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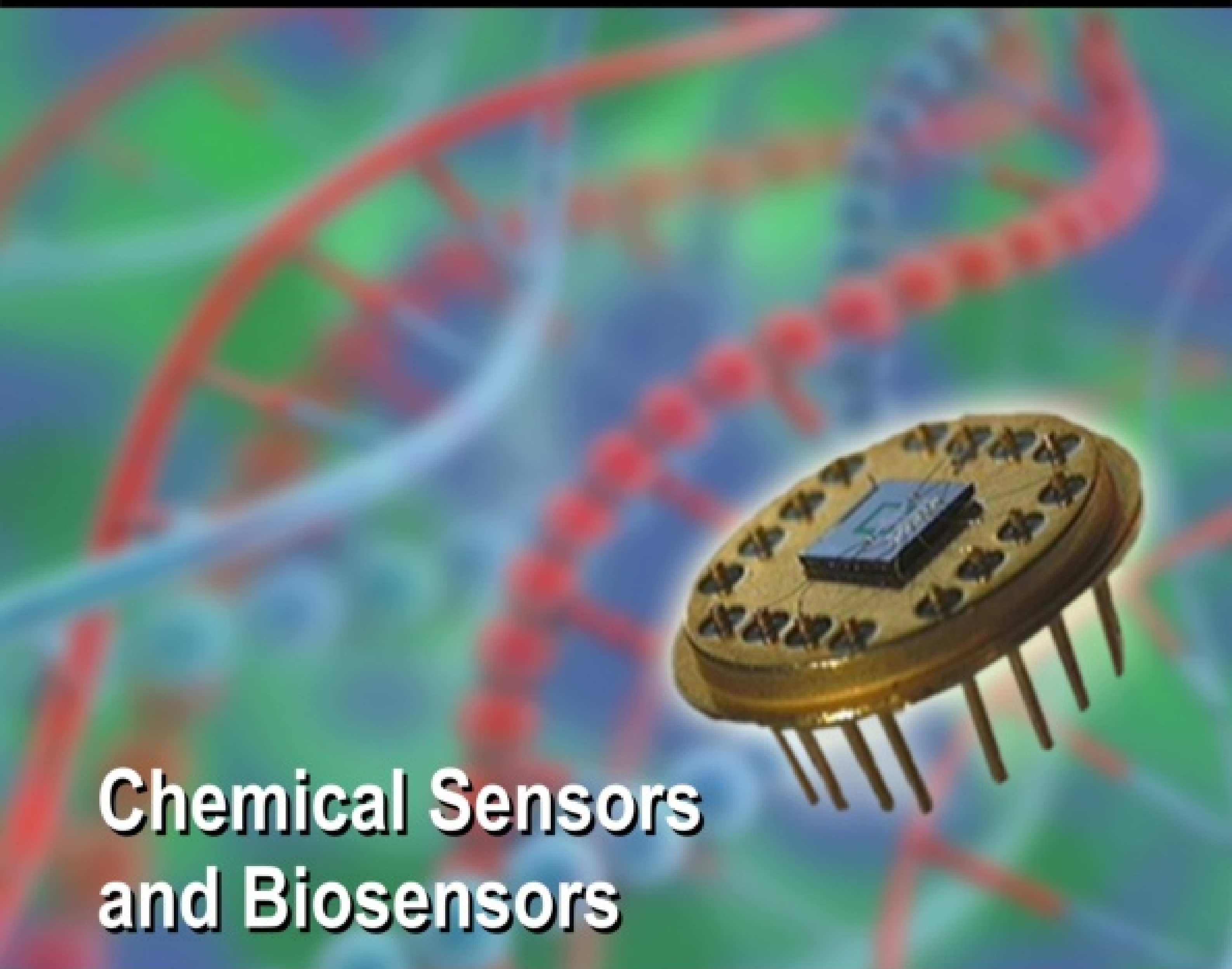

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A Dew Point Meter Comprising a Nanoporous Thin Film Alumina Humidity Sensor with a Linearizing Capacitance Measuring Electronics

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Abstract: A novel trace moisture analyzer is presented comprising a capacitive nanoporous film of metal oxide sensor and electronics. The change in capacity of the sensor is due to absorption of water vapor by the pores. A simple capacitance measuring electronics is developed which can detect any change in capacitance and correlates to ambient humidity. The circuit can minimize the parasitic earth capacitance. The non linear response of the sensor is linearized with a micro-controller linearizing circuit. The experimental result shows a resolution of -4°C DP and accuracy within 2%. *Copyright © 2008 IFSA.*

