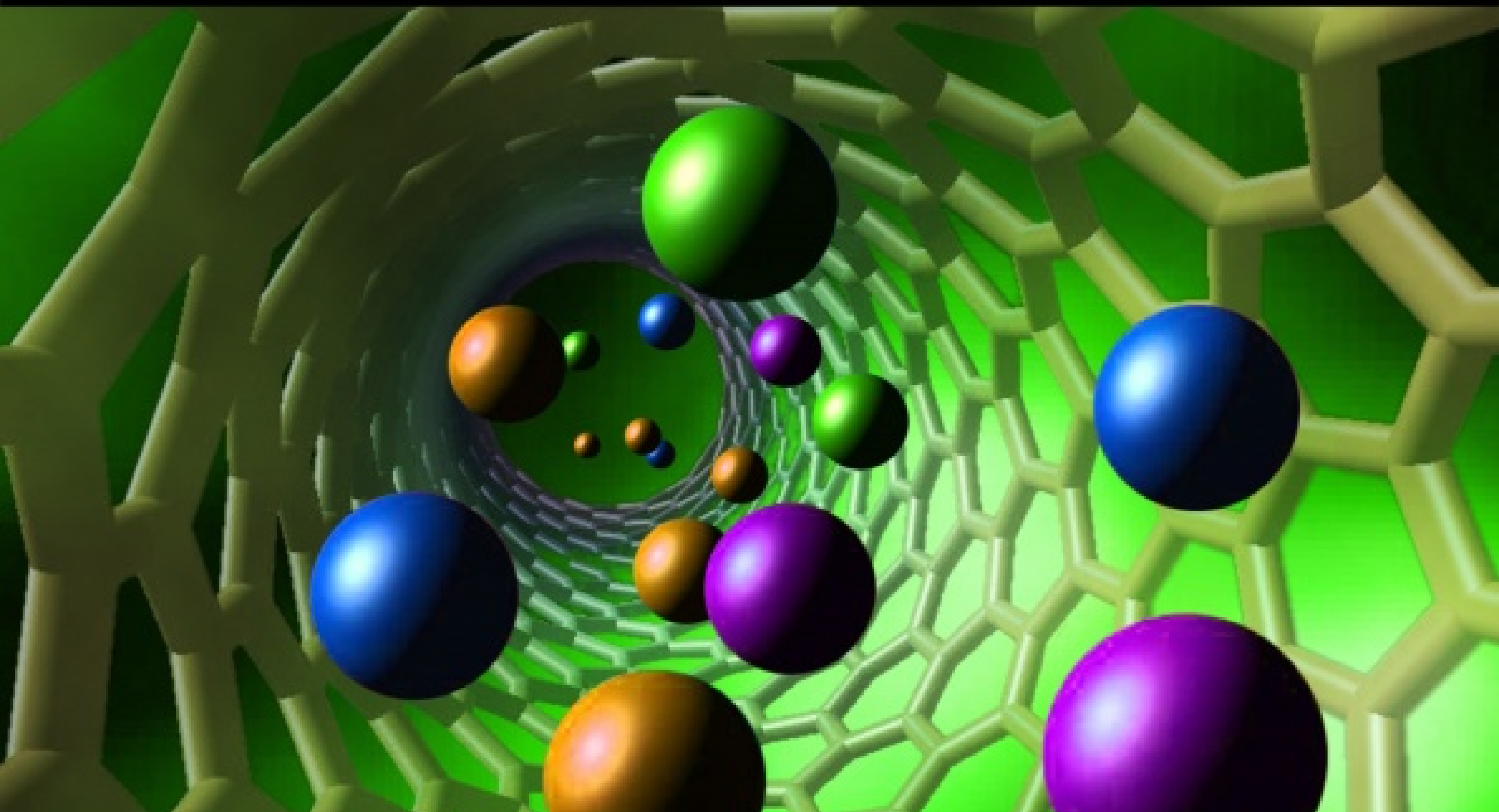
ISSN 1726-5749

SENSORS & TRANSDUCERS

11/07 vol. 85



Nanosensors and Nanodevices

International Frequency Sensor Association Publishing

IFSA

Sensors & Transducers

Volume 85
Issue 11
November 2007

www.sensorsportal.com

ISSN 1726-5479

Sensors & Transducers Journal (ISSN 1726-5479) is a peer review international journal published monthly online by International Frequency Sensor Association (IFSA). Available in electronic and CD-ROM.

