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Authors

Sadek, A Z
Baker, Christina Opimo
Powell, D A
[et al.](#)

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Polyaniline Nanofiber Based Surface Acoustic Wave Gas Sensors—Effect of Nanofiber Diameter on H₂ Response

Abu Z. Sadek, *Student Member, IEEE*, Christina O. Baker, David A. Powell, *Member, IEEE*, Wojtek Wlodarski, *Member, IEEE*, Richard B. Kaner, and Kourosh Kalantar-zadeh, *Member, IEEE*

Abstract—A template-free rapidly mixed reaction was employed to synthesize polyaniline nanofibers using chemical oxidative polymerization of aniline. Hydrochloric acid (HCl) and camphor sulfonic acid (CSA) were used in the synthesis to obtain 30- and 50-nm average diameter polyaniline nanofibers. The nanofibers were deposited onto layered ZnO/64° YX LiNbO₃ surface-acoustic-wave transducers. The sensors were tested toward hydrogen (H₂) gas while operating at room temperature. The dopant for the polyaniline nanofiber synthesis was found to have a significant effect on the device sensitivity. The sensor response was found to be larger for the 50-nm diameter CSA-doped nanofiber based sensors, while the response and recovery times were faster for the 30-nm diameter HCl-doped nanofibers.

Index Terms—Conducting polymer, H₂ sensor, polyaniline nanofiber, rapidly mixed, surface acoustic wave (SAW).

I. INTRODUCTION

CONDUCTING polymers have received increasing interest as an alternative to metal oxide semiconductors for smart sensors. This is due to their room-temperature operation, low fabrication cost, ease of deposition onto a wide variety of substrates, and their rich structural modification chemistry [1], [2]. Among the family of conducting polymers, polyaniline is one of the most highly studied materials because of its simple synthesis, environmental stability, and straightforward non-redox acid doping/base dedoping process to control conductivity [3], [4]. By changing the doping level and morphology, the conductivity of polyaniline can be tuned [5]. Polyaniline has three main oxidation states: fully reduced leucoemeraldine, half-oxidized emeraldine (Fig. 1), and fully oxidized pernigraniline. Polyaniline in the emeraldine oxidation state can be reversibly switched between electrically insulating emeraldine base (Fig. 1: top) and conducting emeraldine salt forms (Fig. 1: middle and bottom) [6], [7]. The conductivity of polyaniline can

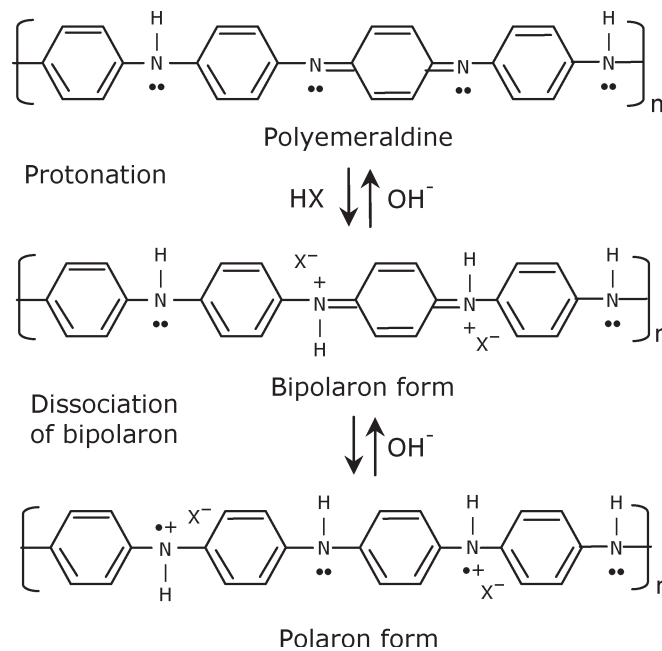


Fig. 1. Polyaniline in the emeraldine oxidation state can exist in either its undoped (top) or doped form. The doped intermediate bipolaron form (middle) converts rapidly to the electronic conducting form (bottom).

be varied by ten orders of magnitude by controlling the degree of imine nitrogen protonation (doping). The doping level can be altered by using variable amounts of a wide variety of protonic acids. The dopants can be removed by a reversible reaction with any strong base such as ammonium hydroxide.

