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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 measurements: 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 Kretschmann arrangement
- 1525nm HeNe couples to SPR at specific angles resulting in a loss band in angular reflection
- System calibrated using ellipsometry data
- Reflected intensity fitted to theory as a function of angle

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
- Refractive index highly dependant on exact deposition parameters
  - Non equal changes in real and imaginary components
  - 1% H₂ – 10% decrease in real
  - 1% H₂ – 5% decrease in imaginary

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