Sensors & Transducers Journal

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Volume 85
Issue 11
November 2007

www.sensorsportal.com

ISSN 1726-5479

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Sensors & Transducers Journal, Vol.85, Issue 11, November 2007, pp. 1714-1720

Sensors & Transducers

ISSN 1726-5479

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Trace Moisture Response Property of Thin Film Nano Porous γ-Al_2O_3 for Industrial Application

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Received: 11 July 2007 /Accepted: 20 November 2007 /Published: 26 November 2007

Abstract: Nano porous thin film γ-Al_2O_3 is prepared by sol-gel method by heat treating process at 550^0C.The samples are made of various coating thickness and investigated as trace moisture sensor at room temperature within the range of -72^0C to -42^0C dew point (DP). It is found that at a particular coating thickness the sensitivity and response time are found to be suitable for commercial application. The capacitance change over the dew point (DP) range from -72^0C to -42^0C is found to be prominent for making a sensitive device. *Copyright © 2007 IFSA.*

Keywords: Thin film, Nanopore, γ-Al_2O_3, Coating thickness, Suitable for making device

1. Introduction

Thin film technology is found to be in many applications [1-9], including microelectronics, optics, magnetic, hard and corrosion resistant coating, micro-mechanics etc [10-17]. Progress in each of these areas depends upon the ability to selectivity and controllability deposit thin film thickness ranging from tens of angstroms to micro meter with specified physical properties. This, in turn, requires control often at the atomic level of film microstructure and microchemistry. There are a vast number of deposition method available and in use today [18-20]. However, all the methods have their specific limitation and involve compromises with respect to process specifications, substrate material limitations, expected film properties and cost. This makes it difficult to select the best technique for any specific application. The most important factor in preparation of thin film material is to control porosity and pore size for sensing low to high level moisture. Sol gel thin film moisture sensor has not yet come to commercial market. The objective of the goal is to develop low cost technology in this area**.** The aims of these observations are at developing sensors by sol gel process [21-24] avoiding costly conventional process such as evaporation, chemical vapour deposition and sputtering where yield losses are very high and the cost is significant and expensive. The advantage of sol-gel process is low cost. Composition and micro structure of the material can be controlled.

2. Experimental

2.1. Preparation of the Thin Film

A porous thin film of γ-Al_2O_3 was prepared from 20 gm of Al-Sec.-Butoxide ($C_{12}H_{27}Al_2O_3$] dissolving in 150 cc hot water and keeping the solution at 90^oC under stirring for 30 minutes. Peptization was done by 0.7 cc concentrated HNO_3 and refluxing overnight. After preparing the sol solution calculated amount of binder was added to the sol solution. Dip coating was done to the above solution on the gold plate. The green thin film was dried 4-5 hours. After drying the coated gel was cured at 450^oC for 2 hour. For higher film thickness the dipping and drying process was repeated a number of times. The sintering was done only after the final dipping.

2.2. Methods of Characterization

The powder XRD patterns were measured by X-ray diffraction instrument (Philips PW 1730) using Cu K_α radiation. The morphology of the film was characterized by Field Effect Scanning Electron Micrograph (FESEM) and Atomic Force Micrograph (AFM). The characteristic curves of humidity sensitivity were carried out with LCR meter (model no. HP4284A) at room temperature. The controlled humidity environments were achieved using gas cylinders with different trace moisture level at ambient temperature which, yields from -64^{0}C to -46^{0}C DP respectively. The thin films were developed on a gold plate which was itself one of the electrodes and second electrode was screen printed (Model no-65 DEK, UK) on the surface of thin film. The figure of the sample is shown in Fig. 1.

