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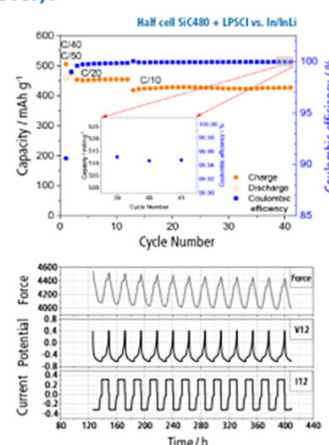
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Preparation of Pd Thin Films and their Application in Surface Acoustic Wave Devices for Sensing Hydrogen

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Hydrogen has significant applications in diverse sectors ranging from the petroleum industry to fuel cells, chemical food processing, metallurgy and pharmaceutical industries. Hydrogen serves as a rocket fuel and has a host of other applications in aerospace such as powering life support systems and computers. Fabrication of semiconductor circuits in the electronics industry requires the use of a controlled atmosphere which often contains hydrogen.

Although this gas is a lucrative, carbon-free fuel with important applications, there are risks involved with its use. Hydrogen is a colorless, odorless gas, which makes it difficult to detect. It is an asphyxiant and is extremely flammable in air (1). To limit the hazards associated with using this potentially explosive gas, reliable detectors are needed. A dependable hydrogen sensor should be sensitive and specific to hydrogen only in order to avoid the problem of false alarms. The response should also be fast and reversible. Years of research have been dedicated to the development of fast, reusable, economical hydrogen sensors capable of detecting leaks at low concentrations.

Pd can absorb about 900 times its own volume of hydrogen leading to the formation of PdH_x which occurs in two steps.(1) First, hydrogen adsorbs onto the surface of Pd and then chemisorbs in to the subsurface metal layer and diffuses into the bulk leading to hydride formation. PdH_x is known to exist predominantly in the α (conductive) phase at room temperature. Adsorption and subsequent absorption of hydrogen in α Pd increases the concentration of hydrogen atoms to a critical value, at which point the hydride transforms into the β phase, characterized by an expansion of 3% in length and 10 % in volume(4). This change is reversible as hydrogen easily desorbs from the β phase surface at room temperature, restoring the α Pd phase. The reversibility of this hydrogen induced lattice expansion (HILE) process makes Pd an ideal material for the fabrication of hydrogen sensors (4, 6).

SAW devices are fabricated on a planar lithium niobate substrate with two closely spaced split electrode Ti/Al transducers at one end of the device and a nonsplit electrode reflector located at the opposite end. The Pd thin film is deposited on the delay line in between the transducers and the reflector (2, 3). An acoustic wave is launched from one transducer, propagating across the surface of the device and traveling through the region of the device with the thin Pd film (2, 3). Any physical/chemical modification in the region near the surface of the substrate or of any sensing film deposited on the substrate will perturb the propagating acoustic wave and alter its velocity, phase and/or amplitude. These changes are reflected as changes in the radio frequency response of the device, which is used for detection.

The characteristics of the surface acoustic wave are sensitive to changes in the mass loading, viscoelastic properties and/or electrical conductivity of the sensing layer. The SAW device is therefore sensitive to any physical or chemical process that changes these film properties. The interaction with hydrogen induces a change in the conductivity of the Pd sensing layer. The effects of mass loading and changes in viscoelastic properties will also be investigated.

Pd nanoparticles with diameters ranging between 10-15 nm were synthesized using the method proposed by Teranishi. (5). The sizes of these nanoparticles were determined using Transmission Electron Microscopy (TEM) and Atomic Force Microscopy (AFM). The Pd nanoparticles prepared using this method has an organic protective layer of polyvinylpyrrolidone (PVP), a polymer which needs to be removed prior to hydrogen exposure. This is required for the formation of electrical contacts that contribute to conductivity pathways, either in an ohmic transport regime when actual mechanical contact between particles becomes possible following lattice expansion or in a quantum mechanical tunneling regime where the electron density distributions overlap. Thin films of the Pd nanoparticles were prepared by drop casting. The effects of ozone and plasma cleaning on the resultant response of the film to hydrogen were investigated. The sensitivity of the ozone or plasma treated films to hydrogen was investigated through exposure to 4% H₂ in N₂, the explosion threshold, in an environmental chamber. Morphological changes were monitored through *in situ* AFM. Images taken before, during and after H₂ exposure can reveal reversible morphological changes. After identifying the optimal hydrogen sensitive Pd films similar films were deposited on SAW devices for testing the real-time response.

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REFERENCES

1. Adams, B. D.; Chen, A., The role of palladium in a hydrogen economy. *Materials Today* 14, (6), 282-289.
2. Buvailo, A.; Xing, Y.; Hines, J.; Borguet, E., Thin polymer film based rapid surface acoustic wave humidity sensors. *Sensors and Actuators B: Chemical* 156, (1), 444-449.
3. Buvailo, A. I.; Xing, Y.; Hines, J.; Dollahon, N.; Borguet, E., TiO₂/LiCl-Based Nanostructured Thin Film for Humidity Sensor Applications. *ACS Applied Materials & Interfaces* 3, (2), 528-533.
4. Kalli, K.; Othonos, A.; Christofides, C., Characterization of reflectivity inversion, alpha- and beta-phase transitions and nanostructure formation in hydrogen activated thin Pd films on silicon based substrates. *Journal of Applied Physics* 2002, 91, (6), 3829-3840.
5. Teranishi, T.; Miyake, M., Size Control of Palladium Nanoparticles and Their Crystal Structures. *Chemistry of Materials* 1998, 10, (2), 594-600.
6. Xu, T.; Zach, M. P.; Xiao, Z. L.; Rosenmann, D.; Welp, U.; Kwok, W. K.; Crabtree, G. W., Self-assembled monolayer-enhanced hydrogen sensing with ultrathin palladium films. *Applied Physics Letters* 2005, 86, (20), 203104-3.