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

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Richard M. Carter[a], Peter Morrall[b], Robert R.J. Maier[a], Ben J. S. Jones[b], Scott McCulloch[b], James S. Barton[a]
a Heriot-Watt University, School of EPS, Department of Physics, Edinburgh, UK ; b AWE plc., Aldermaston, UK

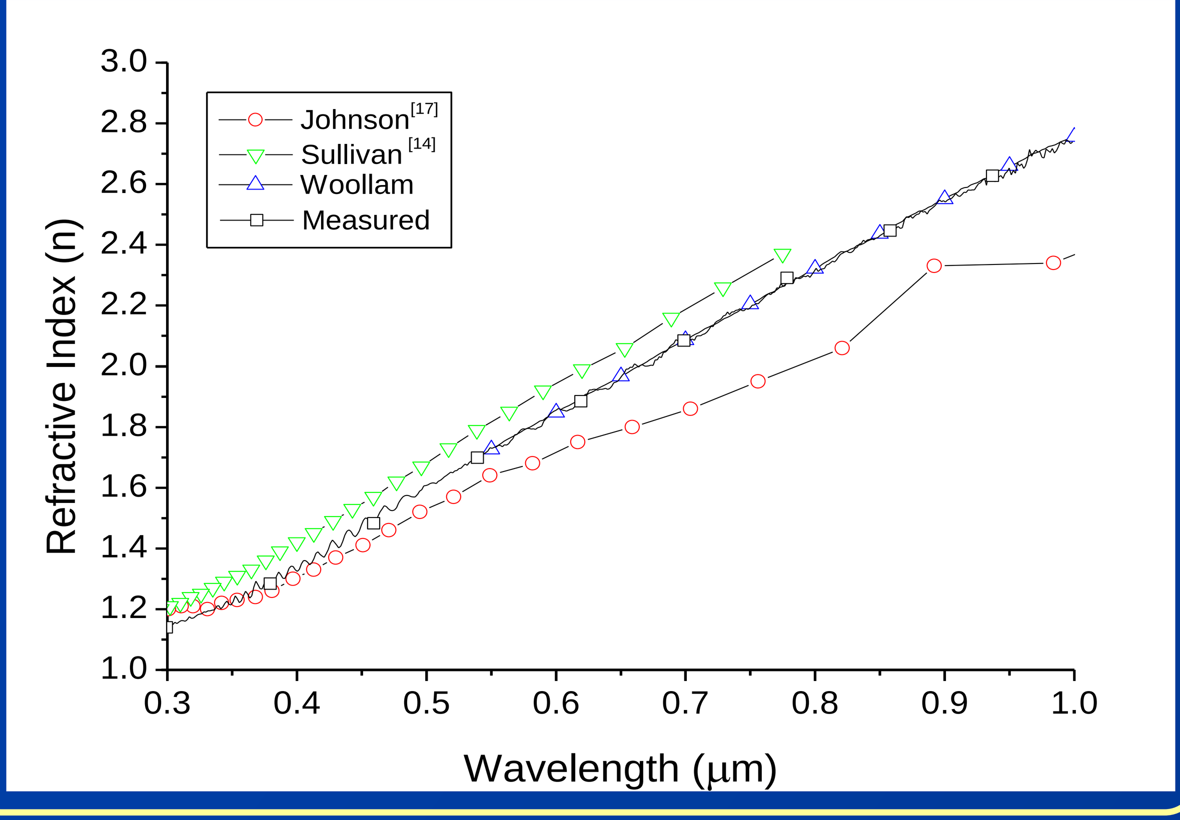
