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

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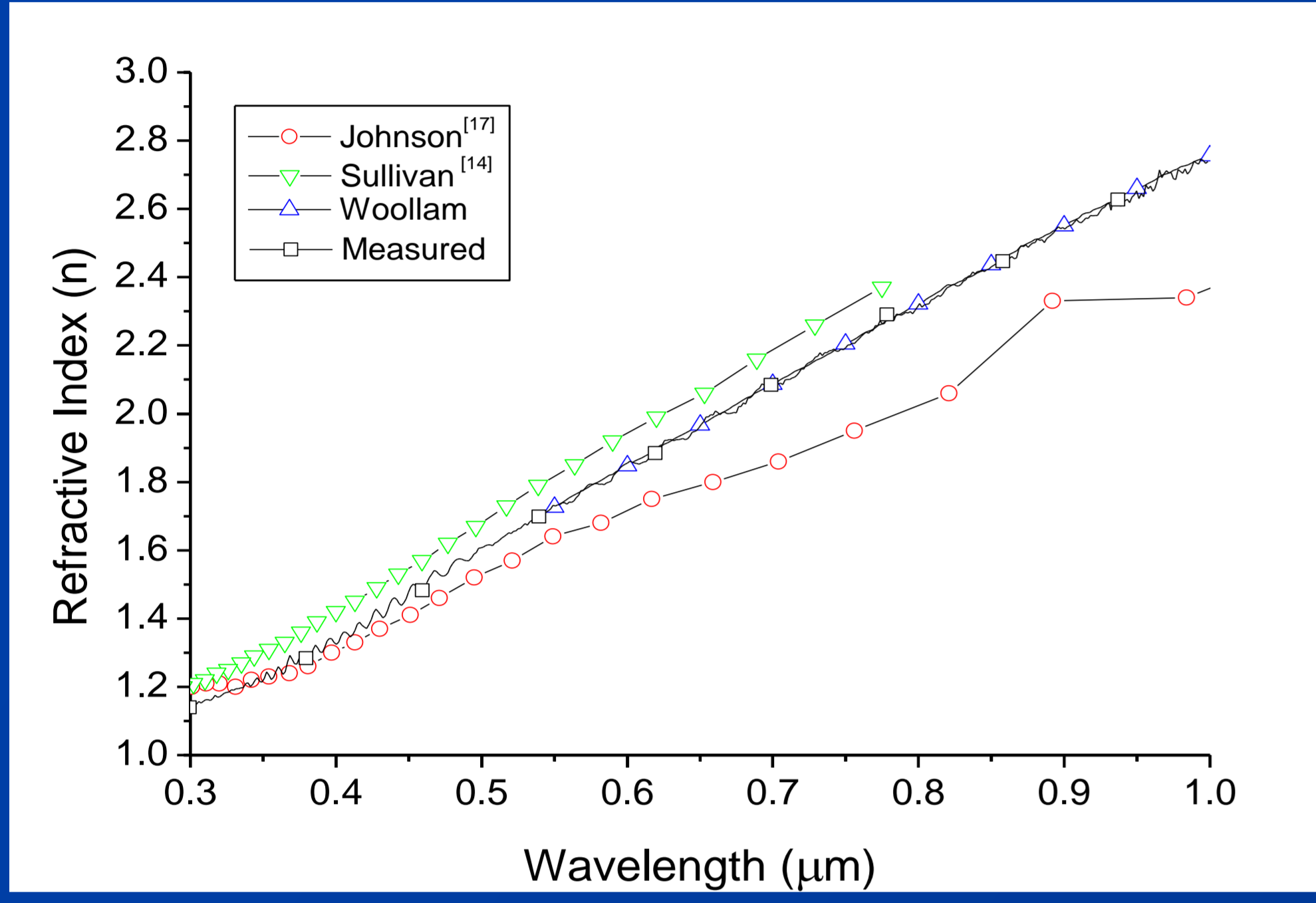
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1. Motivation

