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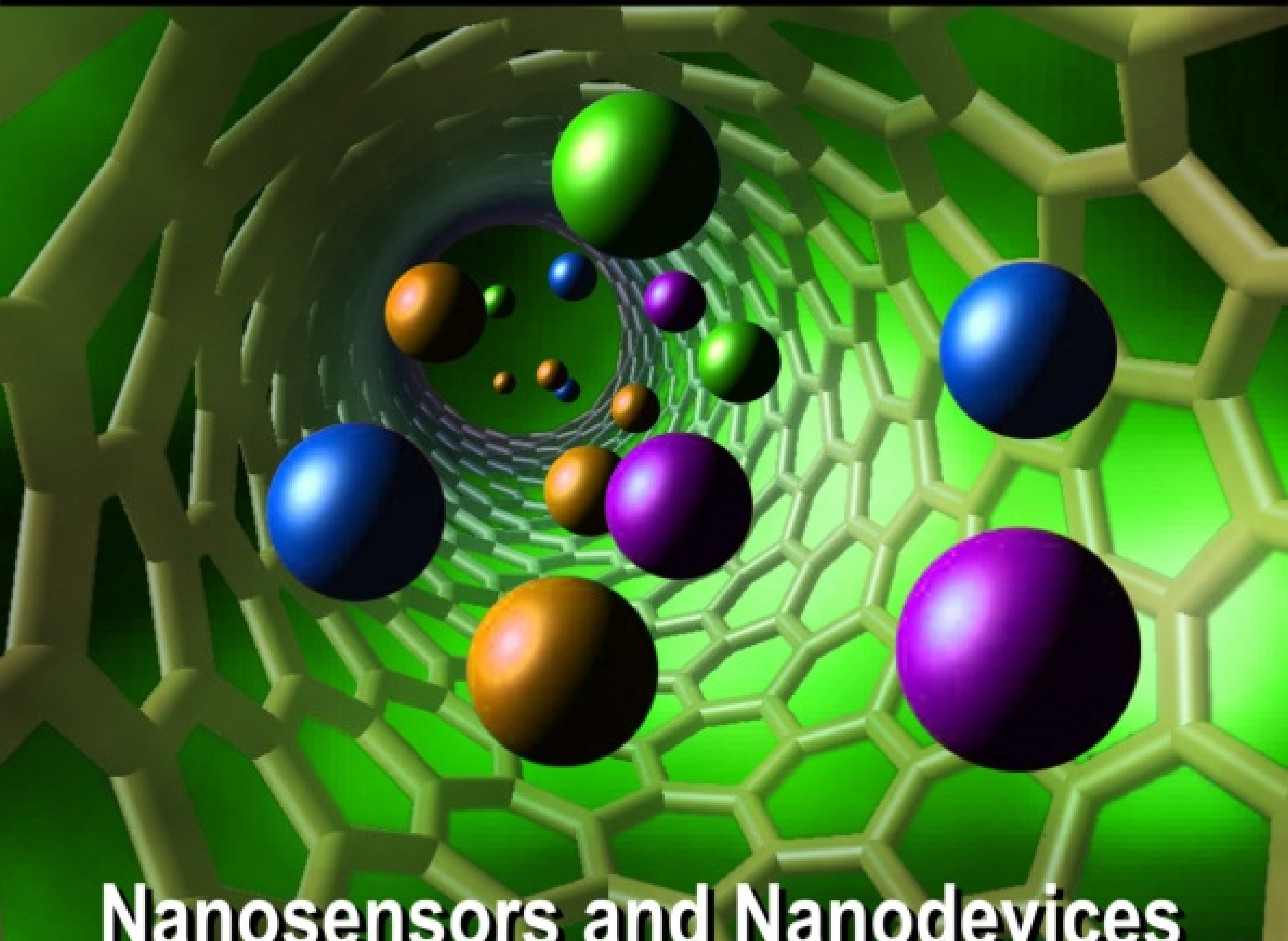

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Nanocomposites Sn-Si-O and Sn-Mn-O for Gas Sensors

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Abstract: Thin film nanocomposites Sn-Si-O and Sn-Mn-O were prepared by high frequency ion-beam reactive sputtering. The element composition (X-ray microanalyser JXA-840), the surface morphology (atomic force microscope FemtoScan-001), electrical properties (Hall effect) and gas sensitivity were investigated. The similar character of influence films element composition on the grain size of polycrystals and electrical and gas sensitive properties of the samples was determined. The increase of additive amount in nanocomposites leads to the decrease of grain size and enhancement of adsorption activity of surface states in the film. *Copyright © 2007 IFSA.*

Keywords: Nanocomposites, Element composition, Electrical-physical properties, Gas sensitivity

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1. Introduction

Nanocomposite films on the base of tin dioxide are wide used as gas sensitive elements [1]. Gas sensitivity of tin dioxide depends on presence of additives and their amount in the film, grain size of crystals and microstructure of the film. When grain size is as small as double value of Debye length in the grains of nanocrystal gas sensitivity increases. Moreover catalytic influence of additives leads to the decrease of working temperatures of gas sensors.

