response, high sensitivity, fast response time, chemical and physical stability, wide operating humidity range and low cost. Materials that have been studied for this purpose include polymers, ceramics and composites, which have their own merits and specific conditions of application [1-3].
Amongst them, ceramic oxides have shown advantages in terms of thermal, physical and chemical stability, and mechanical strength [4, 5].

Commercial ceramic humidity sensors are based on changes in impedance of porous sintered oxides at different environmental humidities [6, 7]. The impedance changes of the ceramics with humidity are related to the water adsorption mechanism on the oxide surface. Very interesting results have been reported for porous $TiO_2$,-based humidity sensors [8, 9].

In this paper, results about the very high humidity sensitive electrical properties of sol-gel processed thin films are reported.

## 2. EXPERIMENT

The films are obtained by a dip-coating procedure. Titanium isopropylalkoxide (TIPT) was used as titania precursor. The matrix sol was prepared by mixing TIPT with isopropyl alcohol, distilled water, and an acid catalyst (HNO3 65%) and sol have been stabilized with the acetylacetone.

The composition is shown in Table 1.

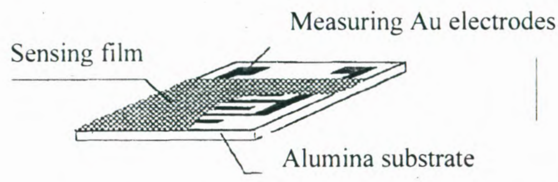


**Fig.1:** A schematic view of the sensor construction

The composite solution was coated on $Al_2O_3$ substrates with comb-type Au electrodes. Fig.1 shows a schematic view of the sensors. Humidity sensing film was then coated on the sensor substrate by either dip-coating.

The deposition is carried out at room temperature in air with a controlled withdrawal speed of 1 mm/s. The films obtained were dried at 80°C for 15 min., and then heated to 400°C for 30 min. EIS measurements were carried out at room temperature, in the frequency range from 1 kHz to 1 MHz, using a RCL Meter PM6306 Fluke and RH varied from 0,13 to 97,7%.

**Tab.1:** Composition of the composite film used to prepare humidity sensors

| Sample | $(izoC_3H_7O)_4Ti$ [$cm^3$] | $HNO_3$ [$cm^3$] | Acetylacetone [$cm^3$] | $izoC_3H_7OH$ [$cm^3$] | $H_2O$ [$cm^3$] |
|---|---|---|---|---|---|
| Ti 4Ac | 5,5 | 1 | 4 | 35,3 | 0,2 |

## 3. RESULTS AND DISCUSSIONS

Complex impedance plotting techniques can help in building the equivalent circuit model and then analyzing the mechanism of the humidity sensing material [10]. The most and common standard way to obtain an impedance spectrum is to measure the impedance directly in the frequency domain by applying a single voltage to the interface. In the impedance spectra, *Z'* is the real part of the impedance *Z* in the direction of real axis, and *Z''* is the imaginary part of *Z* along the imaginary axis. Fig.2 shows the komplex impedance spectra (Nyquist plot of impedance) of the Ti4Ac film at different relative humidity and their equivalent circuit. The semicircle through the origin of the Nyquist plot represents a typical equivalent circuit of a resistor and a capacitor connected in parallel. This indicates that the impedance of the Ti4Ac film is determined by the bulk properties of the film. Therefore, in Fig.2, R represents the resistivity of the Ti4Ac film and equals the magnitude of the diameter of the semicircle.