Reliable hydrogen detection technologies required for safety applications

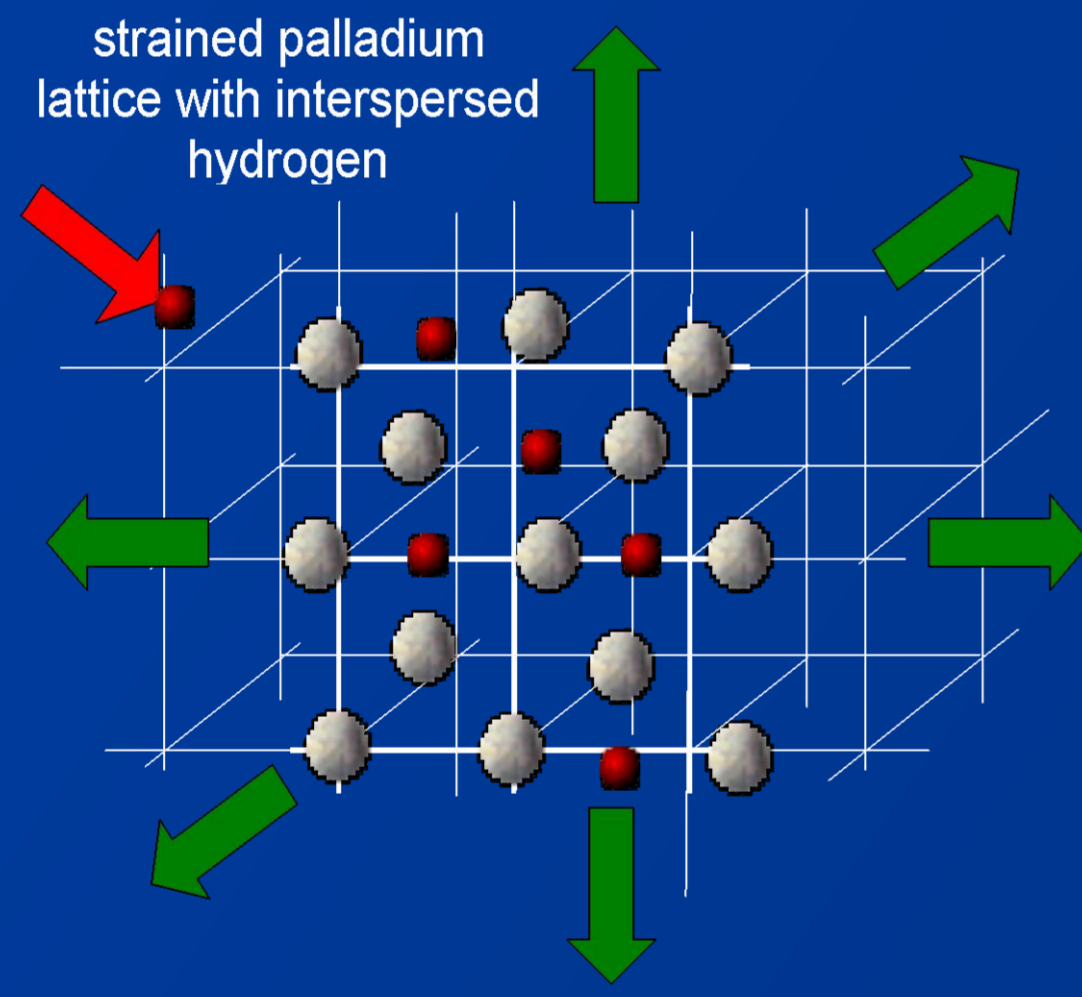
- Hydrogen suggested as future fuel source
- Hydrogen explosive at 4 - 97% concentration in air
- Most systems based on the absorption of hydrogen in palladium (Pd)
- Optical system preferable for safety reasons
 - No heating
 - Zero electrical charge
- Optically well characterised homogeneous thin Pd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