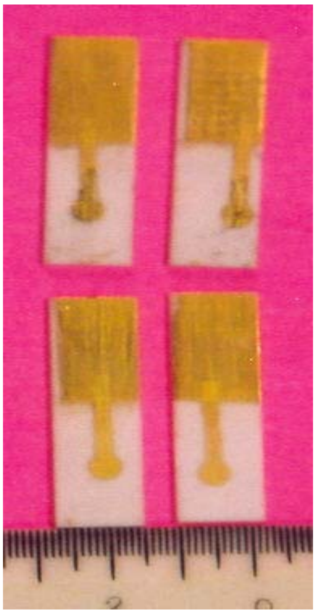

Fig. 1. Thin film sensor.

3. Results and Discussion

3.1. Film Thickness

Fig. 2 and Fig. 3 are the film thickness of sol-gel coat films. From the figures, we see that six coat film is much thicker than that of three coat. Higher the coating thickness lower is the sensitivity to moisture. Three coat is the optimum coating thickness to produce sensor materials. Below this thickness short circuit takes place and the materials can not be used as a sensor.

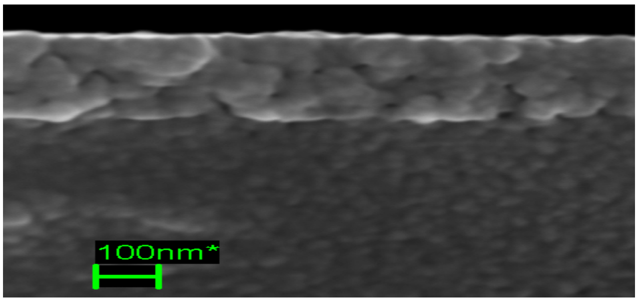

Fig. 2. Film thickness by six times dip coating measured by FESEM.

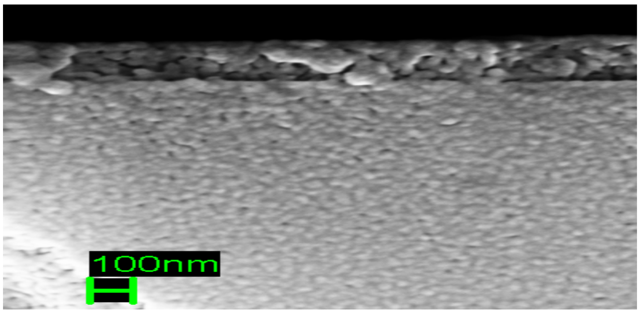

Fig. 3. Film thickness by three times dip coating measured by FESEM.

3.2. X-ray Diffraction

Fig. 4 is the crystalline phase of γ-Al_2O_3 powder, which is prepared from alumina sol on drying, then converted to gel and finally fired at 450^0C in 1.5 hours. From the figure we see width of peak and peak height value is the identical to literature value. γ-phase alumina is highly sensitive to parts per million (PPM) moisture.

3.3. Surface Characterization by AFM

FESEM images of the coating sample are represented in Fig. 5 and Fig. 6. From the figure we see that the surfaces of the both samples are of nano porous dimensions. Topographic view of the surface of the three coat is represented in Fig. 3.and six coat in Fig. 6. The surface is rather smooth in Fig. 3 as compared to Fig. 6. The surface layer is composed of nanopore and gamma alumina grain.