Keywords: Porous α-Al_2O_3, Micro controller, Trace moisture sensor, Thin film

1. Introduction

Porous alumina can be produced at a relatively low cost and a verity of structural configurations [1-3]. Sol-gel processing of alumina allows improved control pore morphology, phase formation, purity and product microstructure compressed to the more traditional techniques like anodic oxidation of alumina sheets [4, 5] and spray drying [6-9]. Even through this process has been well known for decades [10-14] but little use has been found in sensor technology. The most common techniques for the preparation of sol gel films involve primarily spin coating [15-18], dip coating [19-24], spraying and electro deposition.

The aim of the present study is to develop thin nano porous sol-gel films for making trace moisture sensor. The sensors are found suitable for monitoring moisture in nitrogen atmosphere for preparation of uranium and plutonium palate for fast breeder test reactor.

2. Development of Porous Alumina Sensor

Nano porous alumina is formed by sol gel thin film on the gold electrode. The film on the gold surface is used as transducing layer for sensing the ambient trace moisture. The effective dielectric of the porous layer under goes a large change when water vapour diffuses to the inner regions of the pore structure and absorbs on the porous layer. The capacitive sensor developed in this work consistent of a non porous substrate coated with gold on which the thin films are deposited of thickness 120-150 nm. It is covered by another porous electrode of gold. Contacts are taken from the two parallel gold electrodes. The water vapour is free to diffuse through the porous electrodes to the porous alumina film shows increase in capacitance.

3. Preparation of γ-Alumina Film

The sol-gel method for preparation of a boehmite sol was similar to that proposed by Yoldas [22]. Hydrolysis was performed at 90^{o}C by introducing aluminium tri-sec.-butoxide (ASB) into excess amount of water (water alkoxide molar ratio = 100/0.8) under vigorous stirring. After 45 minutes of hydrolysis, the colloidal solution was peptized by adding a given amount of 1.6 (N) HNO_3 solution (acid/alkoxide molar ratio = 0.071/1) and then maintained at 90^{o}C for one hour constant stirring. A transparent sol was finally obtained by continuing refluxing the solution at 90^{o}-95^{o}C for 16 hours. The gold coated alumina support was dip coated with the binder and dispersant mixed boehmite sol by a rotating dipping technique. The support was fixed on the bottom of a rotating shaft and immersed into the solution for 1 min. of slow rotating and then followed by fast spinning to remove the residual sol. After drying in air, the support with xerogel was calcined to 500^{o}C with a heating rate of 2^{o}C/minutes and soaking for 34 hours to obtain a thin top layer on it. The process was repeated six times with dipping and calcinations in order to get a crack-free supported alumina coating.

In Fig. 1, FESEM image of surface coating layer, from the image we see that lots of nano pore distribution through out the surface. Fig. 2 is the AFM image of coating surface, in high resolution surface morphology grains and pores are visible.

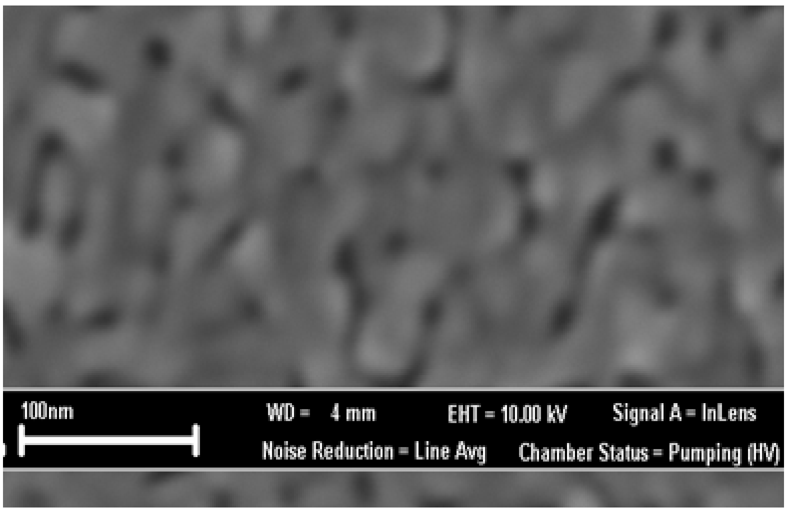

Fig. 1. Surface nano porous structure of sol-gel thin film.