2. Pd H₂ System

Palladium widely used in hydrogen technology

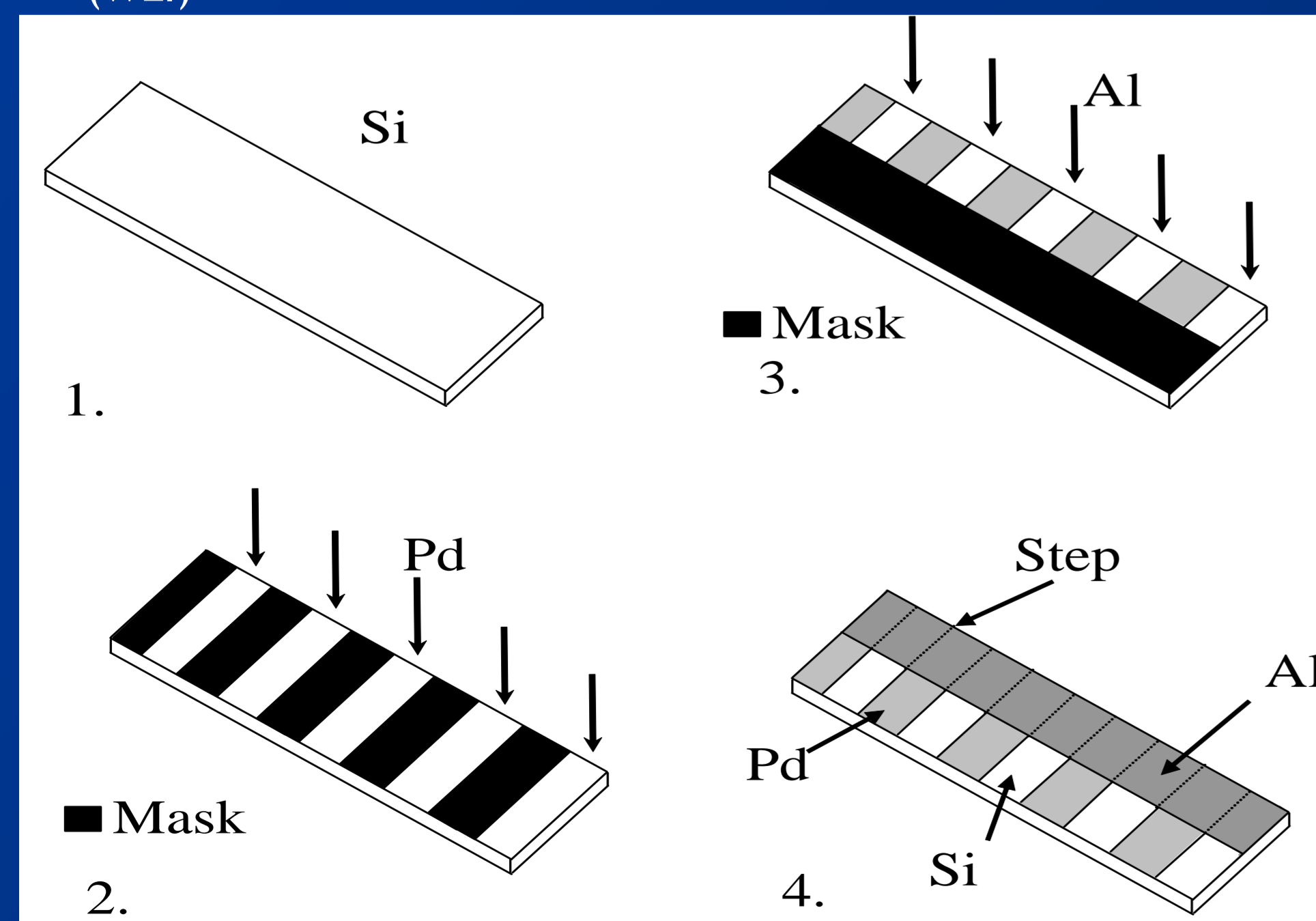
- Catalytic dissociation of molecular hydrogen to atomic hydrogen on Pd surface
- Atomic hydrogen absorbed into Pd lattice structure
- Presence of hydrogen strains lattice altering the conductivity and refractive index
- Hydrogen uptake continues until equilibrium pressure is achieved
- System strongly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