1. Motivation

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



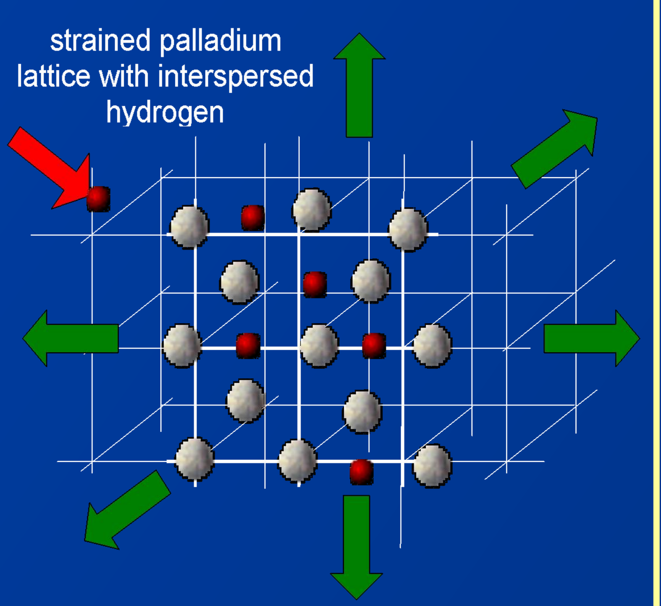
2. Pd H_2 System

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



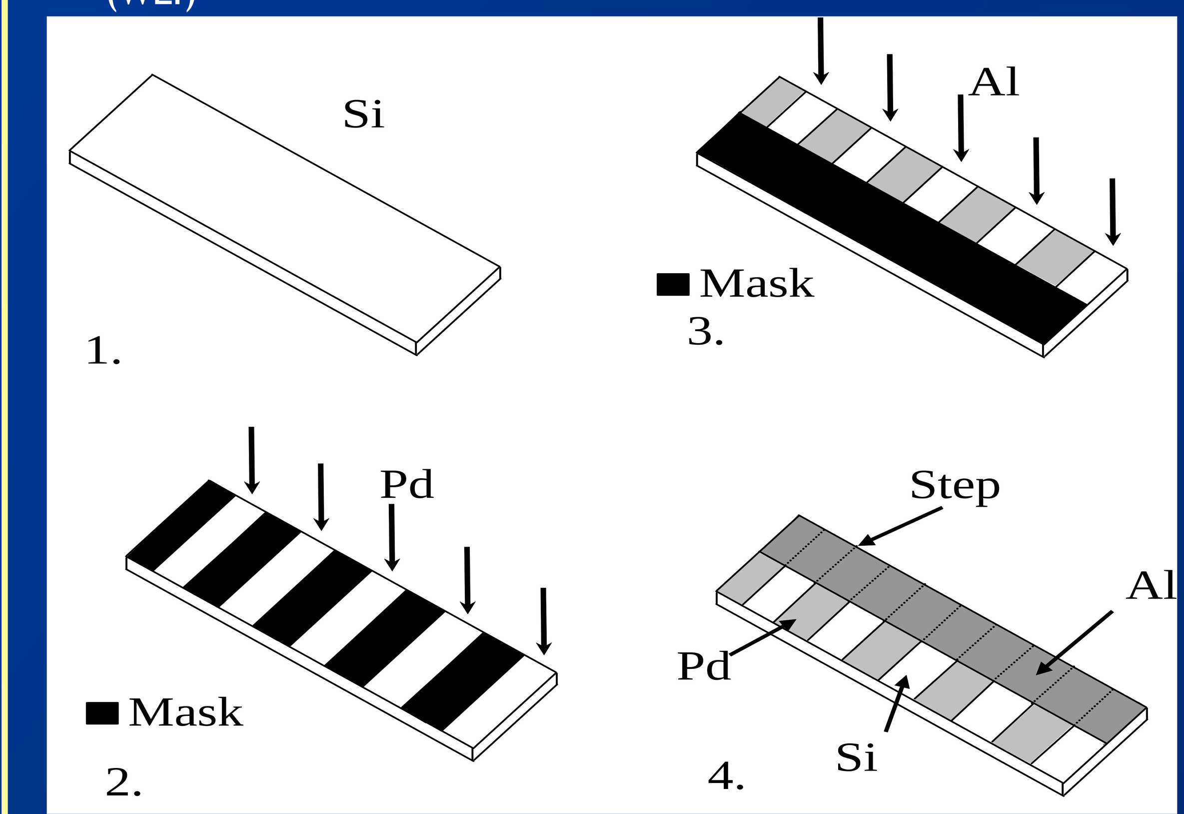
- Hydrogen uptake continues until equilibrium pressure is achieved
- System strongly dependant on temperature