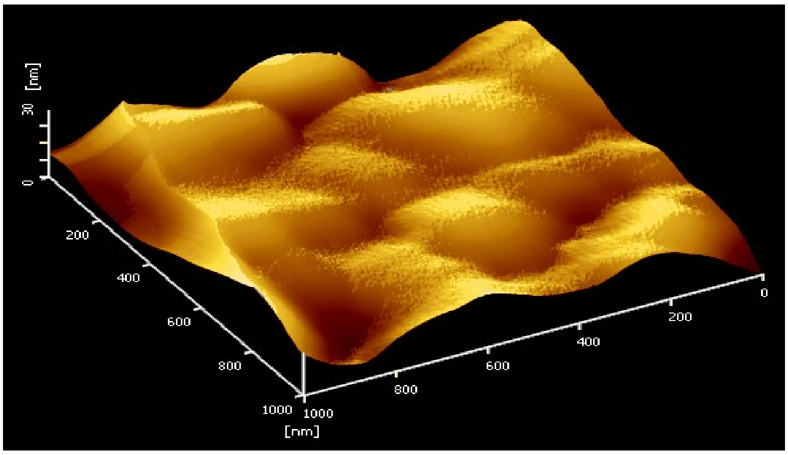

Fig. 2. Coating growth on gold surface with high resolution.

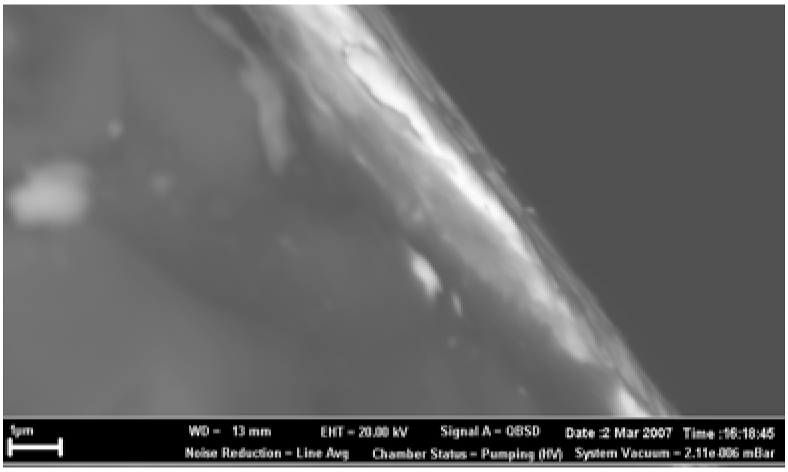

Fig. 3. Thickness of the α-alumina dielectric layer.

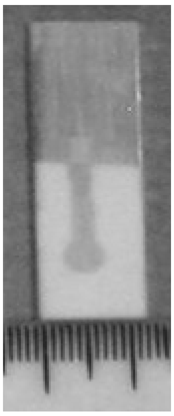

Fig. 4. Bottom gold electrode on α-alumina thin film sensor.

4. Electronics

We have developed an instrumentation technique to measure trace moisture in the range 6 ppm to 50 ppm with a resolution better than 0.5 ppm and accuracy 0.1%. The electronics should preferably a capacitance to voltage converter with a CMOS timer. Where the average DC value of a periodic pulse train has direct relationship to the duty cycle or DC value= V_p (t_1/T) where period T and peak amplitude V_p is kept fixed where DC value will be directly proportional to the pulse width t_1. CMOS dual timer can detect capacitance over a range of 50 pF to 50 nF.

The block diagram of the signal conditioning circuit is shown in the Fig. 5.

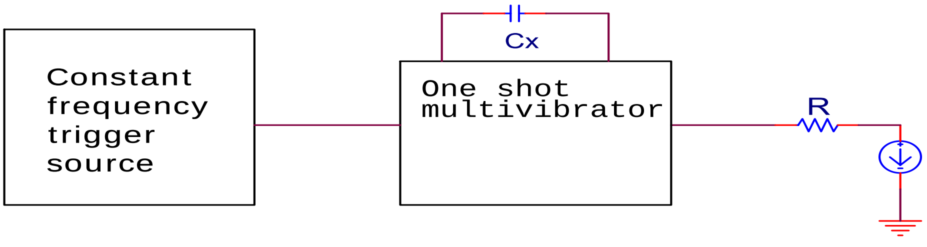

Fig. 5. Block diagram of the signal condition circuit.