The conductivity of polyaniline also depends on the oxidation state of the main polymer chain [8]. Reduction or oxidation of the conducting polyaniline emeraldine salt by chemical or electrochemical processes changes the polyaniline to the insulating leucoemeraldine or pernigraniline oxidation states, respectively. As a result, redox active chemicals and gases can affect the conductivity of the polyaniline by changing its inherent oxidation state. Neutral volatile organic compounds are able to change the conductivity of the doped polyaniline films as a result of polymer swelling, chain alignment, crystallization, solvation or by affecting the doping level [9]–[13].

Conducting polymer gas sensors based on measuring resistance changes in thin-film structures have been studied by a number of researchers [14]–[17]. A detailed review of polymer and polyaniline sensors can be found in [18] and [19], respectively.

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A. Z. Sadek, D. A. Powell, W. Wlodarski, and K. Kalantar-zadeh are with the School of Electrical and Computer Engineering, RMIT University, Melbourne 3001, Australia (e-mail: sadek@ieee.org; david.powell@ieee.org; ww@rmit.edu.au; kourosh.kalantar@rmit.edu.au)

C. O. Baker and R. B. Kaner are with the Department of Chemistry and Biochemistry and California NanoSystems Institute, University of California, Los Angeles, CA 90095-1569 USA (e-mail: cbaker@chem.ucla.edu; kaner@chem.ucla.edu).

Color versions of Figs. 3, 4, 7, and 8 are available online at <http://ieeexplore.ieee.org>.

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Polyaniline nanofibers, with diameters in the 30–100 nm range, possess much larger surface to volume ratios [20] and permit easier addition of surface functionality and interaction compared with traditional polyaniline which is highly agglomerated and poorly dispersible in water. Polyaniline nanofibers have a cylindrical morphology and will form porous structures when deposited as thin films. This structure allows easy diffusion of gas molecules into and out of the film, and the nanoscale fiber diameters lead to a rapid diffusion of gas molecules into the polyaniline. As a result, most reports indicate that nanostructured polyaniline outperforms conventional polyaniline for gas sensing applications [21]–[25].

Conventional chemical synthesis of polyaniline uses aniline, a strong acid dopant and an oxidant that is slowly added into the reaction at low temperature with thorough stirring, which results in irregular granular morphology [8], [26]. The secondary overgrowth of the nanofibers leads to agglomerated particles, so nanofibers can be obtained by suppressing this growth. A rapidly mixed reaction is the simplest for bulk synthesis of polyaniline nanofibers without requiring any template, surfactant, special dopant, or organic solvent [27]. The diameter of the nanofibers is strongly related to the dopant acid used in the polymerization process [28]. A detailed review of polyaniline nanofiber synthesis approaches can be found in [26] and [29].

For gas sensing, polyaniline nanofibers can be deposited onto the active area of a surface acoustic wave (SAW) device as a sensing layer. In a SAW device, the change in electrical conductivity of the sensing layer perturbs the velocity of the propagating acoustic wave due to piezoelectric effects. The center frequency of a SAW device is given by the equation $v = fp$, where v is the surface wave velocity, f is the center frequency, and p is the interdigitated transducer period. Therefore, the variation in velocity can be monitored by measuring the changes in resonant frequency of the SAW device. This change in frequency is proportional to the concentration of analyte present in the environment.