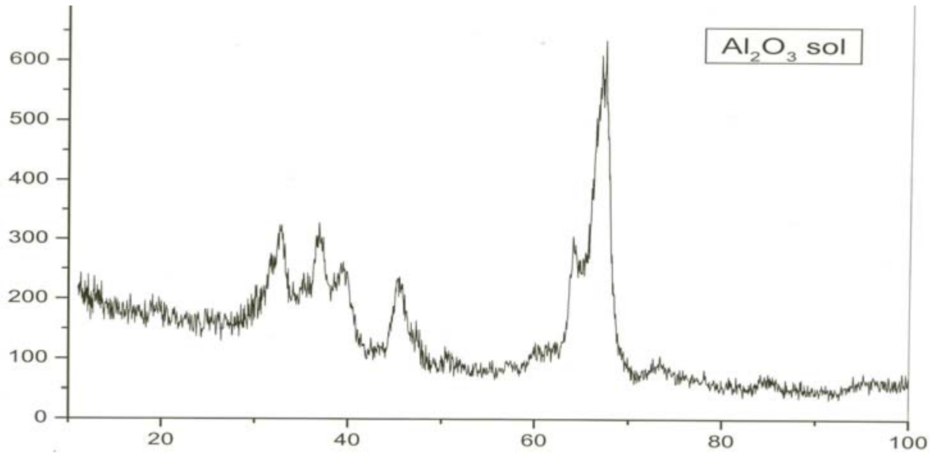

Fig. 4. γ-Al_2O_3 crystalline phase of coating sample.

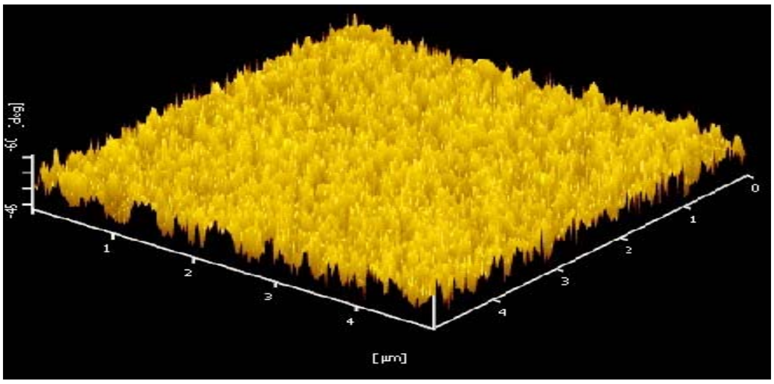

Fig. 5. 3D Structure of thin film with three times dip coating.

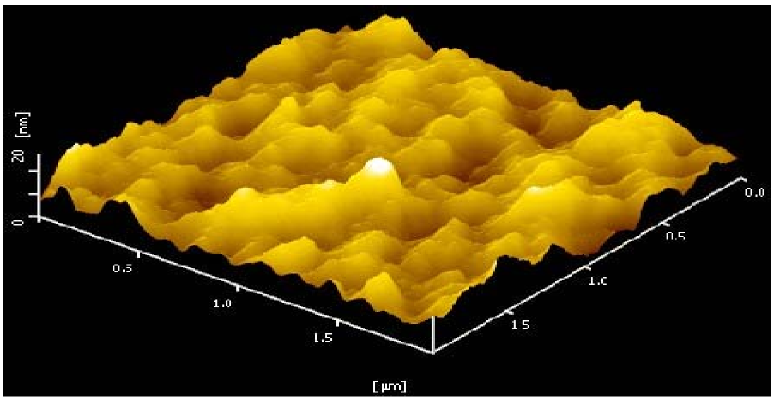

Fig. 6. 3D Structure of thin film with six times dip coating.

3.4. Surface Characterization by FESEM

Surface morphologies of thin film with six time dip coating and three time dip coating are shown in Fig. 7 and Fig.8 respectively.

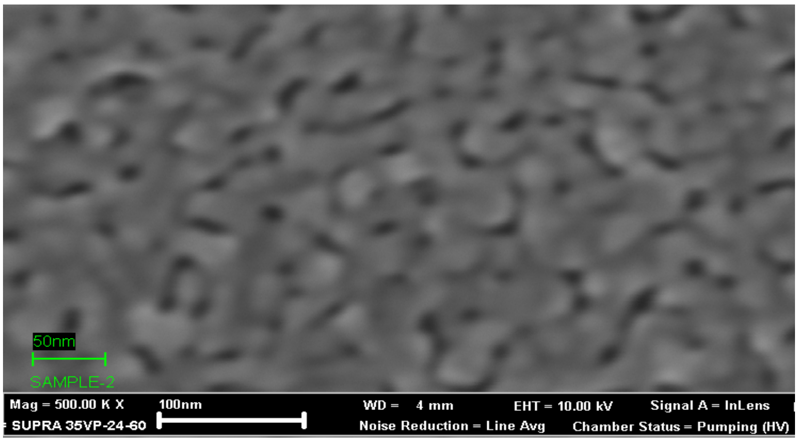

Fig. 7. Surface morphology of thin film with six time dip coating.