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+ ik) and thickness
  - Complex index demands that film thickness is measured independently

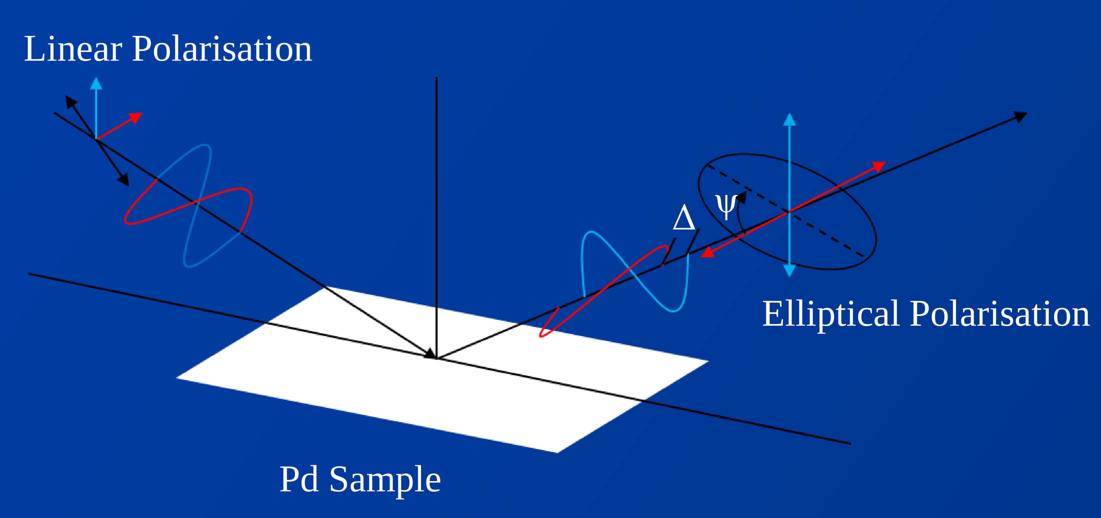
- Sample thickness measured using white light interfermometry (WLI)



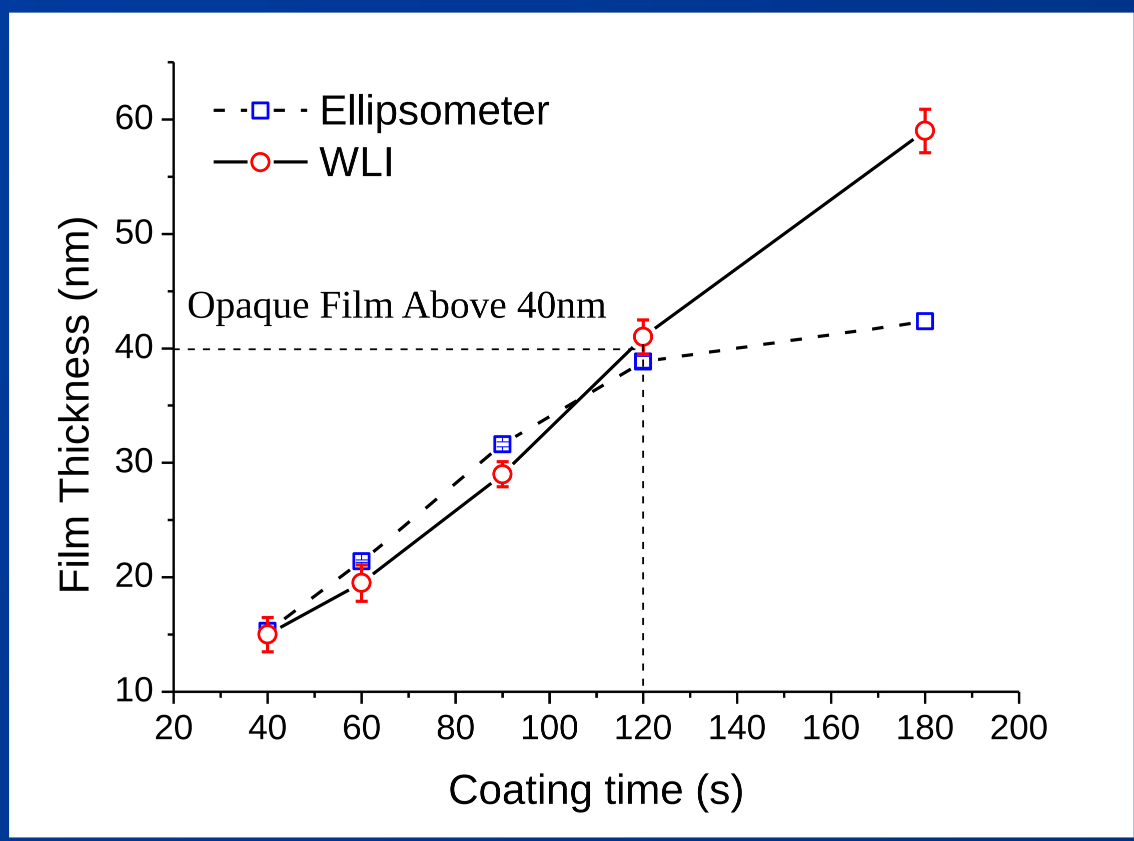
- Two systems require different sample structures
  - Homogeneous flat Pd surface for ellipsometry
  - Step function in Al for WLI