In this paper, polyaniline nanofibers were synthesized from a chemical oxidative polymerization of aniline using a template-free rapidly mixed reaction. The dopant acids hydrochloric acid (HCl) and camphor sulfonic acid (CSA) were used to grow doped forms of polyaniline nanofibers having different diameters. A polyaniline nanofiber sensitive layer was formed on a layered SAW transducer for H_2 gas sensing. Finally, the responses of the sensors to different H_2 gas concentrations were determined and compared.

II. EXPERIMENTAL

In this paper, the layered SAW devices are used as the transducing platform. The substrate is 64° YX $LiNbO_3$ with an intermediate ZnO protective layer. Shear-horizontal leaky surface acoustic wave is the dominant mode in this layered substrate. The sensors consisted of two-port resonators with 38 electrode pairs in the input and output interdigital transducers (IDT), 160 electrodes in each reflective grating, $700 \mu m$ aperture width, and a periodicity of $40 \mu m$. The center-to-center distance between the IDTs was $1920 \mu m$. The IDTs were formed by patterning an 80 nm layer of gold and a

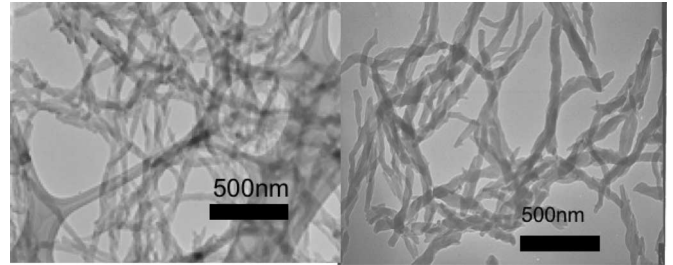


Fig. 2. TEM images of HCl-doped (left on lacey carbon grid) and CSA-doped (right on carbon film grid) polyaniline nanofibers.

20 nm titanium layer. The titanium layer was added to improve adhesion of the gold film.

A ZnO layer of $1.2 \mu m$ in thickness was deposited on the surface of the 64° YX $LiNbO_3$ substrates to form the layered structure. A radio-frequency (RF) magnetron sputterer was used to deposit from a 99.99% pure ZnO target with RF power of 120 W. The sputtering gas was 40% O_2 in Ar with a pressure of 10^{-2} torr, the substrate temperature was $260^\circ C$, and the deposition time was for periods of 60 min.

Previous approaches for making polyaniline nanostructures often require structure-directing templates, which must be removed at the end of the reaction. Recently, we have introduced a template-free rapidly mixed reaction approach to synthesize polyaniline nanofibers using the rapid chemical oxidative polymerization of aniline [27]. The polymerization is performed in an aqueous solution where aniline is rapidly polymerized in 1 M acid by the quick addition of the oxidant (ammonium peroxydisulfate $[(NH_4)_2S_2O_8]$) and dissolved in 1 M acid. The aniline-to-oxidant ratio is approximately four to one, respectively, and the aniline concentration is 0.5 M. Due to the immediate interaction between the monomer and the oxidant, the primary reaction product, nanofibers, is the main morphology present. After completion of the reaction, the product is collected for purification. Filtration, centrifugation, or dialysis with water gives pure doped polyaniline that can be dedoped by washing or dialyzing with aqueous ammonia or sodium hydroxide. In this paper, the polyaniline is cleaned in a five-step centrifugation process. Centrifugation is done until a dense pellet forms. Resuspension is done with water on the first centrifugation, then 1 M doping acid. Water is used for the remaining resuspension steps with the final pellet resuspended to 2 g/L polyaniline. The nature of the acid used in the synthetic process was found to have a significant effect on the physicochemical properties of the resulting nanofibers. The average diameter of the polyaniline nanofibers is about 30 nm when HCl is used as a dopant acid during synthesis and about 50 nm when CSA is used as the dopant acid during synthesis, with lengths up to several micrometers. Transmission-electron-microscopy (TEM) images (Fig. 2) reveal that the polyaniline nanofiber layers consist of a large quantity of wirelike nanostructures.