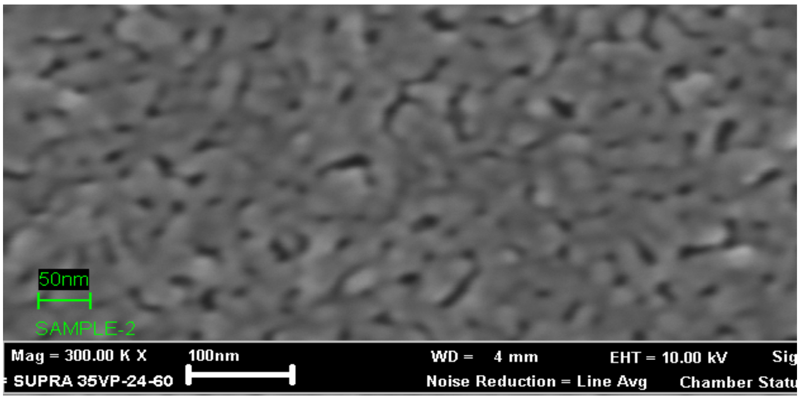

Fig. 8. Surface morphology of thin film with three time dip coating.

3.5. Trace Moisture Sensitive Property

The results of capacitance measurements as a function of trace moisture at room temperature are shown in Fig. 11. It can be seen that the change in capacitance at three coat is higher at a range up to -48^{0}C DP showing higher humidity, sensitivity and resolution property. After six times coating the capacitance and resolution greatly decreased by five times due to higher thickness with the increase of moisture up to -48^{0}C DP. The three coat exhibiting the maximum humidity sensitive property and linearity was selected for evaluation of hysteresis and response-recovery time.

Capacity on moisture and frequency depend on the three coat sample. It is seen that when the frequency is 100 Hz the capacity increases greatly with increasing moisture. However, when the higher frequency was used, such as 1 KHz, 10 KHz and 100 KHz the change in capacitance became less with increasing humidity.

The response and recovery time were shown in Fig. 9 and Fig. 10. The time taken by the sensor to achieve 90% of total capacitance change is defined as the response and recovery time. The response time of 3rd coated sample is 12 second (Fig. 10) shorter than the 6th coated sample shown in Fig. 9.

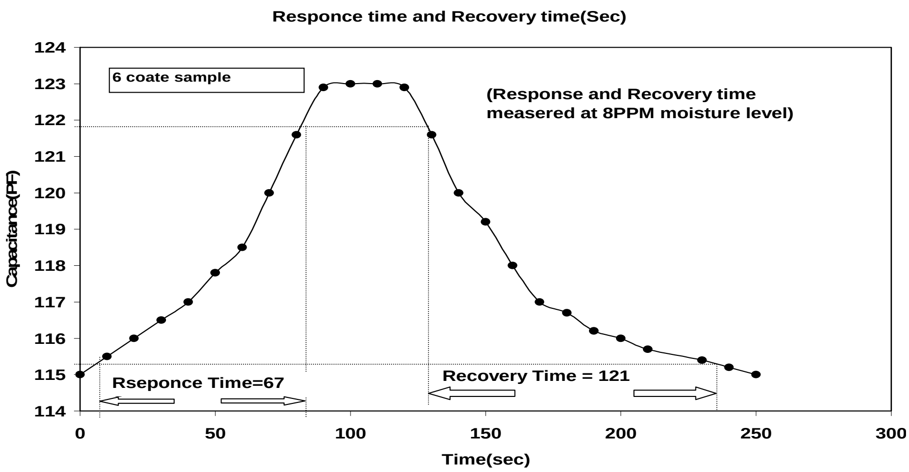

Fig. 9. Six time coating sample of Response and recovery time.