3. Technique and sample preparation

RF sputter coating provides repeatedly homogeneous surface

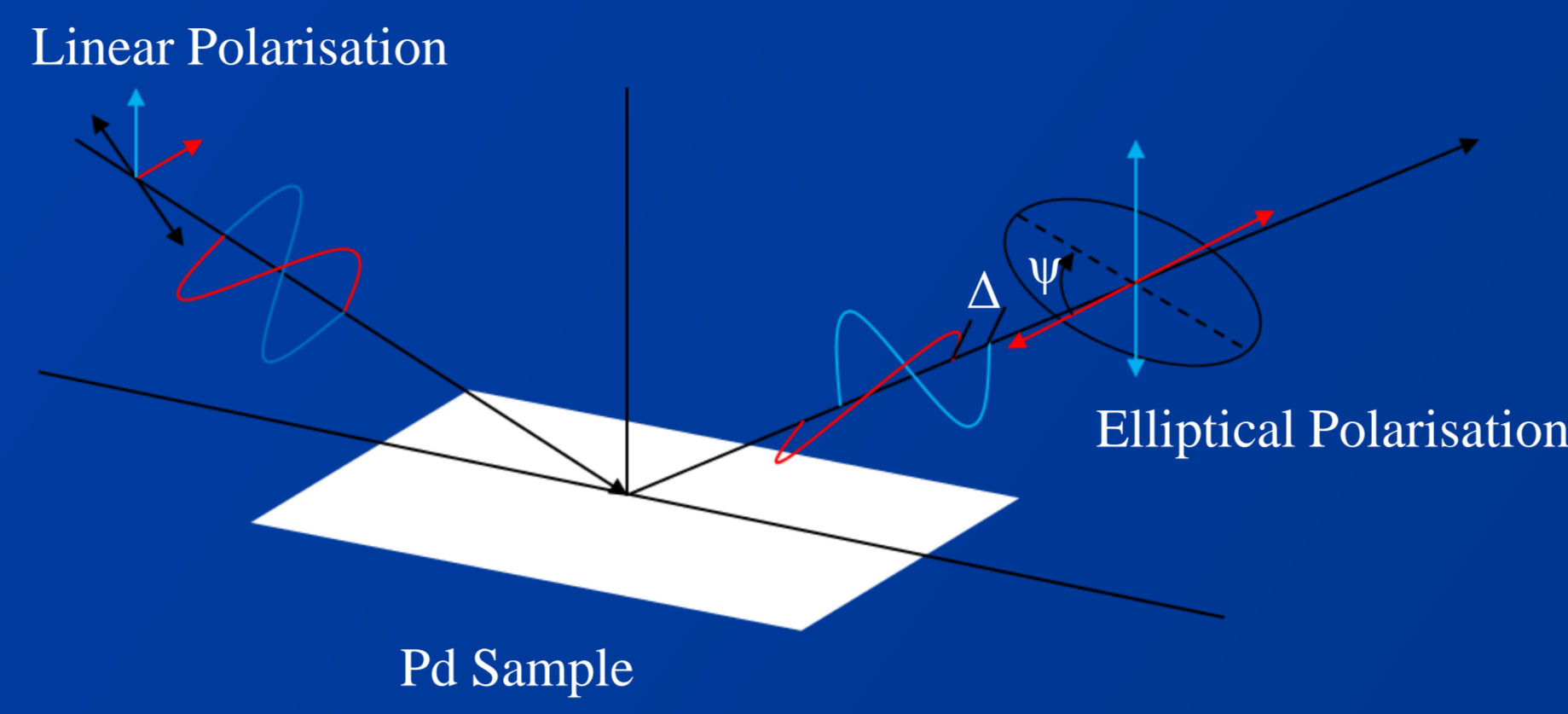
- Permittivity measured using ellipsometry
 - System returns two degrees of freedom from three unknowns: complex refractive index ($n+ik$) and thickness
 - Complex index demands that film thickness is measured independently
- Sample thickness measured using white light interferometry (WLI)