Doped polyaniline nanofiber dispersions were drop cast onto the active area of the transducers using a micropipette. They were then left to dry in a clean dry environment for one day. Two types of sensors were created with a polyaniline nanofiber sensitive layer: one synthesized with an HCl dopant and the other with a CSA dopant. A schematic structure of the SAW

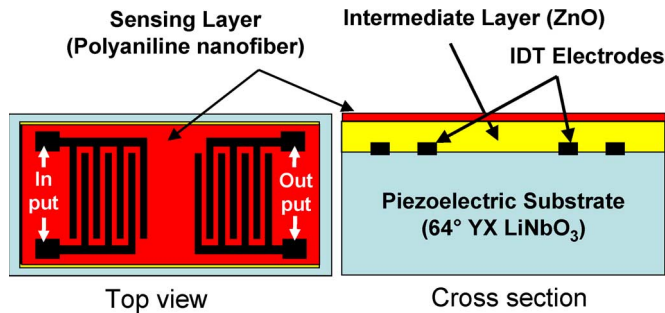


Fig. 3. Schematic structure of the SAW sensor.

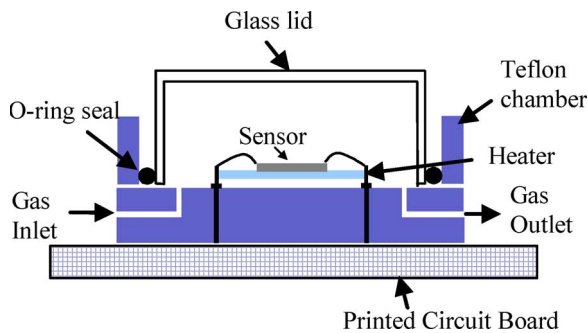


Fig. 4. Experimental setup for sensor testing [6].

sensor is shown in Fig. 3. The average thickness measured for both nanofiber thin films is $0.3 \mu\text{m}$, and the deviation in thickness across the active area of the SAW transducer is $0.05 \mu\text{m}$. The gas sensors are made up of two important physical components: the sensitive polyaniline nanofiber layer, which interacts with the gas media by changing conductivity, and the SAW transducer, which changes its operating frequency with any conductivity change. Using the layered SAW device as a positive feedback element in a closed-loop circuit with an amplifier, an oscillator was formed. A frequency counter was used to measure the operational frequency of the transducer upon exposure to the H_2 gas. The operational frequency of both sensors was found to be approximately 108.2 MHz in dry synthetic air at room temperature.

The sensors were mounted inside an enclosed environmental cell (Fig. 4). A computerized mass-flow-controller system was used to vary the concentration of H_2 gas in synthetic air and to measure the operating frequency of the sensors. The gas mixture was delivered at a constant flow rate of 0.2 L/min. The sensor responses were displayed in real time and saved for off-line processing and analysis. Gas exposure time was fixed for each pulse of H_2 gas, and the cell was purged with synthetic air between each pulse to allow the surface of the sensor to recover to atmospheric conditions. The HCl-doped sensor was exposed to a hydrogen gas pulse sequence of 0.06%, 0.125%, 0.25%, 0.50%, 1%, and 0.06% concentrations, and the CSA-doped sensor was exposed to a hydrogen gas pulse sequence of 0.06%, 0.125%, 0.25%, 0.50%, 1%, and 0.125% in synthetic air at room temperature. A Fluke high-resolution counter (PM66860B) was used to measure the operational frequency.