Each time a pulse occurs, the one-shot initiates an output pulse whose width t_1 is determined by the unknown capacitance C_x. Now this variation of this pulse width is converted to an average dc voltage by RC network. This dc voltage is applied to a differential amplifier and then taken into the ADC 0808 input. The ADC 0808 is converted the analog voltage to equivalent digital data which is read by microcontroller every instant and according to this digital data microcontroller control the multiplexed-display to display the moisture contain according to the look-up-table as made during characterization of the sensor.

5. Characterization

The sensor was characterized in a controlled chamber with temperature maintain at 30^0C. The ppm Vs moisture inside the chamber was varied between 6 ppm to 50 ppm in a controlled manner with the help of needle valve which controls the moisture level. The corresponding change in capacitance due to change in pulse width was measured by LCR meter and corresponding voltage was measured by the CMOS ICM7556. The output voltage of the ICM7556 ranges from 2.5V to 3.0V respectively. The quasi-linear properties was finally linearized by a programmed look up table in the microcontroller (IC no AT89C52) circuit for digital output. For calibration SHAW model SADP-TR-R was used in parallel with the controlled chamber.

6. Conclusion

A simple but accurate trace moisture measuring system was developed and characterized. The presented hygrometer combines with the nano porous alumina thin film dielectric and capacitance measuring electronic circuit. The nano porous alumina was fabricated by sol-gel thin film in between two electrodes for better capacitive sensing. A simple CMOS timer was developed to measure and monitor the capacitive changes with accuracy and resolution better than (0.1 %). Good linearity over the range 6-50 ppm was achieved with the help of a microcontroller.