4. Ellipsometry results

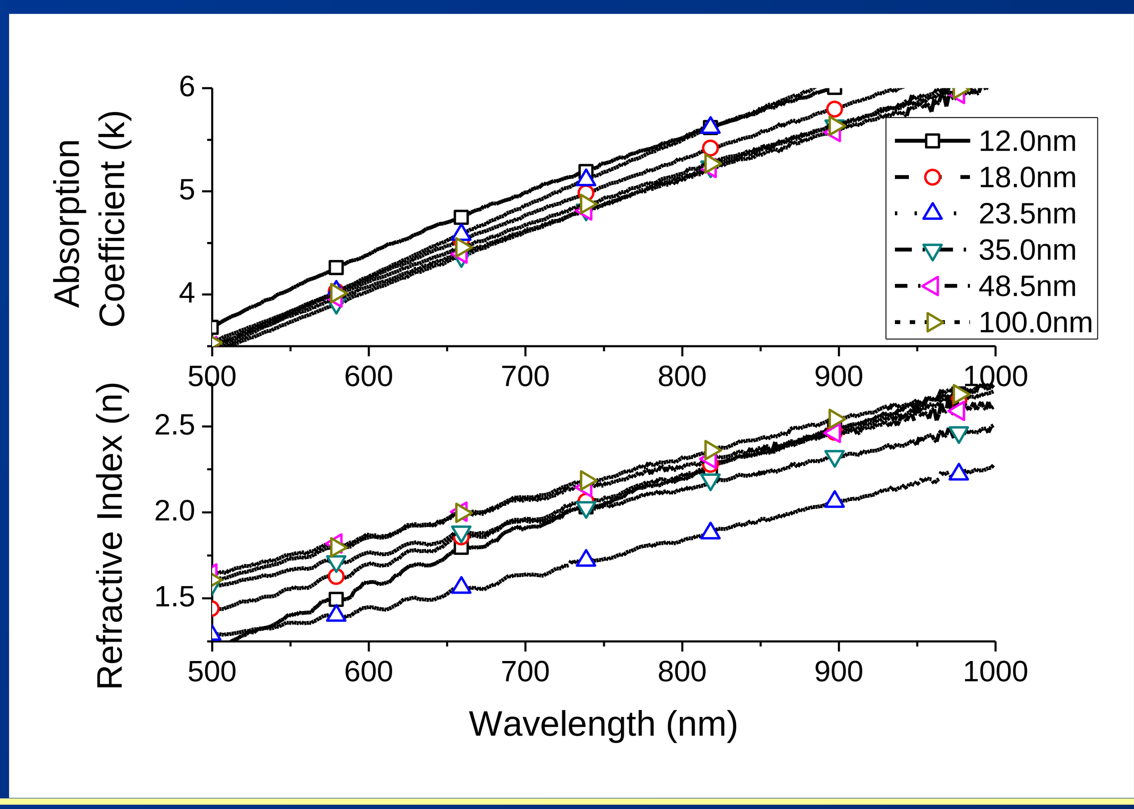
Ellipsometry can estimate film thickness based on constant permittivity