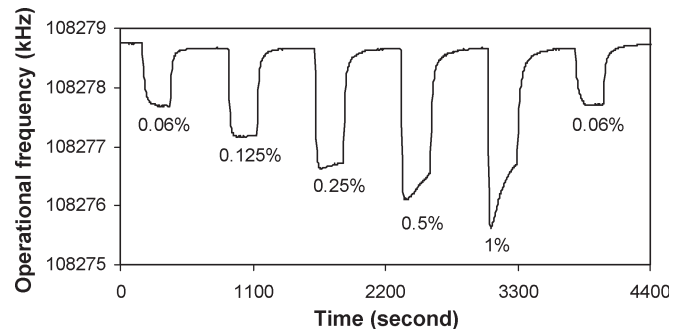


Fig. 5. Dynamic response of an HCl-doped polyaniline nanofiber based $\text{ZnO}/64^\circ \text{YX LiNbO}_3$ SAW sensor toward H_2 at room temperature [6].

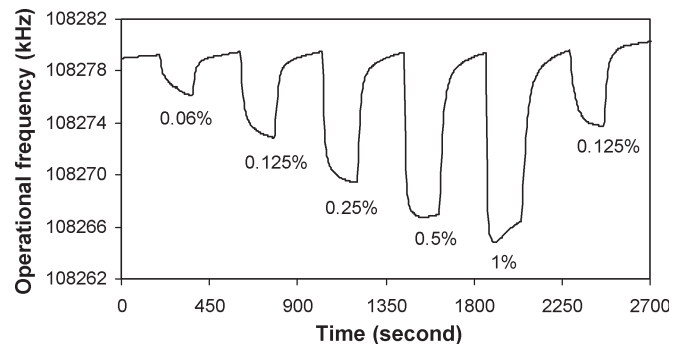


Fig. 6. Dynamic response of a CSA-doped polyaniline nanofiber based $\text{ZnO}/64^\circ \text{YX LiNbO}_3$ SAW sensor toward H_2 at room temperature.

III. RESULTS

The dynamic responses of the HCl- and CSA-doped polyaniline nanofiber sensors to different H_2 concentrations are shown in Figs. 5 and 6, respectively. The introduction of H_2 gas to the sensor surface causes reduction of the device's resonant frequency for both of the sensors. With hydrogen exposure, the conductivity of the polyaniline nanofiber layer increases, resulting in a decrease in the acoustic-wave velocity, thereby decreasing the resonant frequency. The results are similar to those obtained by the authors for conductometric measurements on identical HCl-doped sensors. Due to this similarity, the high electromechanical coupling coefficient of LiNbO_3 and the low molecular weight of H_2 molecules, it is assumed that the conductivity change is more significant than the mass change.

The mechanism for the conductivity change in polyaniline due to the interaction with hydrogen is still unclear. The SAW-device results and a separate measurement of resistivity show that resistance decreases in doped polyaniline on exposure to H_2 . The hydrogen may form a bridge between nitrogen atoms on two adjacent chains or there may be a partial protonation of some of the imine nitrogens [30]. It has also been suggested that the resistance change is caused by the formation of water [31].

The sensor response is defined as the variation in operating frequency of oscillation due to the interaction with the target gas. The responses are 3 and 14.6 kHz toward 1% of H_2 for HCl- and CSA-doped polyaniline nanofiber sensors, respectively. The response magnitude variation for both

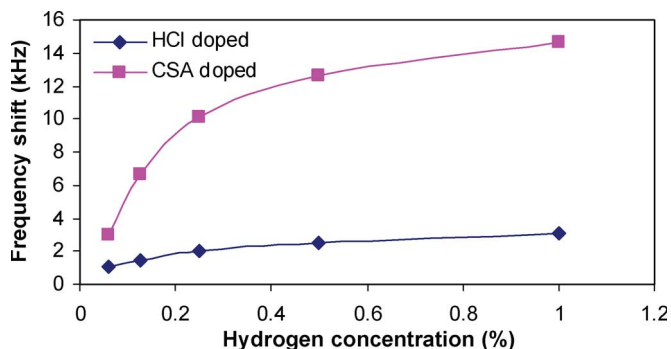


Fig. 7. Frequency shift (in kilohertz) versus H₂ gas concentrations (in percent) at room temperature.

