

# SENSORS & TRANSDUCERS **12** vol. 147 /12

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## Sensor Market Trends



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# Contents

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## Research Articles

<b>Constantly Evolving Smartness, Intelligence and Innovation Ensure Seamless System Integration</b> <i>Dr. Rajender Thusu</i> .....	1
<b>An Intelligent Temperature Measurement Technique Using J Type Thermocouple with an Optimal Neural Network</b> <i>Santhosh K. V., B. K. Roy</i> .....	6
<b>Thermal Sensitivity of Solid Polymer Coated Surface Transverse/Love Wave Based Resonators on AT-cut Quartz for Sensor Applications</b> <i>I. D. Avramov and K. D. Esmeryan</i> .....	15
<b>Effect on Passive Localization from the Shape Distortion of Triplet Linear Array Based on Piezoelectric Transducers</b> <i>Fei Xu, Xianlong Liu</i> .....	27
<b>Detecting Water Content in Hydrocarbon Emulsion Using Acoustic and Ultrasonic Detectors</b> <i>Rateb Issa, Ibrahim Al-Abbas and Hussein Sarhan</i> .....	36
<b>Study of Vibration Characteristics of a Multi Cracked Rotating Shaft Using Piezoelectric Sensor</b> <i>Rajeev Ranjan, N. K. Mandal</i> .....	45
<b>An Insole Device Based on Piezoelectric Sensor to Assess Plantar Pressure during Daily Human Activity</b> <i>Yemin Guo</i> .....	53
<b>A Novel Tuning Method for Repeatability Problem Solving of RF MEMS Disk Resonators</b> <i>Masoud Baghelani, Habib Badri Ghavifekr, Afshin Ebrahimi</i> .....	61
<b>FEM Based Optimization of Thin Membrane for Thermoelectric Energy Harvesting Devices</b> <i>Divya Jatain, Monoj Kumar Singha, Ajay Agarwal, Manoj Taleja</i> .....	68
<b>Visual Odometry in Dynamical Scenes</b> <i>Dong Zhang and Ping Li</i> .....	78
<b>An Experimentation on Anti-Reset Windup Scheme for Level Process Station</b> <i>I. Thirunavukkarasu, Mohammed Ibrahim Fareed Abuaiah, V. I. George, S. Shanmuga Priya</i> .....	87
<b>Design and Development of an Instrument to Determine the Fluoride Ion Concentration in Certain Tooth Pastes</b> <i>Saraswathi Parigi, Sreelekha Kande, Nagaraju Boya, Raghavendra Rao Kanchi</i> .....	95

<b>Data Mining Approach to Polymer Selection for Making SAW Sensor Array Based Electronic Nose</b> <i>Sunil K. Jha and R. D. S. Yadava</i> .....	108
<b>Development of a Non-invasive Micron Sized Blood Glucose Sensor Based on Microsphere Stimulated Raman Spectroscopy</b> <i>Alireza Bahrampour, Neda Jahangiri, Majid Taraz</i> .....	129
<b>MOS Device Chemical Response to Acceptor Stimuli</b> <i>Rina Lombardi, Ricardo Aragón and Héctor A. Medina</i> .....	143

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International Frequency Sensor Association (IFSA).



International Frequency Sensor Association (IFSA) Publishing

## Digital Sensors and Sensor Systems: Practical Design

**Sergey Y. Yurish**



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*Digital Sensors and Sensor Systems: Practical Design* will greatly benefit undergraduate and at PhD students, engineers, scientists and researchers in both industry and academia. It is especially suited as a reference guide for practitioners, working for Original Equipment Manufacturers (OEM) electronics market (electronics/hardware), sensor industry, and using commercial-off-the-shelf components

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## MOS Device Chemical Response to Acceptor Stimuli