- 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



- Complex index directly calculated based on WLI film thickness measurement
  - Errors in WLI produce error in refractive index
  - Thicker films, ~100nm, are independent of film thickness giving accurate measurement



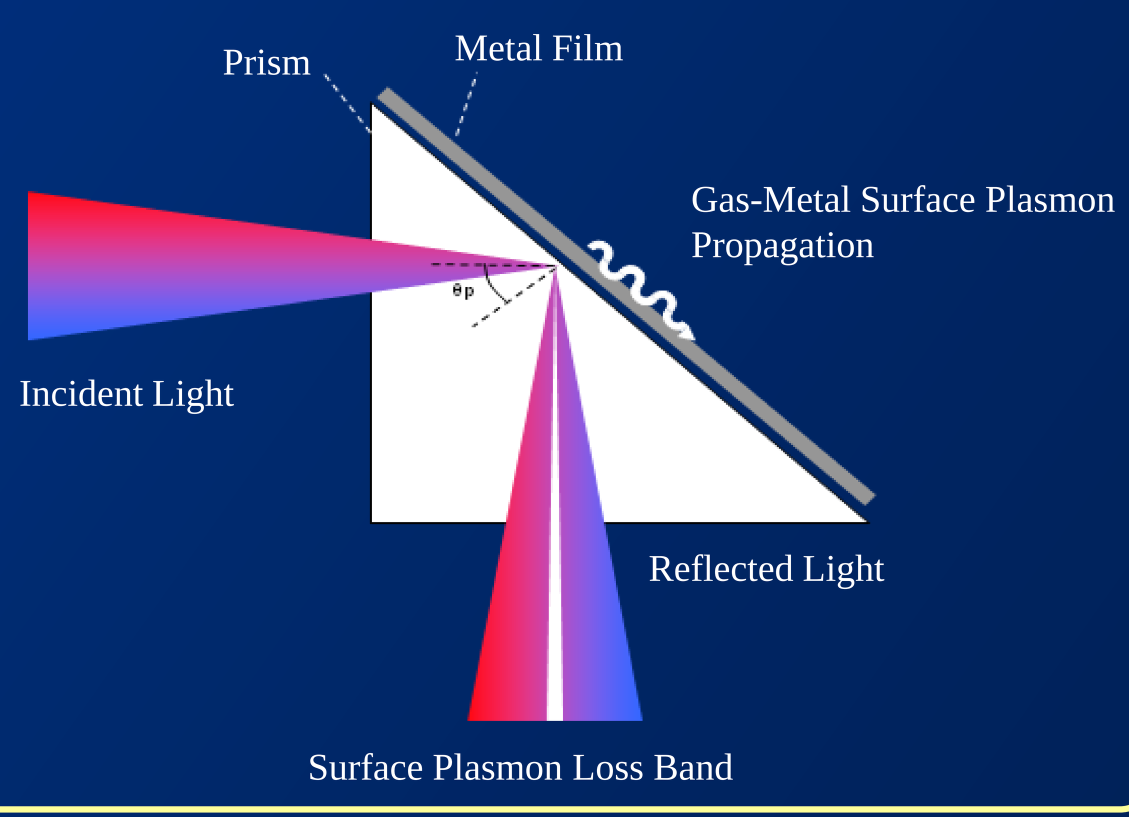
5. Surface Plasmon Resonance (SPR)