The aim of our work is investigation of influence of additives nature and grain size of polycrystals on the adsorption activity of surface states in nanocomposites Sn-Si-O and Sn-Mn-O.

2. Samples and Experimental

Thin film nanocomposites were prepared by high frequency ion-beam reactive sputtering of composite metal targets in the ambient of Ar+O_2. Fifteen series of each nanocomposite Sn-Si-O and Sn-Mn-O with different amount of additives (0.5 – 7) at.% were prepared and investigated. Samples of each series were prepared at the same technological conditions. Thermal treatment at 500 ^{0}C during several hours was carried out for the crystallization of amorphous films. The crystallization process in the air was controlled by measurements of electrical resistivity of thin films. Thickness of the films was measured by interference microscope MII-4.

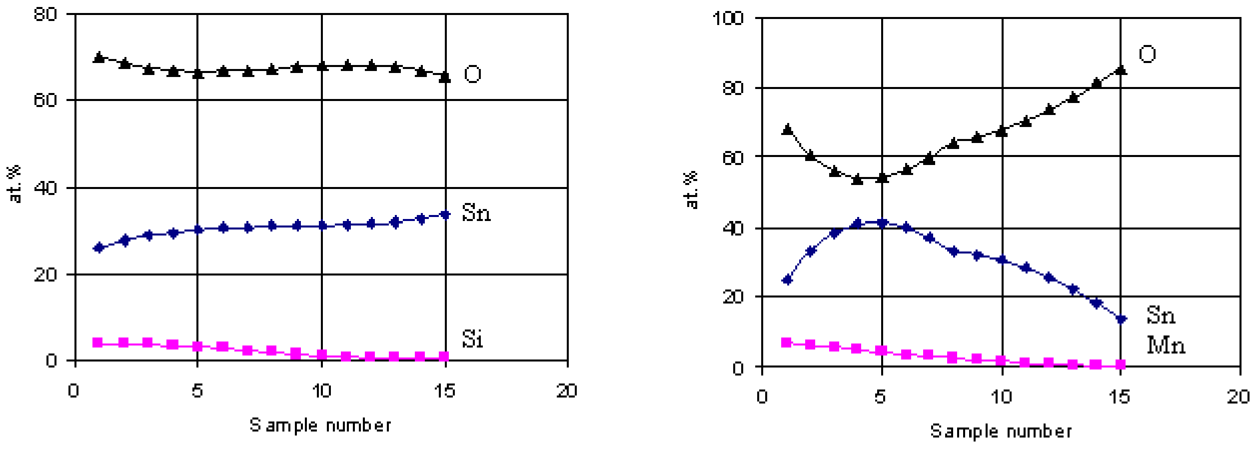
Element analysis of the samples was carried out with the help of X-ray microanalyser JXA-840. Surface structure was studied by scanning atomic force microscope (AFM) FemtoScan-001. Electrical properties of the films were measured by Hall effect according to Van-der-Pauw method in the temperature range (20 – 400) ^{0}C. Concentration and mobility of free charge carriers were investigated in dependence on film composition.

Gas sensitivity S_g of the films was measured in different gases (ethanol, acetone, formaldehyde, ammonia) by standard method [2] as ratio of film resistance in the air R_a and film resistance in the mixture of investigated gas and the air R_g: $S_g = R_a/R_g$ or $S_g' = (S_g - 1) \cdot 100\%$.

3. Results and Discussion

Thin film nanocomposites on the base of tin dioxide have thickness of about 1 µm.

Fig. 1 presents the elements distribution in all investigated samples. Results of element analysis show that additive contents in tin dioxide films are in the range from 0.55 up to 3.9 at. % for Si and from 0.5 up to 7 at.% for Mn.