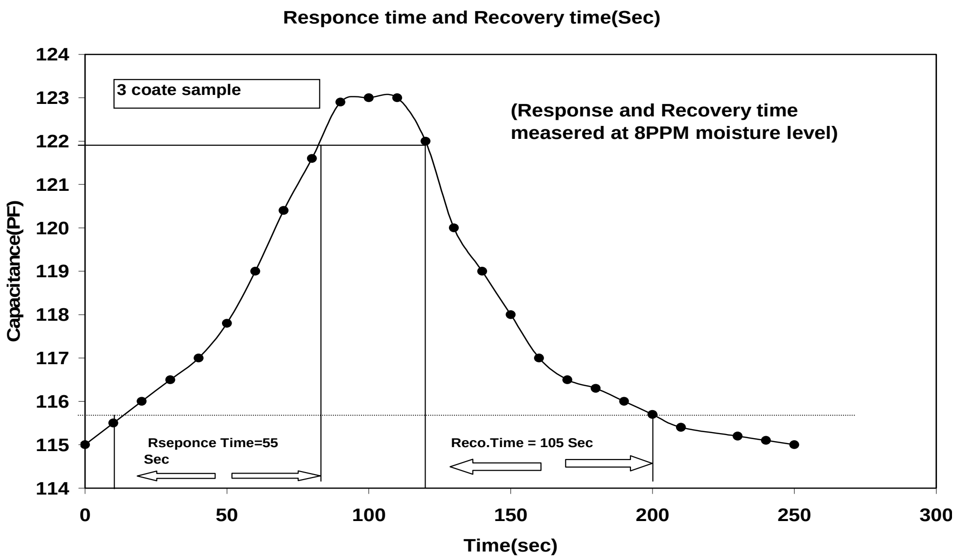

Fig. 10. Three time coating sample of Response and recovery time.

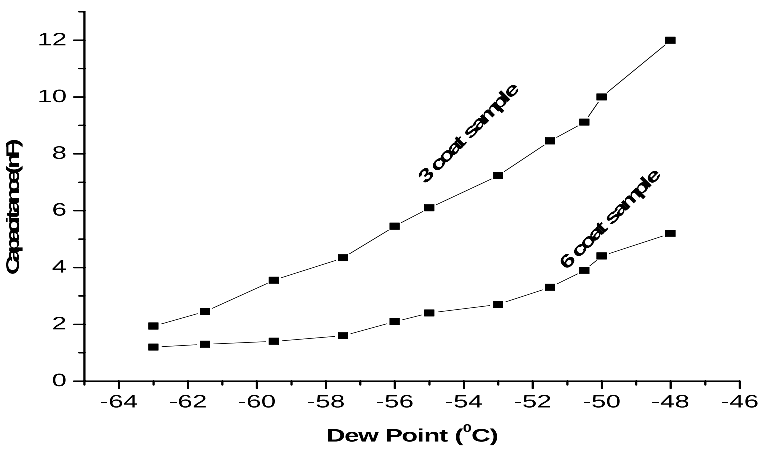

Fig. 11. Comparison between three coat and six coat sample.

Conclusion

Sol-gel thin film γ-Al_2O_3 was studied as humidity sensor material and their structure was characterized by XRD, FESEM and AFM. These tests indicate that the ordered nanoporous thin film coated structure is highly sensitive to trace moisture level.

Acknowledgements

The authors are thankful to Board of Research in Nuclear Sciences, Department of Atomic Energy, Govt. of India for financial support and the authors are also thankful to Dr. H. S. Maiti, Director, Central Glass and Ceramic Research Institute, Kolkata for rendering all sorts of cooperation for conducting the research work. The authors sincerely appreciate the kind help and assistance of Mr. A. K. Mandal, Dr. S. Sen in doing the SEM experiments at CGCRI.