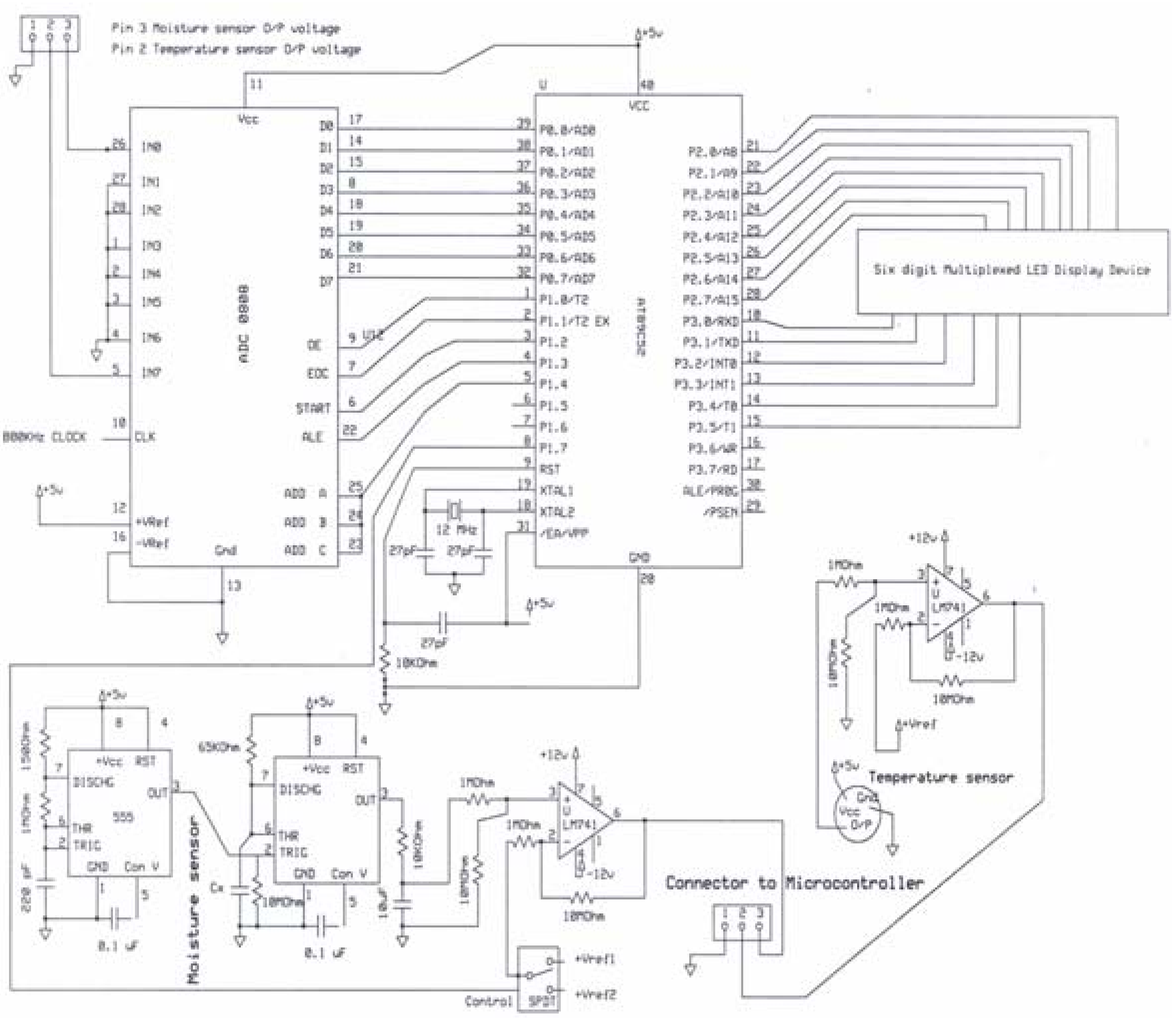

Fig. 6. Microcontroller based complect linerasition circuit diagram with CMOS timer ICM7556.

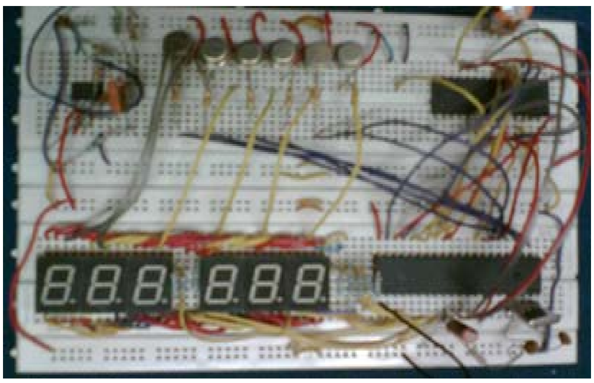

Fig. 7. Photograph of the electronics.

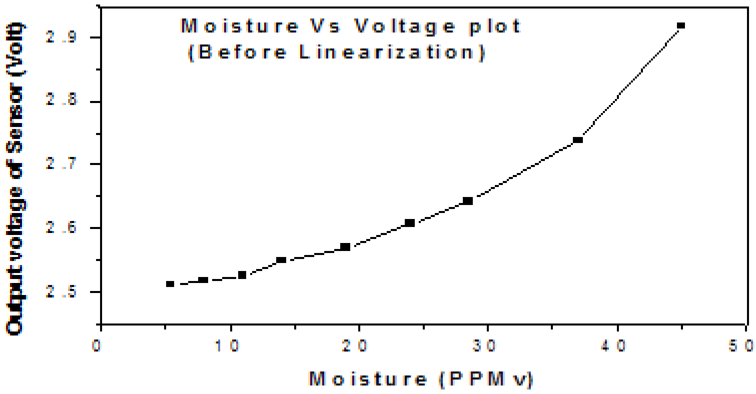

Fig. 8a. Sensitivity of sensor in combination with electronics.

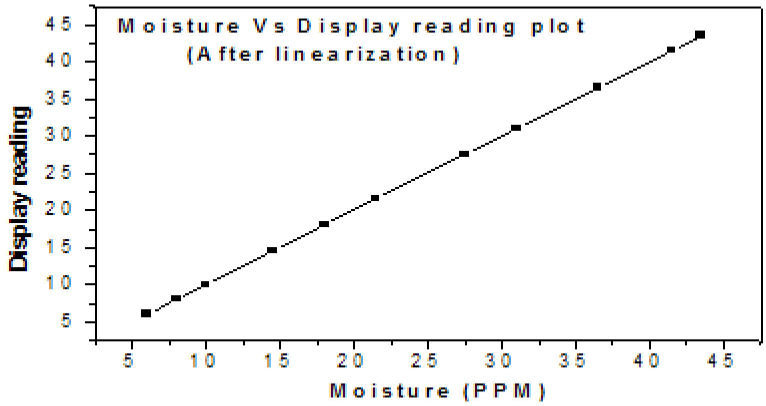

Fig. 8b. Display reading calibration curve of the developed meter.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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