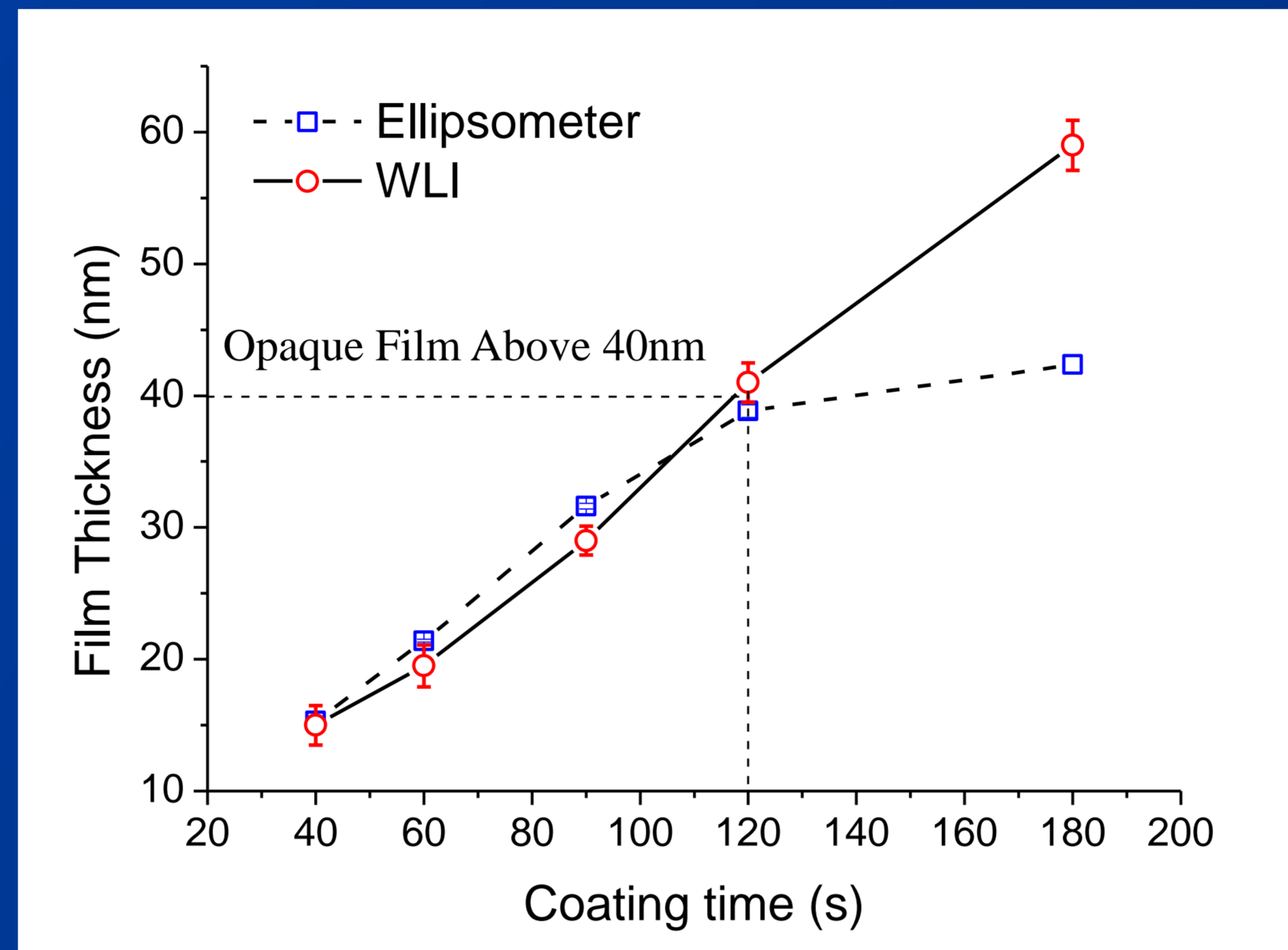
- Two systems require different sample structures
 - Homogeneous flat Pd surface for ellipsometry
 - Step function in Al for WLI