(a) (b)

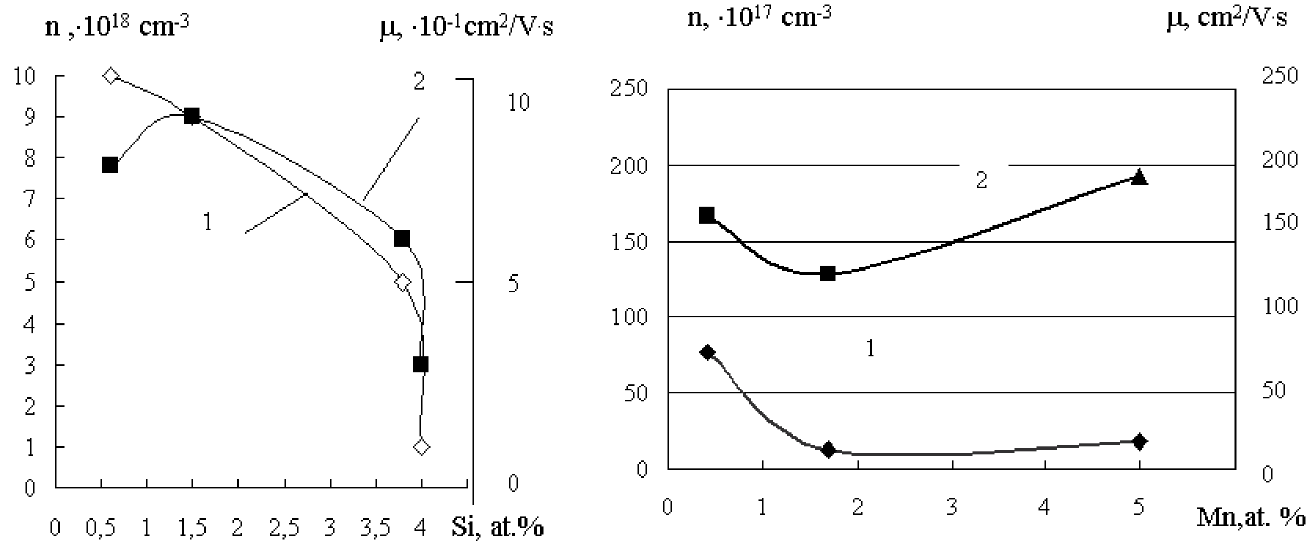
Fig. 1. Element composition of film nanocomposites: a - Sn-Si-O, b - Sn-Mn-O.

Investigation of surface morphology by AFM shows that composites have a nanocrystalline structure, and grain sizes are changed in dependence on doping amounts [3]. It was found that with increasing of additives content the grain sizes decrease. In nanocomposite Sn-Mn-O with maximal amount of additive 7 at.% Mn the grain size is about 20 nm, that is less than grain size in nanocomposite with minimal amount of additive 0.5 at.% Mn (~50 nm). It should be noted that the biggest effect of additive on the grain size was found in nanocomposite Sn-Si-O: grain size in nanocomposite with minimal amount of Si (0.55 at. %) is about 50 nm and in nanocomposite with maximal amount of Si (3.9 at. %) grain size is about 3 nm.

Tin dioxide and metal (Si, Mn) oxides compose neither alloys nor solid solutions. SnO_2 molecules and molecules of metal oxides co-exist in nanocrystalline film in the form of conglomerates that was detected by AFM study. Thus it was determined that in nanocomposites on the base of SnO_2 the presence of additives of another type of oxides prevents the growth of big grains and can be used for the control of structure and properties of gas sensitive layers.

The electrical properties of the samples depend on the element composition of thin films (Fig. 2). It was found that the increase of silicon amount from 0.6 at. % up to 4.0 at. % leads to the decrease of concentration of free charge carriers in film nanocomposite Sn-Si-O at one order of magnitude from 10^{19} up to 10^{18}cm^{-3}· There is also the decrease of the mobility of charge carriers in the range (0.3 – 1.0) cm^2/(V·s) (Fig. 2a). It means that the presence of silicon additive causes the decrease of concentration of free charge carriers in SnO_2 film. This effect is perhaps the result of formation of dielectric SiO_2 layers in the volume of tin dioxide film.

Some another behavior have concentration and mobility of free carriers in Sn-Mn-O nanocomposite (Fig. 2b). With increase of additive content from 0.5 up to 1.7 at % Mn the concentration decreases from 7.61·10^{18} to 1.24·10^{18} cm^{-3}, then in the range (1.7 – 5) at. % Mn concentration slightly increases up to 1.75·10^{18} cm^{-3}. There is the same tendency in behavior of mobility: with increase of Mn content it decreases from 160 to 130 cm^2/V·s, then in the range (1.7 – 5) at. % Mn mobility increases up to 190 cm^2/V·s.