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Guide for Contributors

Aims and Scope

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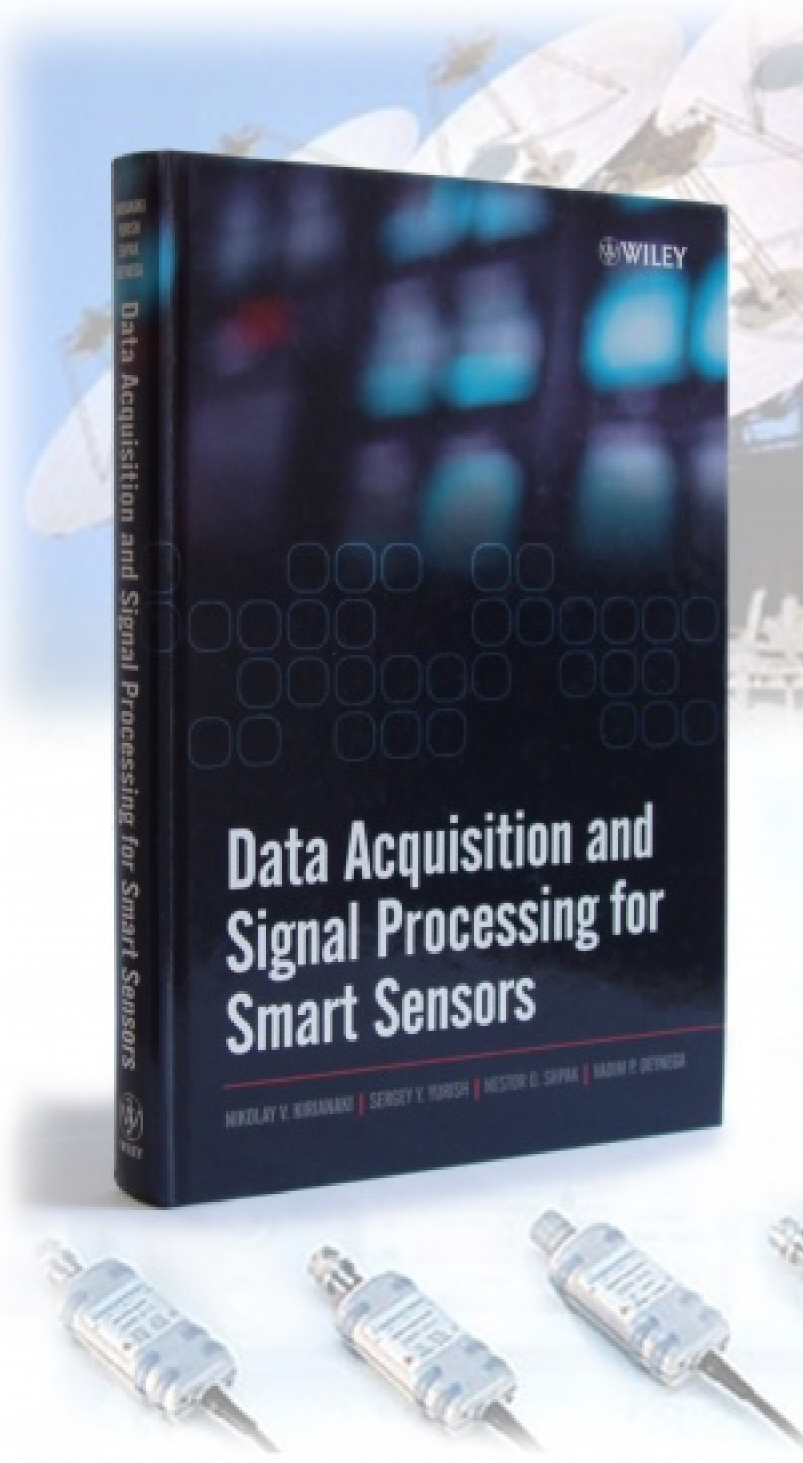
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