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

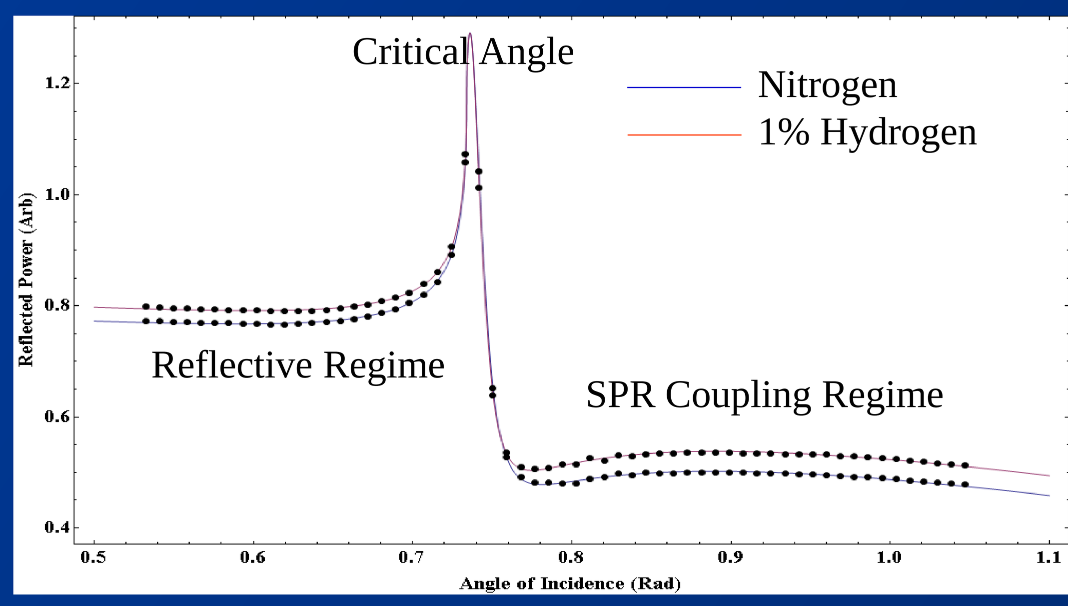
- System calibrated using ellipsometry data
- Reflected intensity fitted to theory as a function of angle