4. Ellipsometry results

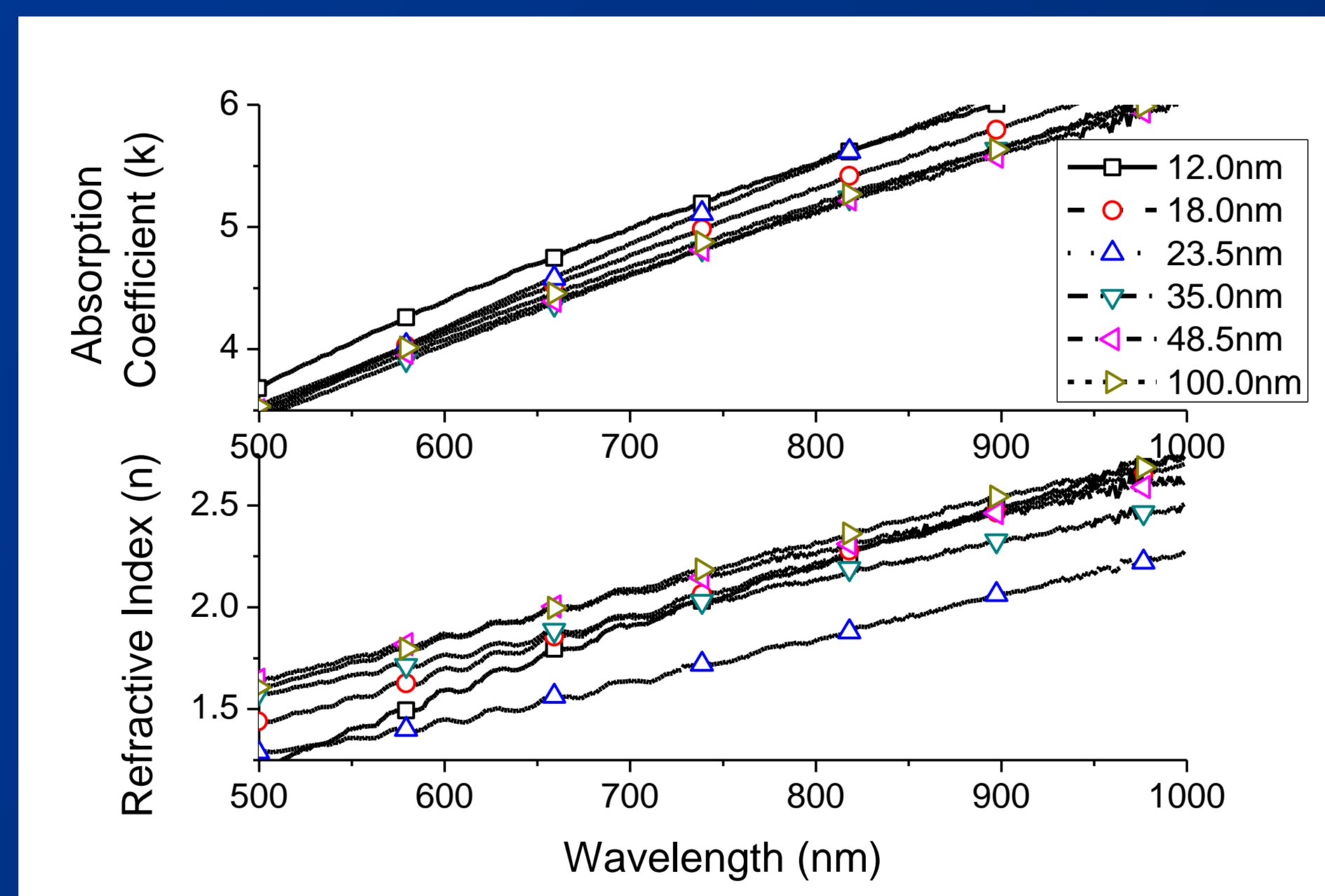
Ellipsometry can estimate film thickness based on constant permittivity