(a) (b)

Fig. 2. Concentration (1) and mobility (2) of free charge carriers vs additive content in film nanocomposites: a - Sn-Si-O, b - Sn-Mn-O.

These experimental results allow to estimate the value of Debye length (L_D) in the grains of nanocrystals. The estimation shows that in dependence on concentration of free carriers (n) the value of L_D is in the range from 4,34 mn (n=10^{18} cm^{-3}) to 30 nm (n=10^{17} cm^{-3}).

It was found that temperature of maximal gas sensitivity towards different gases depends on type of additive and grain size as well. Fig. 3 shows temperature dependencies of gas sensitivity of film nanocomposite Sn-Si-O to vapors of alcohol (4930 ppm), acetone (3900 ppm) and ammonia (7830 ppm). Film nanocomposites with amount of Si 1.5 at. % have maximal sensitivity to alcohol vapors at 260 ^{0}C, acetone - 140 ^{0}C and ammonia - 140 ^{0}C.

It was determined that undoped films SnO_2 (with grain size about 50 nm) are characterized by temperature of maximal gas sensitivity to ethanol 330 ^{0}C, to acetone and ammonia 360 ^{0}C (Fig. 3, straight lines), to propanol at 400 ^{0}C, to formaldehyde at 300 ^{0}C. Thus films SnO_2 doped with Si detect alcohol, acetone and ammonia vapors at the lower temperatures than undoped tin dioxide films. This effect can be caused by reduction of energy threshold of interaction of gas ions with nanocomposite surface due to nanocrystalline grain sizes and as a result of increasing of catalytic activity of nondispersive oxides.

It is obviously that the presence of silicon additive improved gas selectivity of the film. Fig. 4a shows the dependence of maximal sensitivity to vapors of alcohol (4930 ppm) on silicon amount in film nanocomposite Sn-Si-O. It is evidently, that doping with silicon allows to decrease temperature of maximal sensitivity at several tens of degrees in dependence on impurity amount. Thus, it is possible to reduce the working temperatures of sensors with sensitive layer of film nanocomposite Sn-Si-O that leads to the diminish of the consuming power of the whole device.

The presence of manganese additive leads to the decrease of grain size up to 20 nm and temperature of maximal sensitivity up to 180 ^{0}C to ethanol and propanol, up to 100 ^{0}C to acetone, up to 240 ^{0}C to formaldehyde, and up to 260 ^{0}C to ammonia (Fig. 4 b, c).