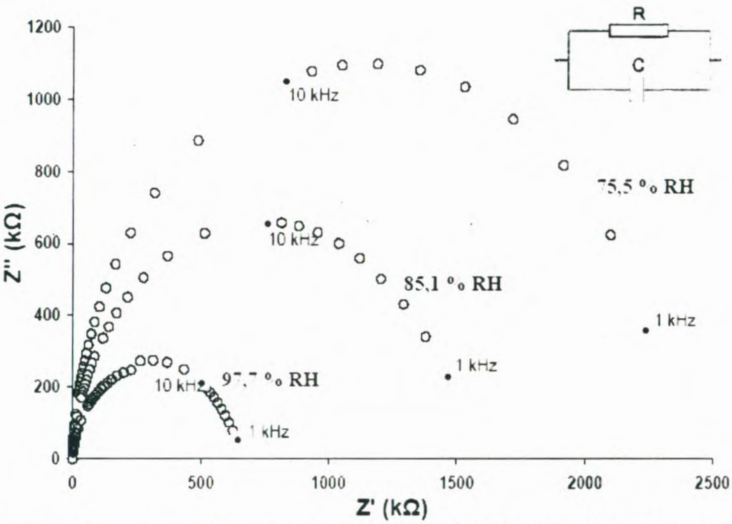


**Fig.2:** Impedance data as a function of RH for Ti4Ac film at 25°C, presented in the complex impedance plane plot

From Fig.2 it is clear that the response of the Ti4Ac film to RH is mostly caused from the change in R, the film resistivity. As has been reported by many authors, the migration of the proton through the adsorbed water molecule layer on the surface of the porous Ti4Ac film is the main process which determines the humidity sensing behavior of the Ti4Ac film [11].

The frequency dependencies of the impedance under various humidity conditions are shown in Fig.3. The result suggests that with increasing frequency the impedance value in independent of the humidity and is defined by the geometric capacitance of the sample. Therefore, measurements of the capacitance of the films were carried out at 1 kHz, because at this frequency the influences of the geometric capacitance of the sample are small.

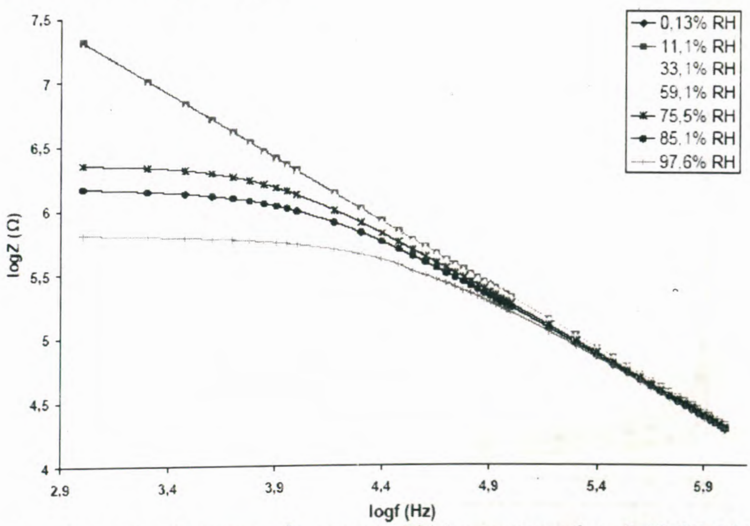


**Fig.3:** Impedance vs. frequency for sample Ti4Ac, measured on 0,13-97,6 % RH

It is well known that the dielectric constant of the material is in proportion to the capacitance, so the dielectric property of the material can be investigated by means of the capacitance property. Fig.4

depicts the change of the capacitance of Ti4Ac humidity sensor with respect to RH at a voltage of ac 1V (1 kHz).