- 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



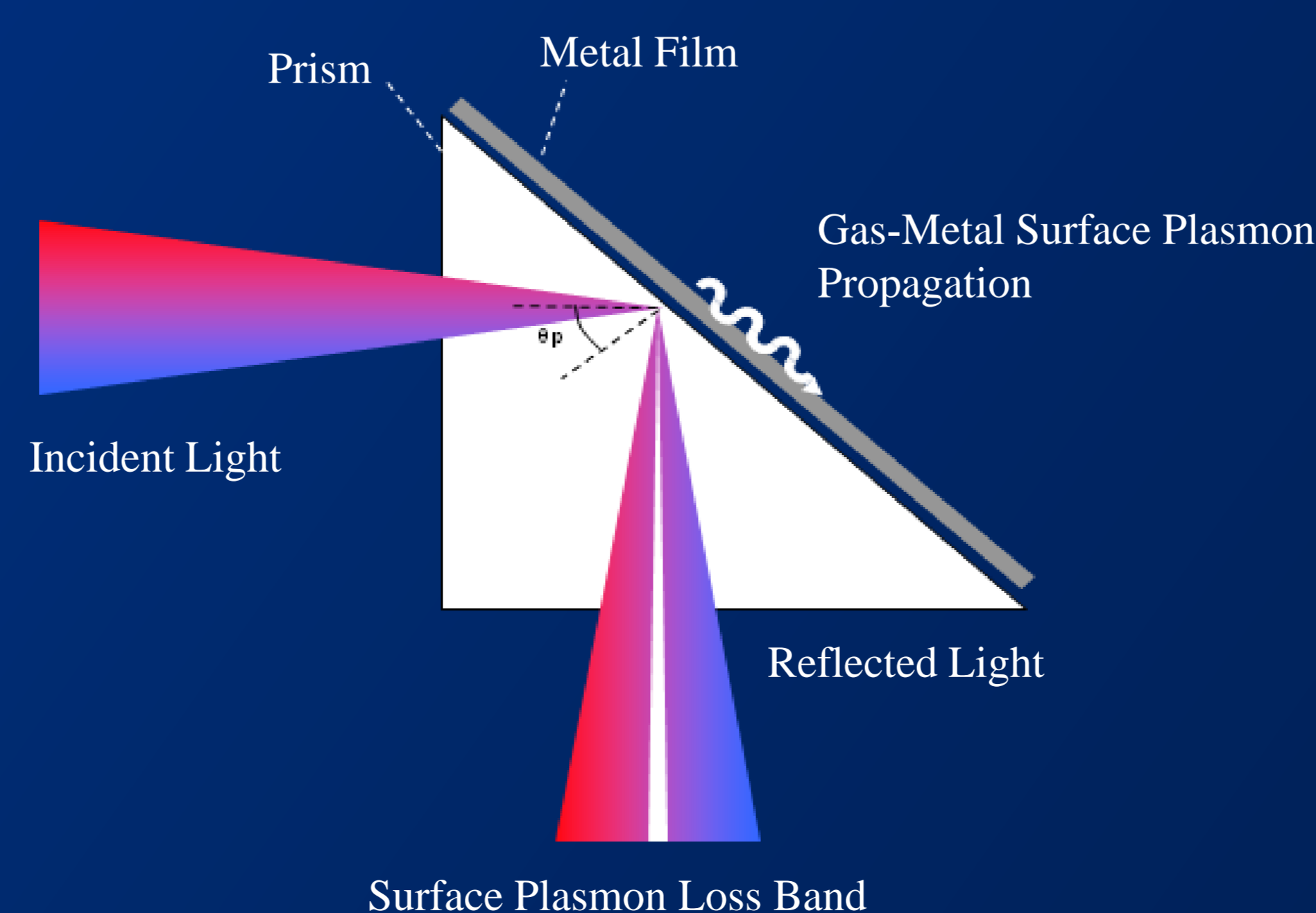
- Complex index directly calculated based on WLI film thickness measurement
 - Errors in WLI produce error in refractive index
 - Thicker films, ~100nm, are independent of film thickness giving accurate measurement



5. Surface Plasmon Resonance (SPR)

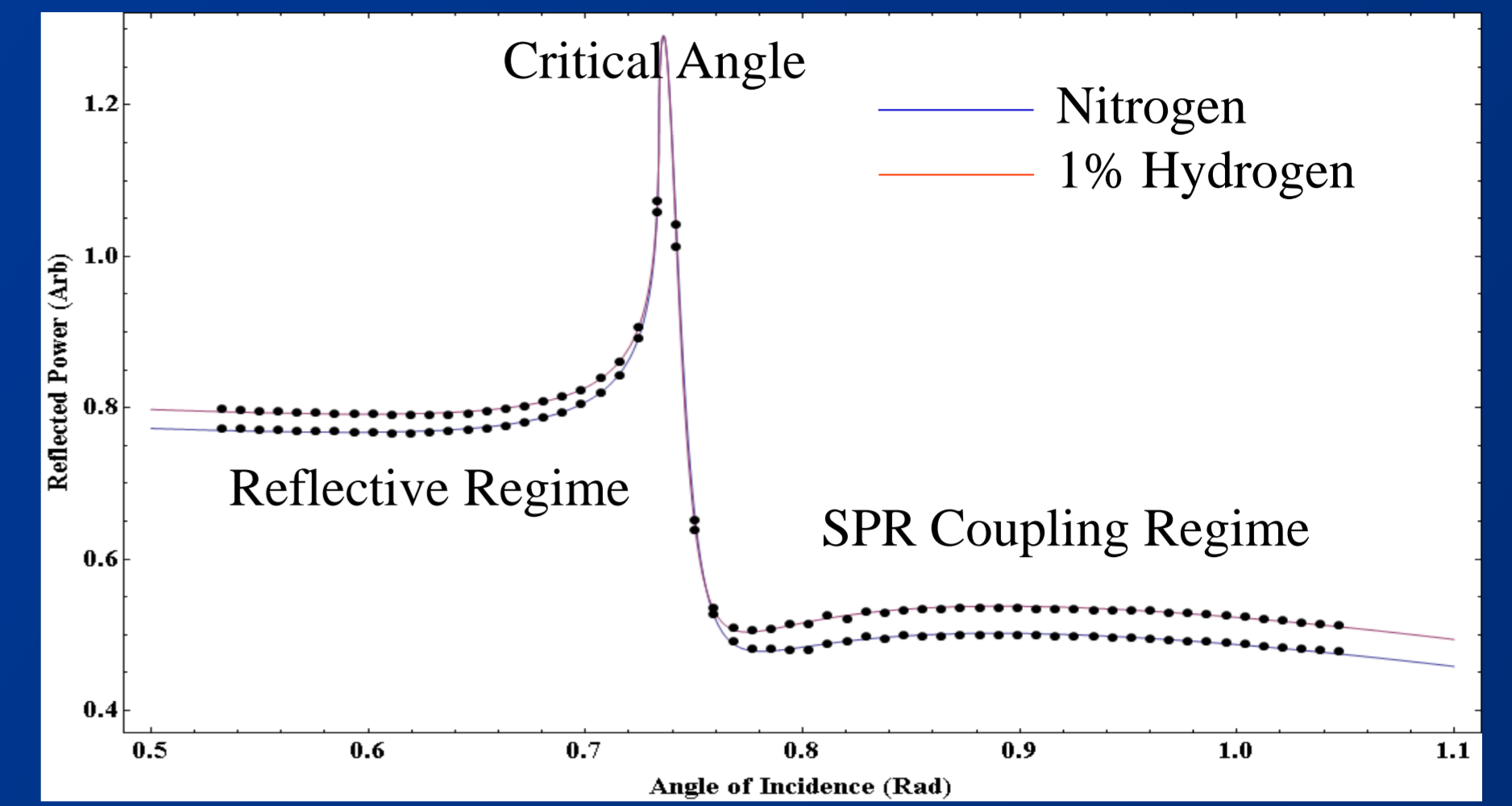
SPR technique used to measure change in complex permittivity due to hydrogen absorption

