Optical Characterisation of RF Sputter Coated Palladium Thin Films for Hydrogen Sensing


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1. Motivation
Reliable hydrogen detection technologies required for safety applications

- Hydrogen suggested as future fuel source
- Hydrogen explosive at 4 - 97% concentration in air
- Most systems based on the absorption of hydrogen in palladium (Pd)
- Optical system preferable for safety reasons:
  - No heating
  - Zero electrical charge
- Optically well characterised homogeneous thin Pd films required
- Little agreement in literature on the optical properties of thin film Pd

2. Pd H₂ System
Palladium widely used in hydrogen technology

- Catalytic dissociation of molecular hydrogen to atomic hydrogen on Pd surface
- Atomic hydrogen absorbed into Pd lattice structure
- Presence of hydrogen strains lattice altering the conductivity and refractive index
- Hydrogen uptake continues until equilibrium pressure is achieved
- System strongly dependent on temperature

3. Technique and sample preparation
RF sputter coating provides repeatedly homogeneous surface

- Permittivity measured using ellipsometry
  - System returns two degrees of freedom from three unknowns: complex refractive index (n, k) and thickness
  - Complex index demands that film thickness is measured independently
- Sample thickness measured using white light interferometry (WLI)

4. Ellipsometry results
Ellipsometry can estimate film thickness based on constant permittivity

- No divergence between estimated film thickness and WLI measurements below 40nm
- No real change in refractive index above ~20nm
- Above 40nm film is opaque - ellipsometry measurement independent of film thickness

5. Surface Plasmon Resonance (SPR)
SPR technique used to measure change in complex permittivity due to hydrogen absorption

- Thin film ~30nm in Krechman arrangement
- 1525nm HeNe couples to SPR at specific angles resulting in a loss band in angular reflection
- System calibrated using ellipsometry data
- Reflects intensity fitted to theory as a function of angle
- Gas concentrations varied within gas cell using mass flow controllers

6. Hydrogen Results
Clear change between loaded and unloaded states

- Resonance of Pd is extremely broad, almost all angles above the critical angle
- Changing permittivity shows general trend
- Possibility of interesting low concentration effect on lattice structure
- Complex index demands that film thickness is measured independently

7. Conclusions

- RF sputter coated Pd thin film index independent of film thickness above ~20nm
- Reflective index highly dependent on exact deposition technique
- Requires samples to be characterised, published data cannot be relied upon
- Change in permittivity due to hydrogen is non linear
- Imaginary and real components of permittivity are not equal
- Possibility of interesting low concentration effects
- Further work is required
  - Greater body of data for low concentrations
  - Effect of temperature on index (with and without hydrogen)
  - Effect of surface contamination, particularly polymers and sulphur
  - Higher concentrations include Pd phase changes

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