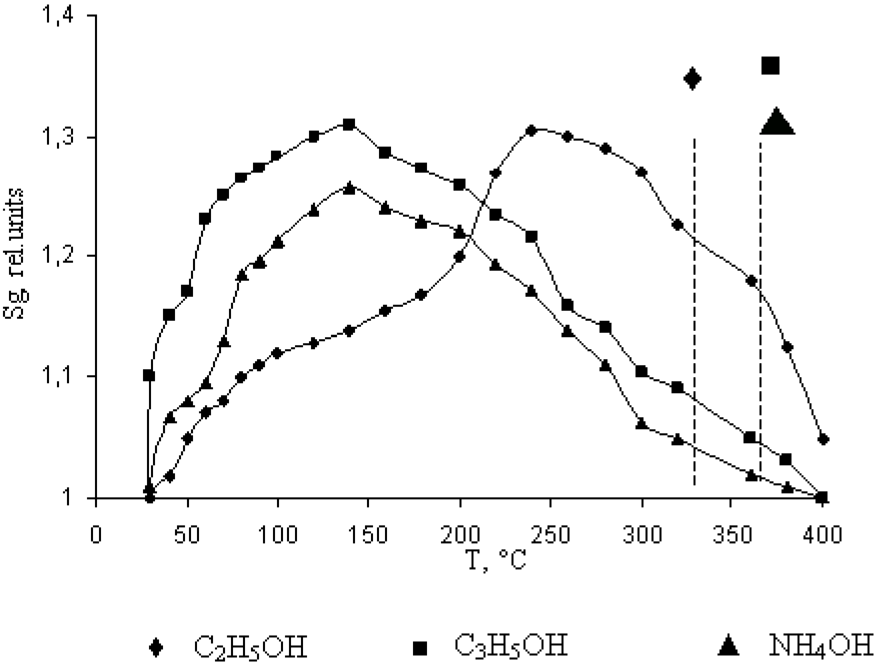
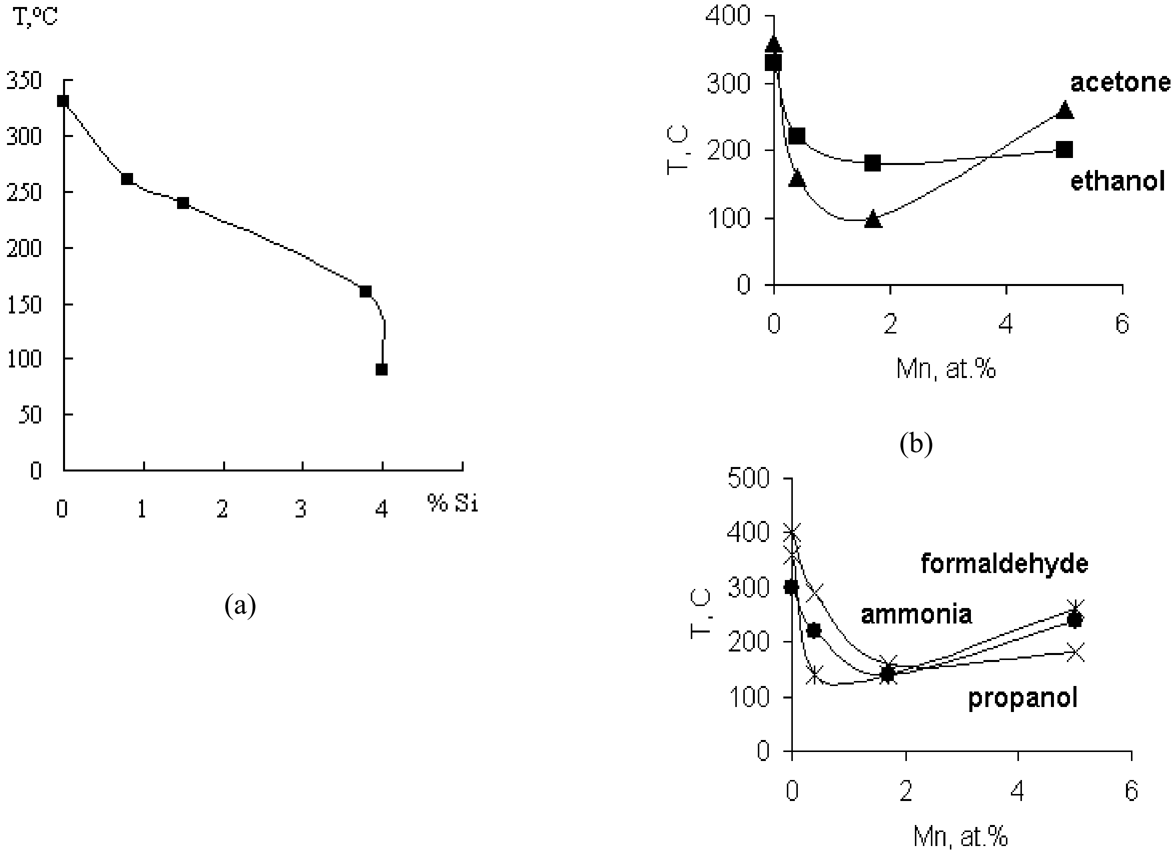


Fig. 3. Temperature dependencies of gas sensitivity Sg film nanocomposite Sn-Si-O (1.5 at.% Si) to vapors of ethanol (4930 ppm) (♦), acetone (3900 ppm) (■) and ammonia (7830 ppm) (▲)



(a)

(b)

(c)

Fig. 4. Temperature of maximal gas sensitivity to different gases vs additives content in film nanocomposites: a – Sn-Si-O; b, c - Sn-Mn-O.

5. Conclusions

Thin film nanocomposites (thickness ~ 1µm) on the base of SnO_2 with additives of (0.5 – 7) at.% Si and Mn were fabricated by high-frequency reactive ion-beam sputtering of composite target in the ambient of Ar + O_2. The average grain size of polycrystals was estimated as 3 – 50 nm in dependence on additives amount.

The study of nanocomposites Sn-Si-O and Sn-Mn-O demonstrated the similar character of influence of metal oxides amount on the grain size of polycrystals in nanocomposites and on gas sensitivity of thin films. Though the influences of additives content on the value of concentration and mobility of free charge carriers in nanocomposites Sn-Si-O and Sn-Mn-O are different.