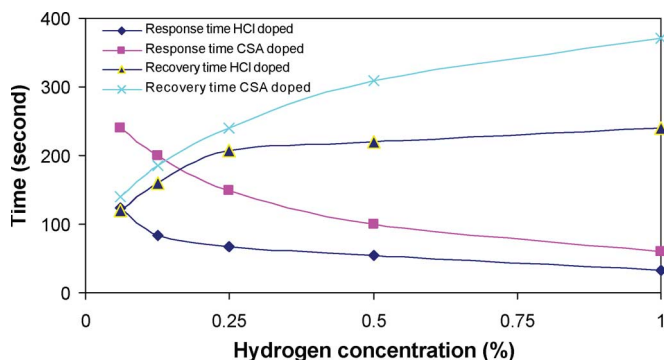


Fig. 8. Response and recovery times (in seconds) versus H₂ gas concentrations (in percent) at room temperature.

sensors to different H₂ concentrations is shown in Fig. 7. The frequency shift increases nonlinearly with the increase of H₂ concentrations for both of the sensors, saturating for large concentrations. The sensitivity of CSA-doped nanofiber sensors is greater than that of the HCl-doped sensors, particularly for higher H₂ concentrations.

The 90% response and recovery time of the sensors are shown in Fig. 8. Relatively fast response and recovery times are observed for both of the sensors. With increasing H₂ concentrations, the response time decreases and recovery time increases for both sensors. However, the HCl-doped sensor has faster response and recovery times compared to the CSA-doped sensor for all concentrations of H₂. This is consistent with the NH₃ sensing results of Liu *et al.* [32], who showed that the response time of the polyaniline nanowire-based sensors with various diameters corresponds to the radius-dependent diffusion time of ammonia gas into the wires.

Three factors may affect the differences observed with HCl and CSA synthesized doped polyaniline nanofibers: 1) The diameter of the nanofibers provides a differing amount of surface area for the H₂ to interact with, 2) the differences in volatility and mobility of the dopants, which may cause variable speeds of reaction, and 3) there may be variation in the doping level of the dried films.

- 1) The diameter of the HCl-doped nanofibers is approximately three fifths that of the CSA-doped nanofibers. This could play a role in the observed differences in sensitivity and response and recovery times of the sensors. The faster

response and recovery time of the HCl-doped sensor can be explained by faster adsorption of H₂. The decrease in sensitivity could be explained by faster gas penetration into the fiber. The higher surface area of the HCl-doped nanofibers allows gas molecules to penetrate the fibers quickly, so there is less delay in the reaction time and saturation.

- 2) Since HCl is a small and volatile dopant, it may be more mobile than bulky CSA, which is a nonvolatile organic dopant. The mobility difference may increase the speed of interaction with hydrogen but at the disadvantage of having less stable devices over long periods of time. We observed an increased stability of the baseline frequency with the CSA-doped polyaniline nanofibers versus the HCl-doped nanofibers. The latter slightly increased in frequency over several weeks. This may be attributed to the volatility of HCl.
- 3) Slight variations in the purification can result in a difference in the number of undoped imine nitrogens between the CSA and HCl-doped polyaniline films. This can cause saturation differences with either a doping mechanism or a chain-to-chain interaction mechanism.

Reproducibility was observed as indicated when a second pulse of 0.06% H₂ for the HCl-doped nanofiber film and 0.125% H₂ for the CSA-doped nanofiber film was introduced into the sensor chamber. It was found that the polyaniline nanofiber based sensors produce repeatable responses of the same magnitude with good baseline stability.