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**Abstract:** Gap gate MOS capacitors exhibit changes in their state of charge, monitored by positive voltage shifts of the photocurrent induced by pulsed illumination under constant D.C. bias, proportionally to SO<sub>2</sub> or NO<sub>2</sub> concentration in air. Chemically induced charges are negative and their surface densities are twice as high for SO<sub>2</sub> as those obtained for NO<sub>2</sub>, consistently with doubly charged anions; dynamic range is correspondingly halved. Both responses are mediated by associative chemisorption with oxygen. *Copyright © 2012 IFSA.*

**Keywords:** Chemical sensors; Gas detectors, MOS devices, NO<sub>2</sub>, SO<sub>2</sub>.

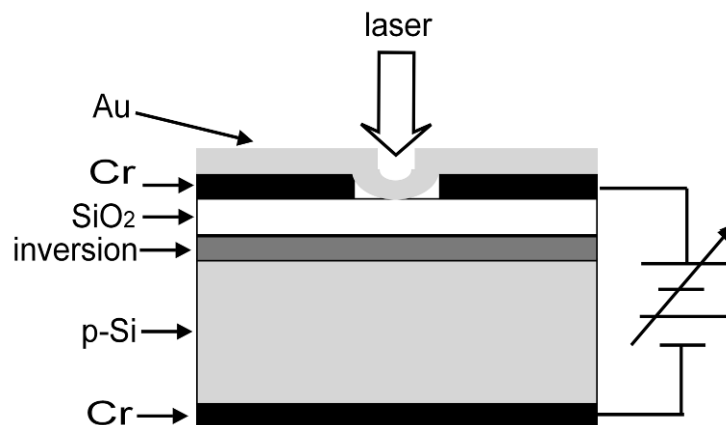
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### 1. Introduction

Chemically sensitive metal oxide semiconductor devices benefit from high selectivity, compared to purely resistive semiconducting sensors, prompting the early work [1] on Pd gate MOS H<sub>2</sub> sensors to be quickly followed by extensions to other hydrogen bearing compounds, such as NH<sub>3</sub> [2, 3] and SH<sub>2</sub> [4], which supply H<sup>+</sup> donor stimuli by dissociative chemisorption. However, the reciprocal acceptor stimuli have been essentially restricted to associative chemisorption of NO<sub>2</sub> on Au in the presence of oxygen [5], including weak responses to NO, deemed commensurate [6] with in situ oxidation to NO<sub>2</sub>. Recently [7], gold gate chemically sensitive field effect devices (CSFED) have been extended to SO<sub>2</sub> sensing, with potential applications in acid rain precursor detection. The experimental requirements for electrical characterization of acceptor stimuli, the influence of operating conditions, as well as the similarities of NO<sub>2</sub> and SO<sub>2</sub> chemisorption on gold, are addressed here.

## 2. Materials and Methods

Gap-gate MOS capacitors [8] were fabricated with 1-10  $\Omega\cdot\text{cm}$ , p-type, boron doped silicon wafers, thermally oxidized to 100 nm. Circular chromium electrodes, 2.5 mm in diameter and 600 nm thick, were deposited by D.C. magnetron sputtering, leaving a 250 micron gap, sufficiently narrow to allow the inversion layer to extend continuously beneath it under positive polarization (Fig. 1).



**Fig. 1.** Schematic cross section (not to scale) of a gap-gate MOS capacitor under positive bias with a continuous inversion layer underneath the gap.