- Gas concentrations varied within gas cell using mass flow controllers



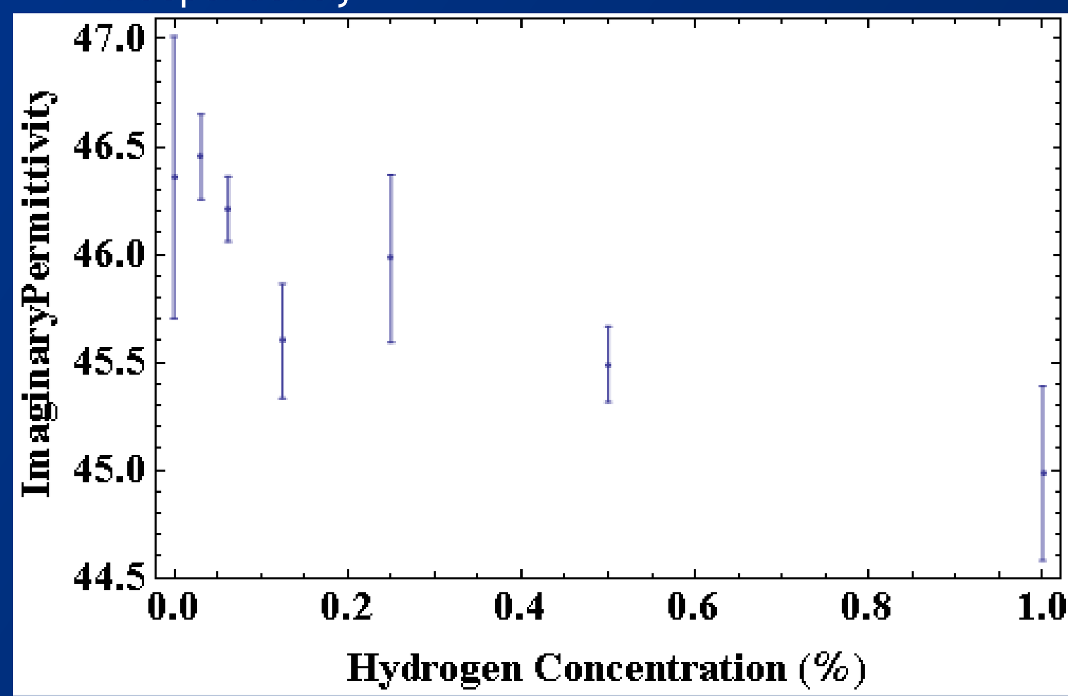
6. Hydrogen Results

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



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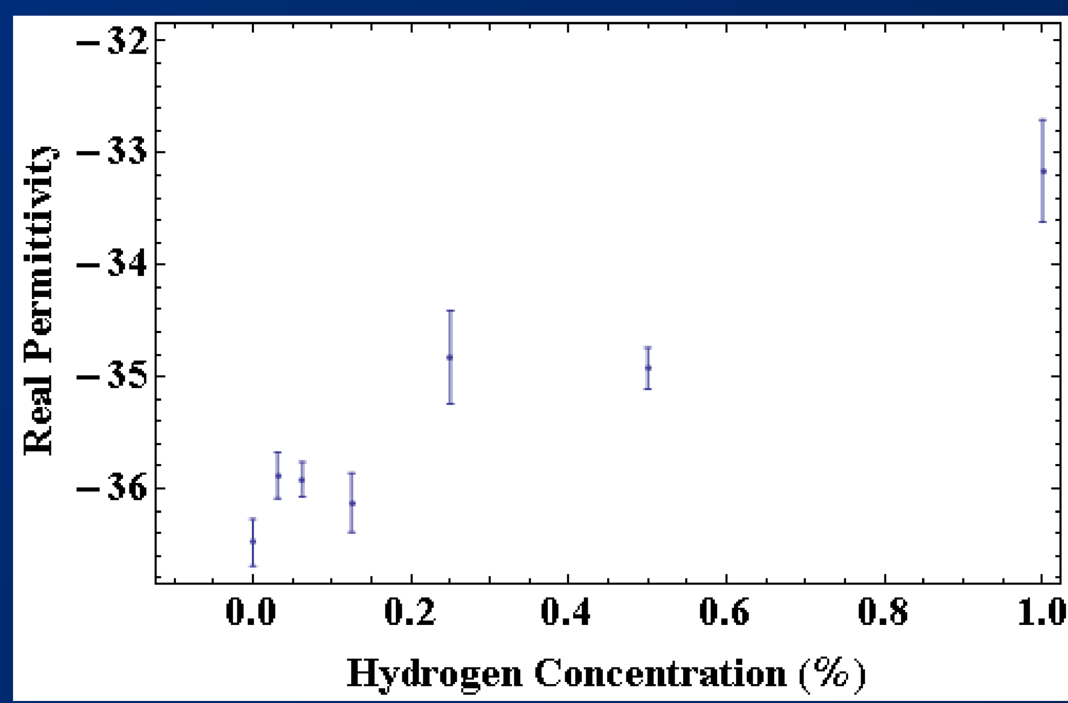

- Non equal changes in real and imaginary components
  - 1% H_2 ~ 10% decrease in real
  - 1% H_2 ~ 5% decrease in imaginary

7. Conclusions

- RF sputter coated Pd thin film index independent of film thickness above ~20nm

- Refractive index highly dependant 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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Contact: Richard Carter, Heriot-Watt University, School of Engineering and Physical Sciences, Department of Physics, Fibre Optics Group in Applied Optics and Photonics, Edinburgh EH14 4AS, UK; e-mail: rmc5@hw.ac.uk ; http://www.aop.hw.ac.uk/