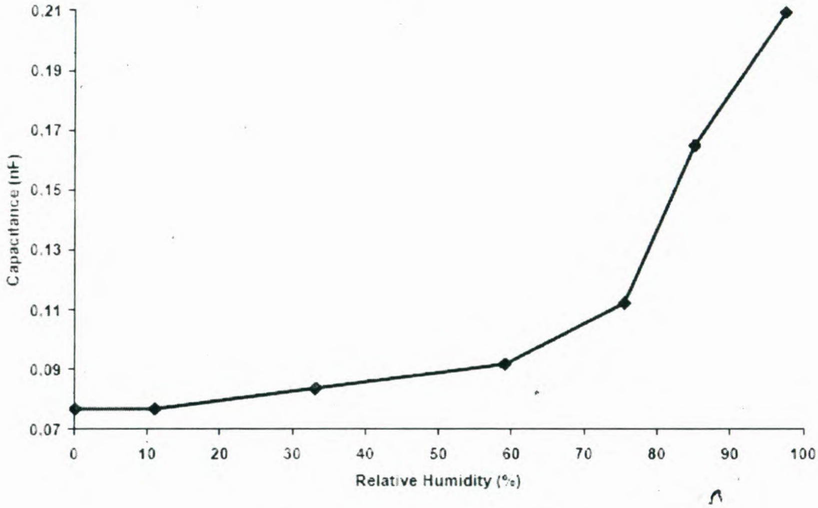


**Fig. 4:** Capacitance vs. RH curve of Ti4Ac sensor

The capacitance is very small and hardly changes in the low RH range and it increases fast in the high RH range. When RH is low, very little water vapor is adsorbed. When RH is high, the adsorbed water increases and the dielectrics property of the water makes the capacitance to increase quickly. The capacitance of the sensor is influenced by the frequency, as shown in Fig.5. We can see that there is no change when the frequency is very high ($10^6$ Hz), and that the lower the frequency is, the larger the change of capacitance is. And the higher the RH is, the larger the capacitance is in the low frequency (1 kHz).

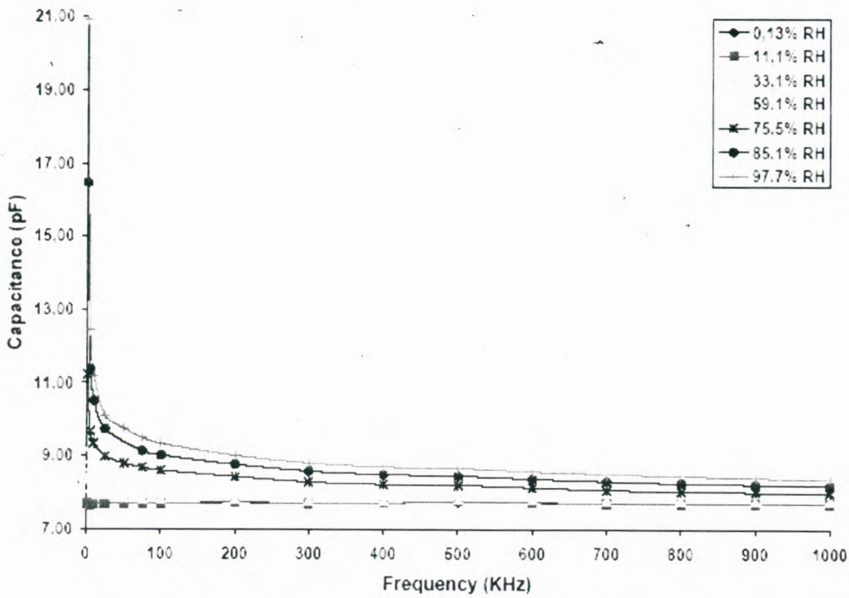


**Fig.5:** Capacitance vs. frequency properties Ti4Ac sensor

Because at a very high frequency the adsorbed water can not be polarized, the dielectric phenomenon does not appear, and the capacitance is determined by the geometric capacitance of the sample. At a low frequency, the capacitance increases as the RH increases, which is due to the polarization of the adsorbed water on the surface of the sensing film [12].

## 4. CONCLUSION

Ti4Ac humidity sensor possesses a good sensitivity at the RH range of 59,1–97,7%. The sol-gel technology is a very promising tool for the preparation of low-cost, high-quality, reliable ceramic films, that are highly sensitive to humidity. These materials are promising for use as active elements in integrated humidity sensors. Such materials, with novel RH-sensing mechanisms, are needed to overcome the existent limits of the ceramics used for commercial humidity sensors. The details of the sensing mechanism needs further investigation.

## 5. REFERENCES


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