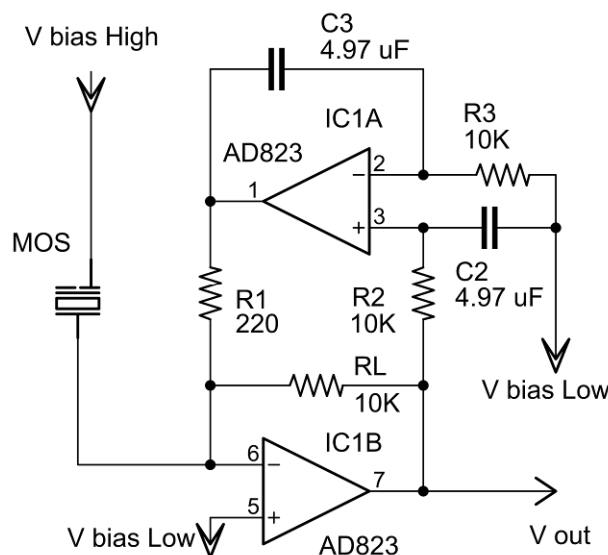
This configuration relieves the chemically sensitive material, in this case a discontinuous 8 nm layer of sputtered gold, of any electrical functionality, overcoming the limitations of nanometric metallic deposits, due to either poor thermal stability or high resistivity. Semiconducting or insulating materials may likewise be used as active materials in gap gate devices [8]. The capacitors were mounted on hybrid alumina substrates including contact pads and nichrome heaters, which were enclosed in an air tight stainless steel chamber, outfitted with ports for all electrical connections, Platinel II thermocouple thermometry and optical stimulation, as well as gas admission and venting. This assembly was supported on a micrometric X-Y stage, for alignment of the incident laser beam.

The state of charge of the device was examined by the pulsed illumination technique [9], as a function of constant applied bias, supplied by a KEITHLEY Source and Measure Model 2400 unit, which also allowed monitoring of the polarization current. The photocurrent ( $i$ ), induced by pulsed illumination with a HITACHI HL6501MG, 658 nm, 35 mW laser diode, thermally stabilized and driven by a THORLABS LTC-100-B unit, modulated at 1 kHz, was monitored phase sensitively with the HL6501 photodiode reference, on a Signal Recovery Model 1265 lock-in amplifier operated in external mode, after pre-amplification by an ad-hoc transimpedance current to voltage converter (Fig. 2), through calibrated either 10 or 5 k $\Omega$  load resistors ( $R_L$ ) and the respective signals were displayed on a Tektronix TDS 220 dual channel oscilloscope. Constant flow, totaling 100 cm<sup>3</sup>/minute, of synthetic air and 1000 ppm SO<sub>2</sub> in N<sub>2</sub> gas mixtures, was secured with independent MKS 1259 mass flow controllers. Measurements at fixed incremental positions across the gap were used to check the presence of a single bifurcation free maximum, which confirms the continuity of the inversion layer at the Si/SiO<sub>2</sub> interface under the gap [10]. Chemical response is conditioned by the thickness of the active layer. The nominal 8 nm value was obtained by piezogravimetric calibration and corresponds to the average grain size, measured by transmission electron microscopy, for noble metals sputtered under similar conditions. No response was observed for thinner deposits.

Depending strongly on the quality of the thermal oxide layer, D.C. polarization currents can easily exceed the mA range, at the comparatively high (150 °C) operating temperatures required to avoid



saturation of the sites available for analyte chemisorption [1]. Left unchecked, this leakage can exceed the compliance limit commensurate with  $R_L$ . The circuit illustrated in Fig. 2 overcomes this limitation by the introduction of an active filter in the feedback loop, in a manner similar [11] to that used for ambient illumination compensation, in photodiode pre-amplification applications; such that low frequency components, below 330 Hz in this case, flow through a low value feedback resistor ( $R_1$ ), whereas the higher frequency photocurrent signal (1 kHz) is channeled through the 10 k $\Omega$  load resistor ( $R_L$ ).