- 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

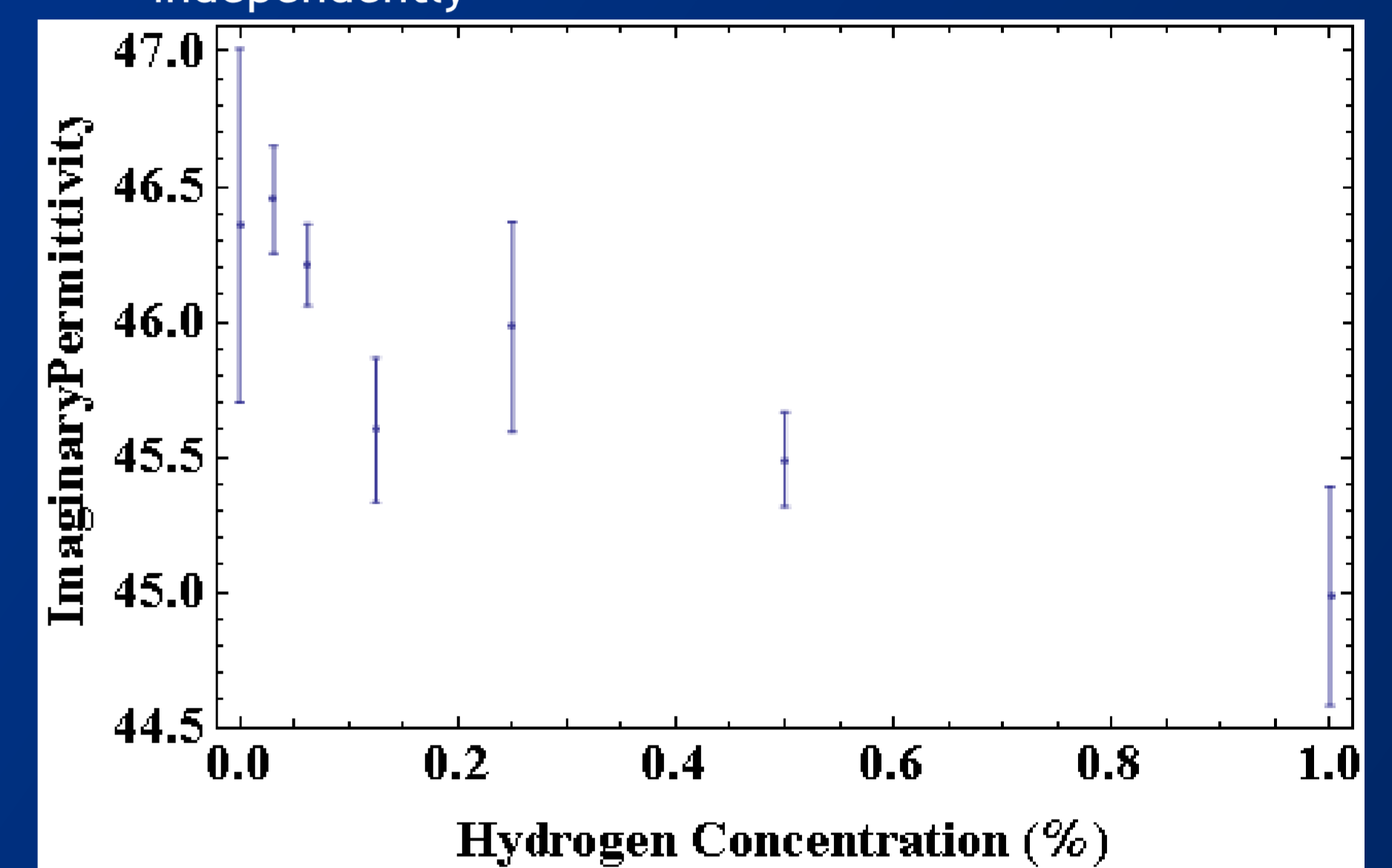


6. Hydrogen Results

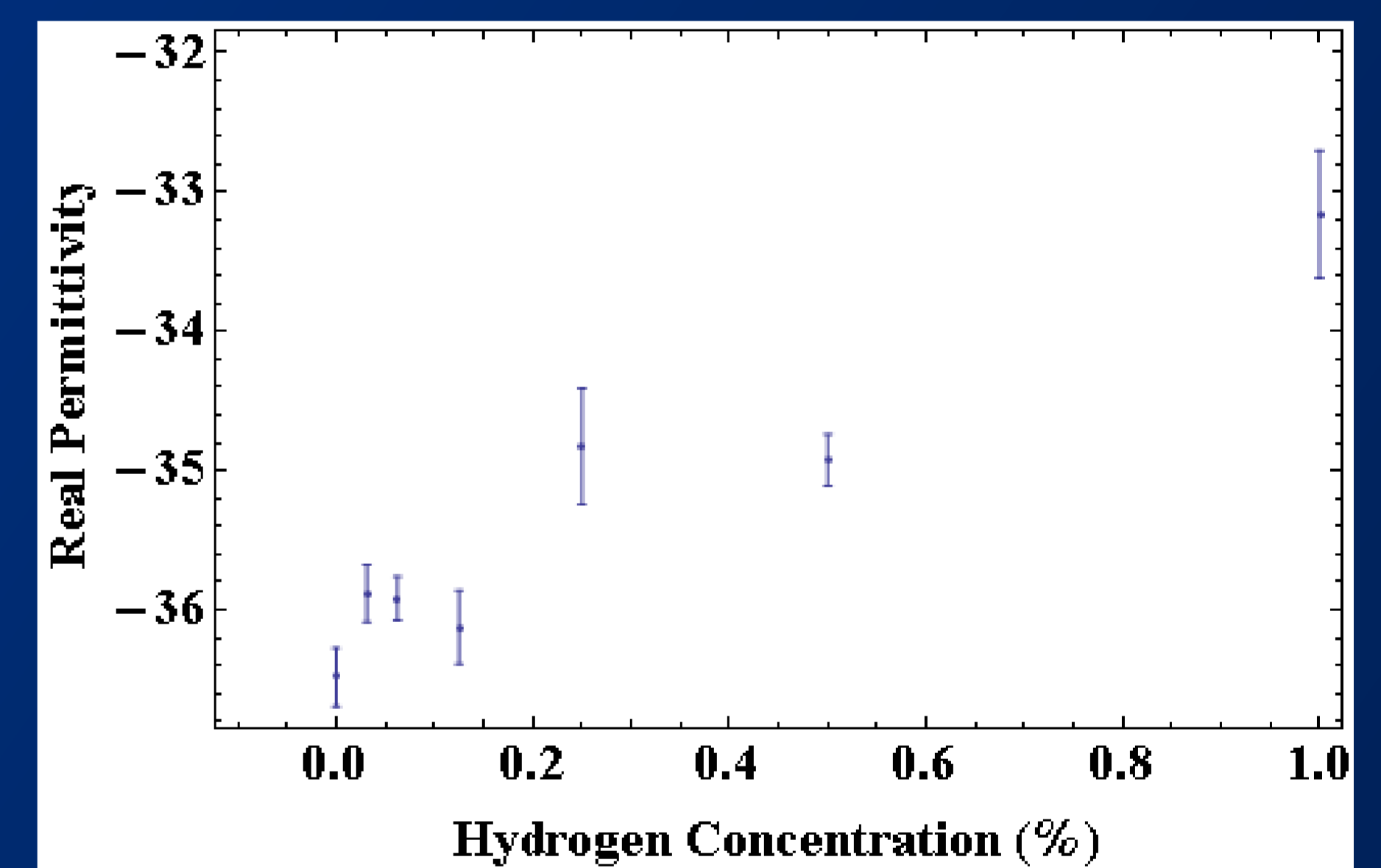
Clear change between loaded and unloaded states



- 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



- Both real and imaginary components decrease in magnitude
- Both real and imaginary components decrease in magnitude
 - Non linear effect



- Non equal changes in real and imaginary components
 - 1% H₂ ~ 10% decrease in real
 - 1% H₂ ~ 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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