IV. CONCLUSION

SAW gas sensors have been fabricated based on polyaniline nanofibers synthesized by a template-free rapidly mixed polymerization of aniline using HCl and CSA as the dopant acids. Novel SAW gas sensors based on these nanofibers have been exposed to different concentrations of H₂ gas at room temperature. The sensor response, which is defined as the variation in resonant frequency, was 3 and 14.6 kHz toward 1% of H₂ at room temperature for HCl- and CSA-doped polyaniline nanofiber sensors, respectively. A relatively fast response and recovery time with good repeatability and baseline stability were observed for both sensors. Such interesting sensing characteristics at room-temperature operation could pave the way to fabricate low power sensors using inexpensive polyaniline based nanofibers.

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Abu Z. Sadek (S'06) received the B.Sc. degree in electrical and electronics engineering from Bangladesh University of Engineering and Technology (BUET), Dhaka, Bangladesh, in 1998 and the M.E. degree in telecommunications engineering from the University of Melbourne, Parkville, Australia, in 2002. He is currently working toward the Ph.D. degree at the Sensor Technology Laboratory, School of Electrical and Computer Engineering, RMIT University, Melbourne, Australia.

His research interests include chemical and biochemical sensors, microtechnology and nanotechnology, acoustic propagation, and conducting polymers.



Christina O. Baker received the B.S. degree in chemistry from Georgia Institute of Technology, Atlanta, in May 2003. She is currently working toward the Ph.D. degree in inorganic chemistry at the University of California, Los Angeles (UCLA), working with Prof. R. Kaner on conducting polymers.

Her current research involves the development of polyaniline nanofibers and metal nanoparticle polyaniline nanofiber composites for applications in nonvolatile memory, chemical sensors, and actuators.



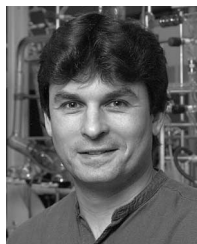
David A. Powell (S'01–M'06) received the Ph.D. degree from the School of Electrical and Computer Engineering, RMIT University, Melbourne, Australia. His thesis is entitled "Modelling of Layered Surface Acoustic Wave Resonators for Liquid Media Sensing Applications."

His interests include chemical and biosensing, microtechnology, acoustic and electromagnetic propagation, periodic media, numerical computing, and novel materials.



Wojtek Wlodarski (M'72) received the M.Sc.E.E., Ph.D, and D.Sc. degrees, all from Warsaw University of Technology, Warsaw, Poland, in 1962, 1971, and 1980, respectively.

He has worked in the areas of sensor technology and instrumentation for over 30 years. He is a Professor with RMIT University, Melbourne, Australia, and heads the Sensor Technology Laboratory at the School of Electrical and Computer Engineering. He has published four books and monographs and over 400 papers and holds 29 patents.



Richard B. Kaner received the Ph.D. degree in inorganic chemistry from the University of Pennsylvania, Philadelphia, in 1984, followed by two and a half years of postdoctoral research at the University of California, Berkeley.

He is a Professor of chemistry and materials science and engineering with the University of California, Los Angeles (UCLA). He joined UCLA as an Assistant Professor in 1987, earned tenure in 1991, and became a Full Professor in 1993.

Dr. Kaner received awards from the Dreyfus, Fulbright, Guggenheim, Packard, and Sloan Foundations for his work on new routes to refractory materials, including high-temperature ceramics, intercalation compounds, fulleride superconductors, superhard materials, and conducting polymers.



Kourosh Kalantar-zadeh (M'94) received the M.Sci. degree in telecommunications from Tehran University, Tehran, Iran, in 1997 and the Ph.D. degree in micro/nano-electronics and bioelectronics from RMIT University, Melbourne, Australia, in 2002.

He is a tenured academic at the School of Electrical and Computer Engineering, RMIT University, Melbourne, Australia. His research interests include chemical and biochemical sensors, nanotechnology, microelectromechanical systems, thermoelectric materials, electronic circuits, and microfluidics. He holds three patents. He has published more than 80 scientific papers in refereed journals and in the proceedings of international conferences. He is currently authoring a book entitled "Nanotechnology Enabled Sensors."

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