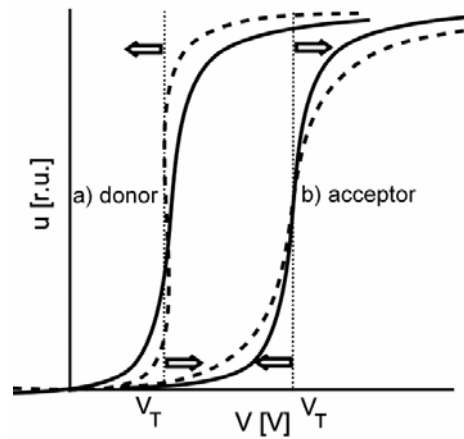
**Fig. 2.** Transimpedance voltage to current converter modified with an active feedback loop to compensate for high D.C. polarization current.

### 3. Results

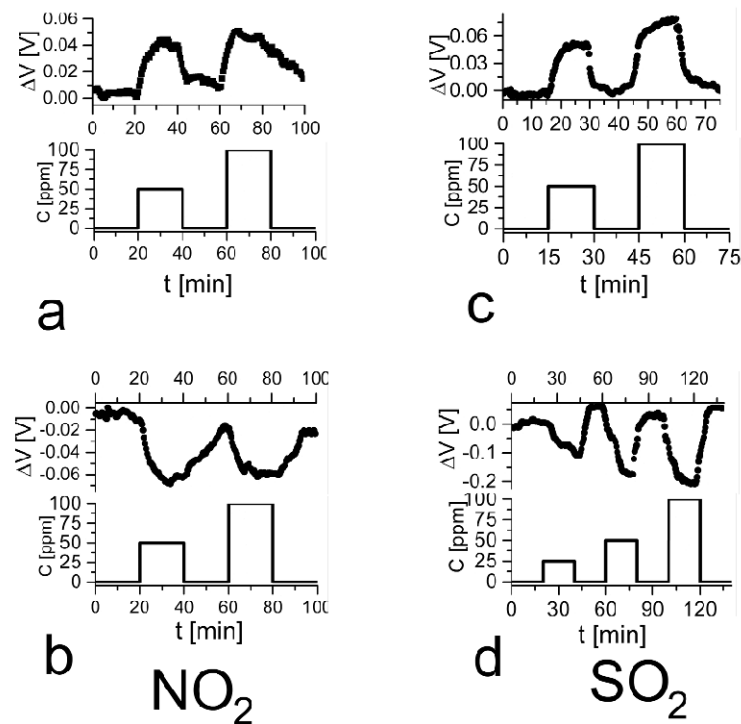
The bias dependence of the photocurrent ( $u$ ), under donor and acceptor stimulation, is schematically represented in Fig. 3 a and 3 b, respectively. Although  $u$ , in arbitrary units, is the experimentally monitored variable, the response is expressed in terms of the voltage shift ( $\Delta V$ ) for constant  $u$ , to provide a common scale for all measurements, which can either be obtained by calibration or measured directly through PID bias control [10]. Calibration of the  $u$ - $V$  dependence was employed in all measurements to ensure constant bias operation.

Above the threshold voltage ( $V_T$ ), with a fully formed inversion layer, the chemically induced positive charge accumulation associated with donor stimuli (Fig. 3a), such as hydrogen on palladium, enhances device inversion, hence lower bias voltage is necessary for constant  $u$ , than under the reference conditions. Below  $V_T$ , in the depletion regime, the positive chemically induced charges contribute to polarization and are compensated at the dielectric-semiconductor surface by negative charges, with diminished interface state population [12].

The behavior under acceptor stimulation (Fig. 3b), as exemplified by  $\text{NO}_2$  in air on gold gates, mirrors that of donors, because negative gate charge accumulation ensues from its acceptor character, hence increasing interface state population and  $u$  signal below  $V_T$ , consistently with negative  $\Delta V$  responses; whereas inversion above  $V_T$  is inhibited, inducing positive responses (cf. Fig. 3 b) [11]. Representative responses of gold gap-gate devices to  $\text{NO}_2$  and  $\text{SO}_2$  stimulation in air [7] are summarized in Fig. 4.



**Fig. 3.** Schematic representation of device responses (dashed lines) to a) donor and b) acceptor stimulation, showing reversals at the respective threshold voltages ( $V_T$ ). Arrows indicate the sense of the voltage shifts.

