# APPLICATION OF POROUS CERAMIC AS SOIL MOISTURE SENSOR IN CONTROLLED ENVIRONMENT

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**Abstract.** In this work, the behavior of ZrO<sub>2</sub>-TiO<sub>2</sub> porous ceramic as soil water content sensor element at different climatic conditions is presented. The analysis of the sensor element was carried out correlating the results of electrical properties, through the measurement of capacitance and impedance variation in function of the soil water content, with the microstructure of the ZrO<sub>2</sub>-TiO<sub>2</sub> ceramic. The ceramic sensor was studied in a sandy clay soil type at different climatic conditions characterized by temperature and relative humidity. The microstructural characterization of the ceramic sensor included scanning electron microscopy observations, X-ray diffraction patterns and pore size distribution using mercury porosimetry.

# Introduction

Ceramic materials to be used as soil water content sensing elements has become an important tool in various applications [1,2]. Among them, it can be pointed out its use in planning and operation of irrigation system aiming maximum production and better quality of crops, with an effective application of the water; another application is the use in draining systems, for urban and rural forced and free ducts, aiming to allow rain water to flow to prevent floods, and, finally, the use in monitoring areas with hillside landslide hazard, which has caused, mainly in the two last decades, accidents in several Brazil cities [3,4,5]. In this way, this work aims advanced ceramics improvement, specifically metal oxides, to be applied as sensing elements for soil water content monitoring.

Researchers of the Engineering and Science Group of Micro and Nanostructure Ceramics and Solid Surfaces (SUCERA) integrated to the Associate Laboratory of Sensors and Materials (LAS) of the Space Researches National Institute (INPE), have well established themselves, for the last 20 years, in the elaboration of diagnosis techniques, characterization and development of ceramic materials [6]. Since 1997, ceramics have been studied for utilization as air humidity sensing elements [7,8] and in the monitoring of the soil water content, mainly as sintered porous elements, manufactured by ceramic conventional processing to allow water molecules to flow freely through their microstructure and water condensation to occur in the capillarity of pores between grain surfaces, resulting in the increase of the medium sensor dielectric constant [1,2,7,8,9].

This work deals with the development of sensing elements, manufactured with zirconium oxide  $(ZrO_2)$  and titanium oxide  $(TiO_2)$  by the conventional ceramic processing, to be applied in the monitoring of soil water content.

## **Experimental procedure**

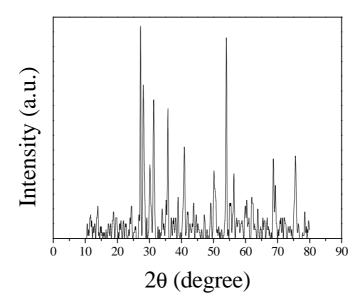
Porous ceramics were prepared through conventional ceramic processing, by using national raw materials. Ceramics sensor elements were fabricated from the suitable choice of ZrO<sub>2</sub> and TiO<sub>2</sub> powders, compacted and sintered with pre-established pressure and temperature.

ZrO<sub>2</sub> and TiO<sub>2</sub> pristine powders, in alcoholic suspension, were mechanically mixed up in a centrifugal mill in the rate 1:1 (in mass) and dried in a rotating evaporator. The powders mixture was circular tablet shaped by uniaxial pressing with 100 MPa. The ceramics sensor elements were characterized by x-ray diffraction, scanning electron microscopy, Hg porosimetry, besides electrical parameters measurements as capacitance and impedance in function of the soil water content, at controlled temperature and relative humidity environments. The electrical characteristics, capacitance and impedance measurements variation, were obtained in the ceramic sensing elements, through a RLT bridge, immersed in a soil that was characterized previously, with the simultaneous addition of controlled amount of water.

The electrical measurements (capacitance/impedance) started in dry soil and were performed up to its saturation, after seven measurements. These measurements were carried out at  $40\,^{\circ}\mathrm{C}$  and  $90\,\%$  relative humidity, in order to simulate actual climatic conditions underwent by the soil at countryside.

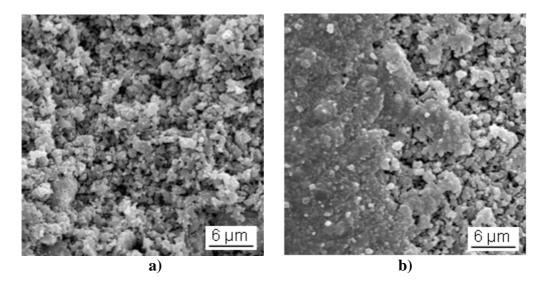
## Results and discussion

The results obtained are presented hereafter. Diffraction pattern in Figure 1, obtained from ceramic tablets sintered at 1100 °C shows the presence of two distinct crystalline phases:  $ZrO_2$  (monoclinic) and  $TiO_2$  (tetragonal).



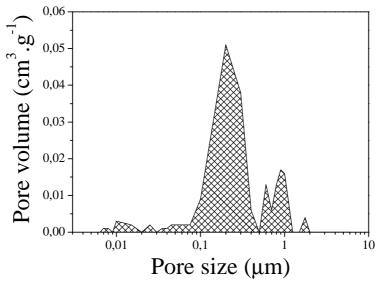
**Figure 1**. X-ray diffraction pattern. ZrO<sub>2</sub>-TiO<sub>2</sub> ceramic tablets sintered at 1100 °C.