The presence of silicon additive leads to the decrease of grain size up to 3 nm and temperature of maximal sensitivity up to 220 ^{0}C towards different gases. The presence of manganese additive leads to the decrease of grain size up to 20 nm and temperature of maximal sensitivity up to 260 ^{0}C to different gases.

Thus, it was determined that in nanocomposites on the base of SnO_2 the presence of additives of another types of oxides prevents the growth of big grains and can be used for the control of structure and properties of gas sensitive layers.

The using of thin film nanocomposites on the base of SnO_2 as sensitive elements in gas sensors can increase selectivity and decrease the consuming power of the whole device that can lead to the improvement of gas sensor performance.

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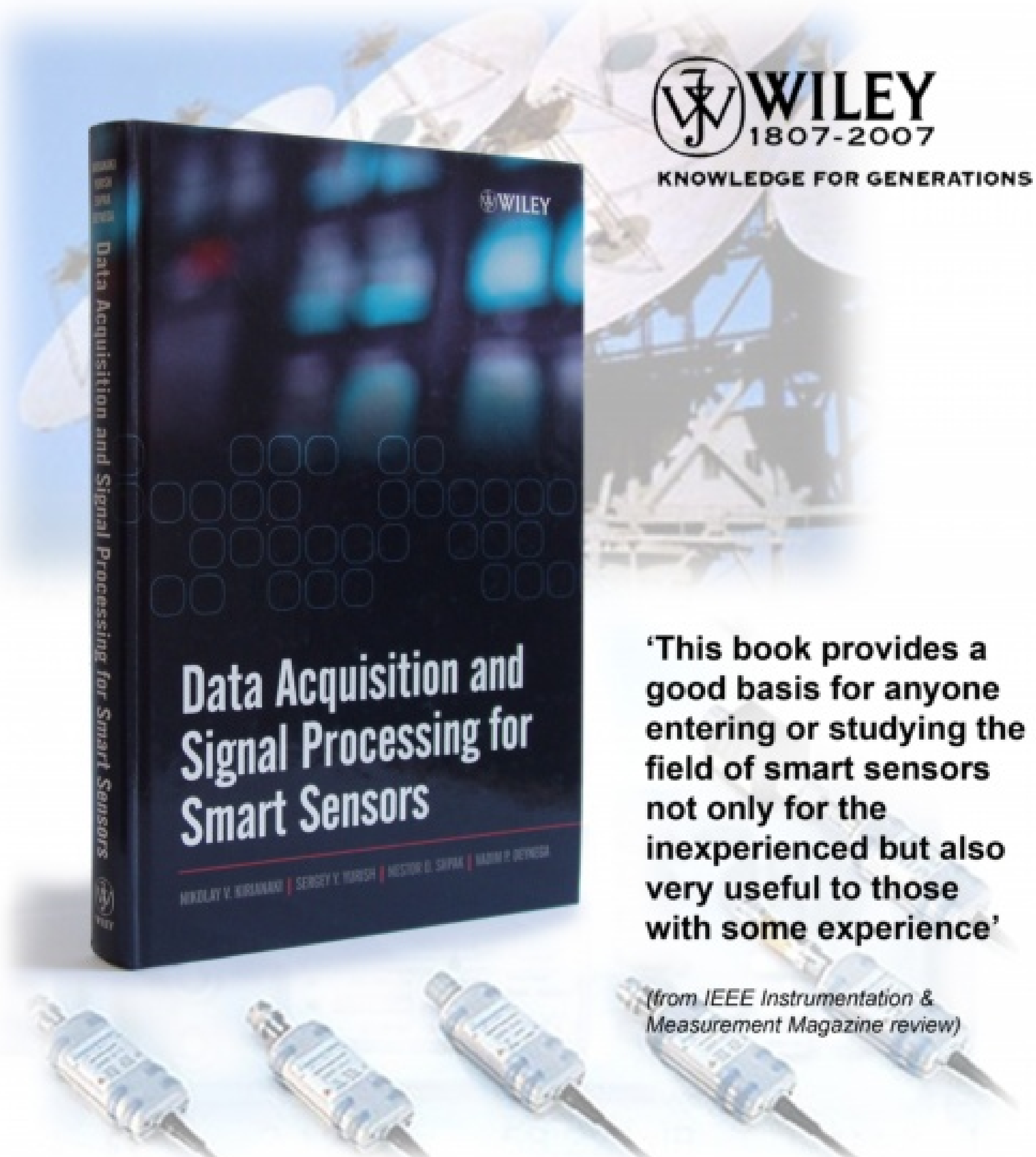

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