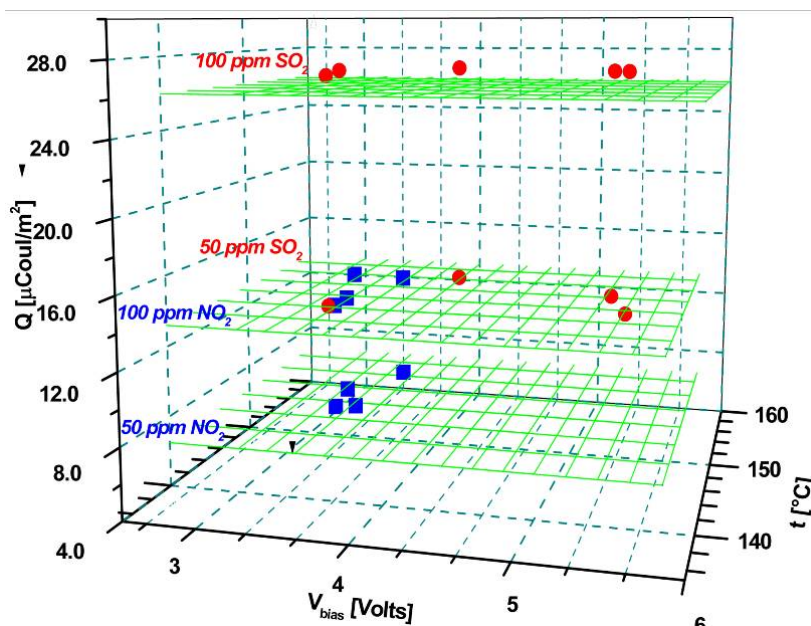


**Fig. 4.** Temporal response to chemical stimulation by NO<sub>2</sub>, for MOS gap-gate devices held at: (a) 147 °C, biased at 3.5 V above  $V_T$ ; (b) 167 °C, biased at 3.3 V, below  $V_T$ ; and by SO<sub>2</sub>, (c) 160 °C, biased at 6.3 V, above  $V_T$ ; (d) 160 °C, biased at 3.1 V, below  $V_T$ .

The photocurrent bias dependence at 160 °C for SO<sub>2</sub> has an acceptor character, similar to NO<sub>2</sub> [13], leading to reversals of the positive temporal response (Fig. 4 a and 4 c), when biased below the threshold voltage (Fig. 4 b and 4 d). Since  $V_T$  increases with temperature, the sign of the response obtained at fixed bias may be reversed by increased operating temperature [13]. In all instances, responses are clearly dependent on operating conditions, such that their sensing application is subject to calibration. Sensitivity increases from zero at  $V_T$ , proportionally to increased positive bias (Fig. 3). A more fundamental parameter is afforded by the charge accumulated at the semiconductor-dielectric interface, which equals that transported through the device and corresponds to [9]:

$$\Delta Q = C_{SC} \Delta\psi + C_0 \Delta V, \quad (1)$$

where  $C_{SC}$  is the capacitance due to the parallel differential ( $C_D$ ) and surface state ( $C_{SS}$ ) components, which are sensitively dependent on device polarization state, whereas  $C_O$  is the geometric capacitance;  $\Delta\psi$  the surface potential modulation, and  $\Delta V$  the measured response, respectively. The capacitance and surface potential parameters can be evaluated as a function of voltage bias and temperature, to calculate the charge induced chemically by any given stimulus [13]. The results for 50 and 100 ppm of  $SO_2$ , as well as  $NO_2$  stimulation (Fig. 5) demonstrate that, regardless of operating conditions, the charge induced per unit stimulation is twice as high for  $SO_2$  than for  $NO_2$  [7], consistently with doubly charged anions.