The scanning electron micrographs of the  $ZrO_2$ -Ti $O_2$  ceramic tablets, in Figures 2a and 2b, show a porous microstructure formation. At the fracture surface (Fig. 2a) the particles necking with the resultant pores formation can be seen. At the top surface (Fig. 2b) one can see a dense region due to the compaction in the steel matrix.



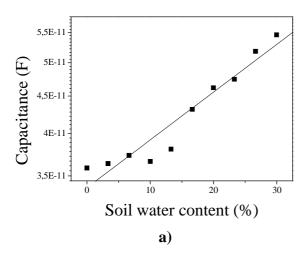
**Figure 2**. Fotomicrographs from the ZrO<sub>2</sub>-TiO<sub>2</sub> sensing elements sintered at 1100 °C. a) Fracture surface, b) Top surface.

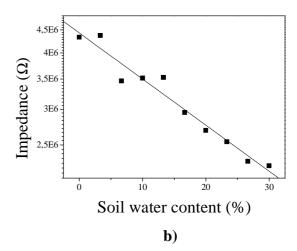
The graph of Figure 3, obtained through the Hg porosimetry technique, presents the pore size distribution of the  $ZrO_2$ - $TiO_2$  ceramic tablets, sintered at 1100 °C. It can be seen that the higher pore volumes are located in the ranges 0.1  $\mu$ m to 0.4  $\mu$ m and 0.5  $\mu$ m to 1.0  $\mu$ m. The pore size distribution versus pore volume curve corroborates the porous microstructure observed in the scanning electron micrographs of Figure 2.



**Figure 3**. The pore size distribution versus pore volume for ceramics sintered at 1100 °C.

The capacitance and impedance measurements performed on ceramic sensing elements in contact with increasing soil water contents (Fig. 4a and 4b), show a behavior coherent with the technical literature, i.e., a lineal trend, which evidences the  $ZrO_2$ - $TiO_2$  sensing elements capacity to quantify the soil water content. Concerning to the climatic conditions (temperature and relative humidity), at which the electrical measurements were performed, one can see that the soil did not saturated at the seventh measurement, as it was expected, but at the tenth measurement. This soil behavior shows the climatic conditions influence on the water content, which, so far, would be enough to saturate it.





**Figure 4.** a) Capacitance and b) impedance measurements of sensing elements sintered at 1100 °C under the climatic conditions of 40 °C and 90 % relative humidity.

### **Conclusion**

The x-ray diffraction, scanning electron microscopy and Hg porosimetry results showed that the suitable choice of the raw material and the tablets confection through conventional ceramic processing made possible to obtain a porous microstructure in a ZrO<sub>2</sub>-TiO<sub>2</sub> solid solution. Capacitance and impedance values in function of the increasing soil water content evidenced the capacity of ZrO<sub>2</sub>-TiO<sub>2</sub> sensing elements, sintered at 1100 °C, to quantify the water content of a soil that underwent the influence of climatic alterations.

#### References

- [1] R. M. Oliveira; M. C. A. Nono and G. P. Britto Filho. Influence of Nb<sub>2</sub>O<sub>5</sub> on the electrical properties of porous ZrO<sub>2</sub>-TiO<sub>2</sub> ceramic used as soil humidity sensor for environmental monitoring. Materials Science Forum, v. 591-593, p. 402-407, 2008.
- [2] R. M. Oliveira; M. C. A. Nono; C. Kuranaga and M. Wada. Development of ZrO<sub>2</sub>-TiO<sub>2</sub> porous ceramic as soil humidity sensor for application in environmental monitoring. Materials Science Forum, v. 530-531, p. 414-419, 2006.
- [3] Information on http://www.fao.org/nr/water.
- [4] A. M. S. Oliveira; S. N. A. Brito. Geologia de Engenharia. Associação Brasileira de Geologia de Engenharia, 1998.
- [5] A. L. C. Castro (Org.) *Manual de Planejamento em Defesa Civil*. Brasília: Ministério da Integração Nacional, Secretaria de Defesa Civil, 1999. 1 v.
- [6] C. Kuranaga; M. C. A. Nono; M. D. Silva and S. L. Mineiro. Influence of microstructure on humidity sensing properties of ZrO<sub>2</sub>-TiO<sub>2</sub> ceramics. In: CONGRESSO DE MICROSCOPIA E MICROANÁLISE NO MERCOSUL (MICROMAT), 2002, Curitiba. *Resumos da Área de Materiais*. Curitiba: Universidade Federal do Paraná, 2002. p. 247.
- [7] C. Kuranaga. *Pesquisa e desenvolvimento de cerâmicas para aplicação como sensores de umidade ambiente*. 1999. Relatório de bolsa (DTI/CNPq) Laboratório Associado de Sensores e Materiais, Instituto Nacional de Pesquisas Espaciais, São José dos Campos.

- [8] KURANAGA, C. Kuranaga. *Pesquisa e desenvolvimento de cerâmicas para aplicação como sensores de umidade ambiente*. 2001. Relatório de bolsa (DTI/CNPq) Laboratório Associado de Sensores e Materiais, Instituto Nacional de Pesquisas Espaciais, São José dos Campos.
- [9] Y. Shimizu, H. Arai and T. Seiyama, Theoretical studies on the impedance-humidity characteristics of ceramic humidity sensors, *Sensors and Actuators*, 7 (1985) 11-22.