**Fig. 5.** Chemically induced charge, measured with representative gap-gate devices operated under different conditions, in response to 50 ppm and 100 ppm of  $NO_2$  (blue squares) and  $SO_2$  (red circles) in air, as a function of D.C. bias and operating temperature. Planes (green grid) at 6.5, 13 and 26 [ $\mu\text{Coul}/\text{m}^2$ ] are aids to the eye.

The maximum charge which can accumulate at the interface of MOS capacitors limits the dynamic range of these devices and depends on the doping level of the semiconductor ( $NA_{2/3}$ ) [14], which corresponds to approximately  $10^{11} \text{ cm}^{-2}$ , in the devices of this work. However, sensor response at saturation is dramatically different for donor and acceptor stimuli. The accumulation of positive charges at the gate-dielectric interface for donor stimuli is sustained at this limit with increasing analyte concentration, and saturation is manifest in the absence of subsequent response increases, whereas the negative charge of acceptor stimuli is drained by forward bias, if the capacitance is insufficient to hold it, thus eliminating further response. This behaviour conditions the applicable concentration range, particularly for anions of multiple charge, decreasing the maximum measurable concentration from 200 ppm for  $NO_2$  to 100 ppm, in the case of  $SO_2$ , with the detection limit at 5% of the corresponding range.

#### 4. Discussion

The consistency found for the ratio of charge per unit stimulus in  $SO_2$  and  $NO_2$  would not be possible in the absence of a common chemisorption mechanism over nanometric gold. This issue has received increased attention over the past two decades, due to mounting evidence, which has challenged the formerly widely held belief that gold lacks catalytic properties.

Adsorption studies on single crystal Au surfaces, as well as density functional calculations have shown that dissociative adsorption of oxygen is unlikely and that low coordinated gold atoms, such as those present on steps and kinks, are required for high catalytic activity [15]. On polycrystalline gold [16], three desorption states are recognized by temperature programmed desorption (TPD) of NO<sub>2</sub>, with energies between 11 and 17 kcal/mol, commensurate with the operating temperatures required by chemically sensitive field effect devices (CSFED), and high resolution electron energy loss spectra (HREELS) are consistent with a AuO<sub>2</sub>O'-nitrito surface chelate, corroborated by similar investigations [17] on Au (111) surfaces, which attribute C<sub>2v</sub> symmetry to this complex. Additional evidence [18] of the same adsorbate has been obtained by infrared reflection absorption spectroscopy (IRAS) and suggests that bonding to the surface is oxygen mediated and that reactions with coadsorbed water lead to nitrous and nitric acids. The reaction of NO<sub>2</sub> with water has likewise [19] been shown to produce a large concentration of oxygen adatoms on Au (111), which enhance its catalytic properties.

The mediating role of oxygen adatoms in NO<sub>2</sub> chemisorption [20] is consistent with the required presence of oxygen for CSFED detection, which is affected by inordinately long transient response and relaxation in its absence [12], however, the surface analytic techniques employed so far are ill suited for the detection of charge. Ab initio calculations for nitric oxide adsorption on gold [21] suggest that binding occurs through soft electrostatic interactions and that charge transfer is enhanced for ionic species, compared to neutral complexes.

Finally, recent work on supported catalysts [22] has demonstrated that "the oxide support can be much more than a simple spectator", since the deposition of Au nanoparticles on TiO<sub>2</sub> produces a system with increased capacity for SO<sub>2</sub> adsorption and reaction, enhancing oxygen vacancy migration from the bulk of the oxide to the surface. Photoemission experiments, in this case, support the formation of SO<sub>4</sub>.

Conductimetric semiconductor sensors [23] of various configurations have also shown sensitivity to NO<sub>2</sub>. However, these comparatively simpler devices respond to changes in semiconductor surface state occupancy, which alters both carrier density and mobility, to induce positive or negative resistivity changes, not uniquely dependent on analyte type and concentration; consequently, resistive devices are ubiquitous but selectivity is compromised by complex chemical sample matrices. By comparison, the MOS devices described here are charge transducers, which require specific chemisorption mechanisms to induce ionic analyte species, thus enhancing selectivity and yielding information on analyte charge sign and surface density, independently of device operating conditions.

## **5. Conclusions**

NO<sub>2</sub> and SO<sub>2</sub> chemisorption on nanometric gold gap-gate CSFED constitute acceptor stimuli, leading to accumulation of negative charge, manifest in positive shifts of the photocurrent bias dependence above the threshold voltage. The charge per unit stimulus concentration for SO<sub>2</sub> closely doubles that for NO<sub>2</sub>, consistently with the charge associated with the respective anions. The probable response mechanism involves oxygen adatoms, to provide suitable adsorption sites for either compound. Comparative immunity to donor stimulus interference favors their potential application in micro-equivalent acid-rain precursor monitoring, however cross-sensitivity is unavoidable, because both acceptor stimuli share a common chemisorption mechanism on gold.

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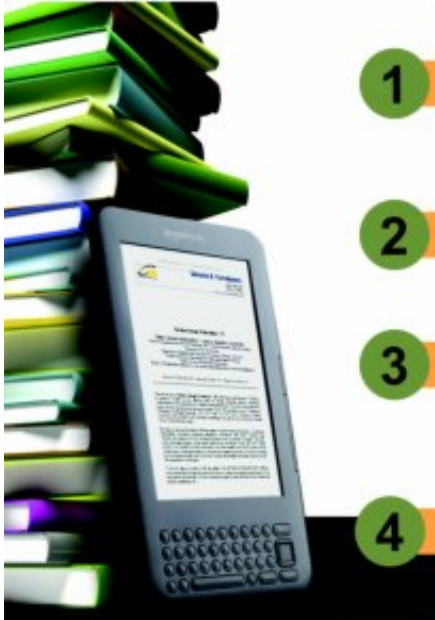
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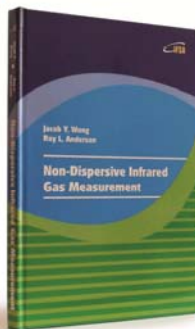
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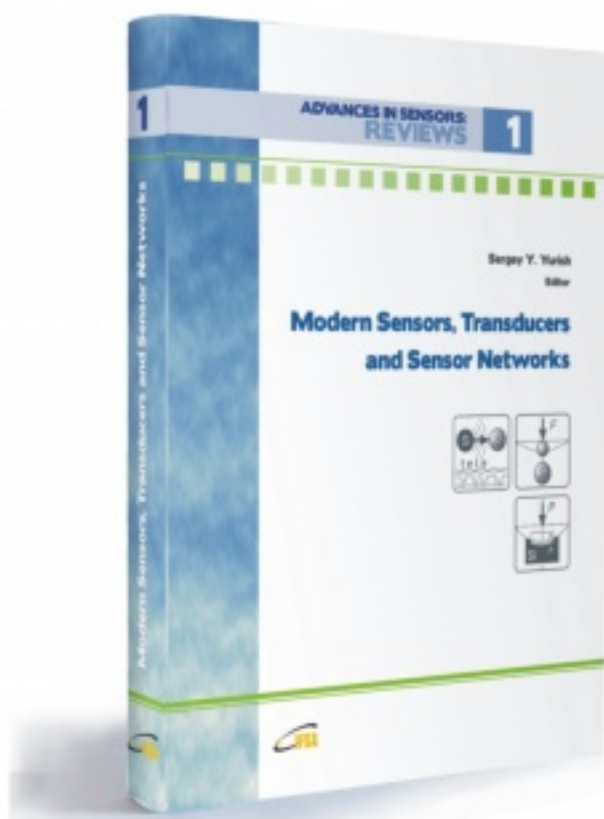
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