

New Techniques for Determining Electronic Properties of Nitrogen Doped Carbon Nanospheres

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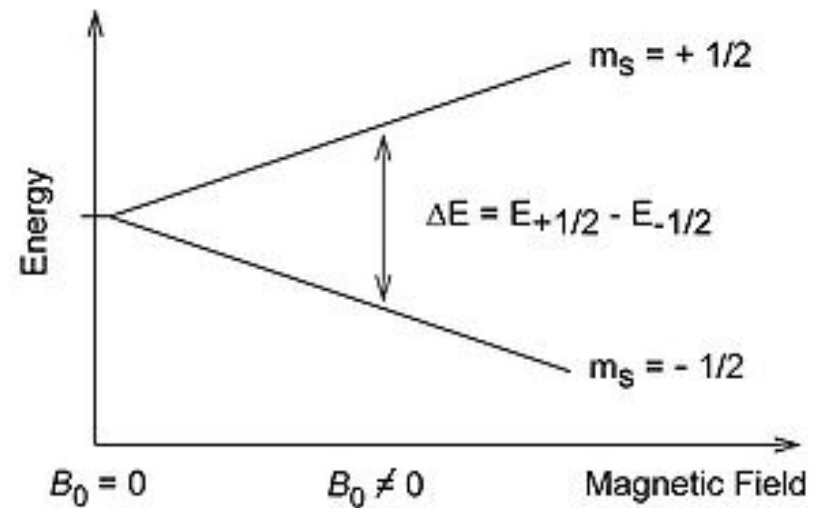
Introduction Part 1

- Carbon nanomaterials are of great scientific and technological interest at present due to their wide applicability.
- Carbon nanospheres (CNS) were produced using a horizontal CVD reactor.
- CNS of known dopant level were characterized using Electron Paramagnetic Resonance (EPR)
- Nitrogen content of unknown samples was determined using an EPR spectrometer

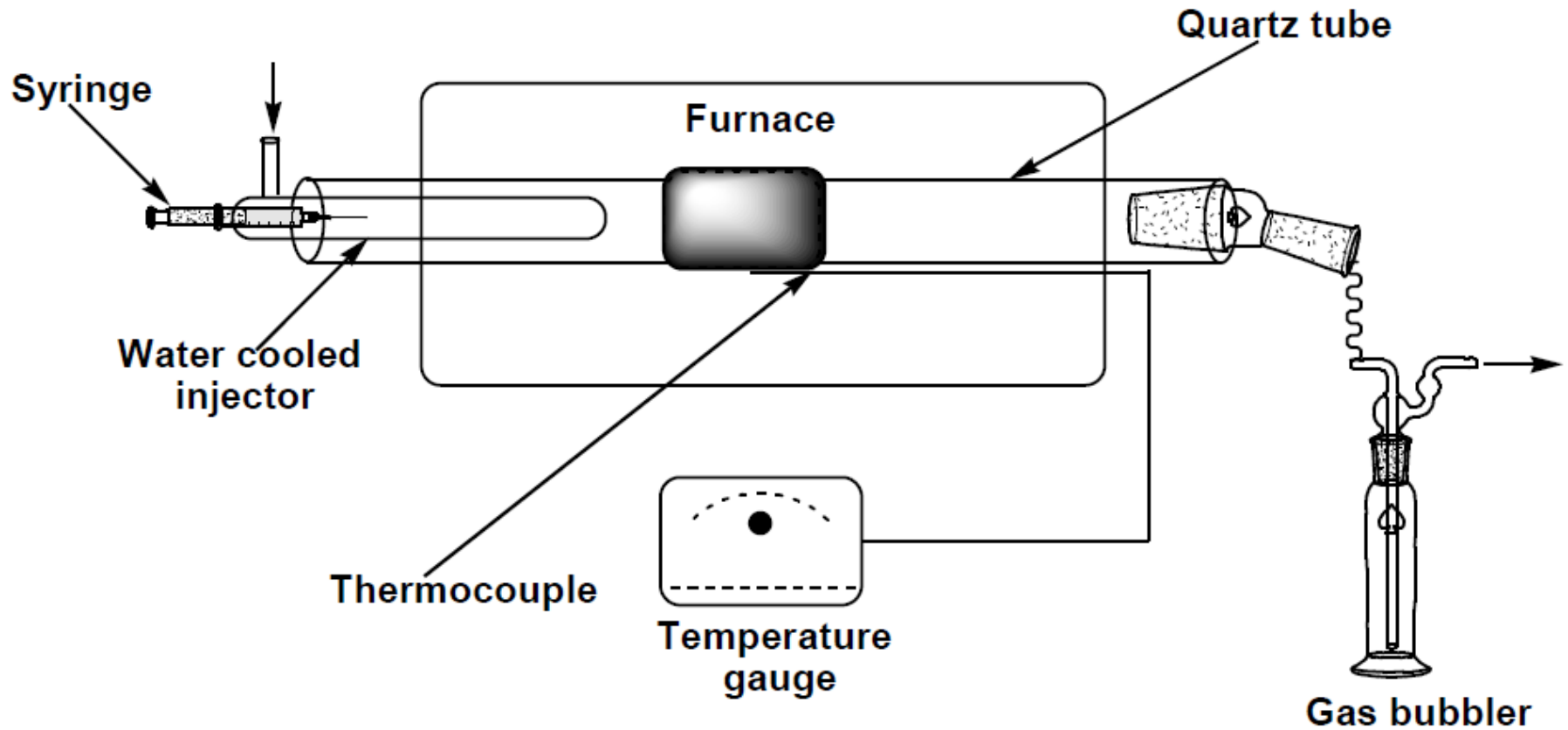


Electron Magnetic Resonance

- EMR (also known as ESR and EPR) experiment was performed using the Bruker Spectrometer shown below. The experiment was performed in CW mode.



Horizontal CVD reactor



XPS Determination of N dopant

SDA = 0% Nitrogen

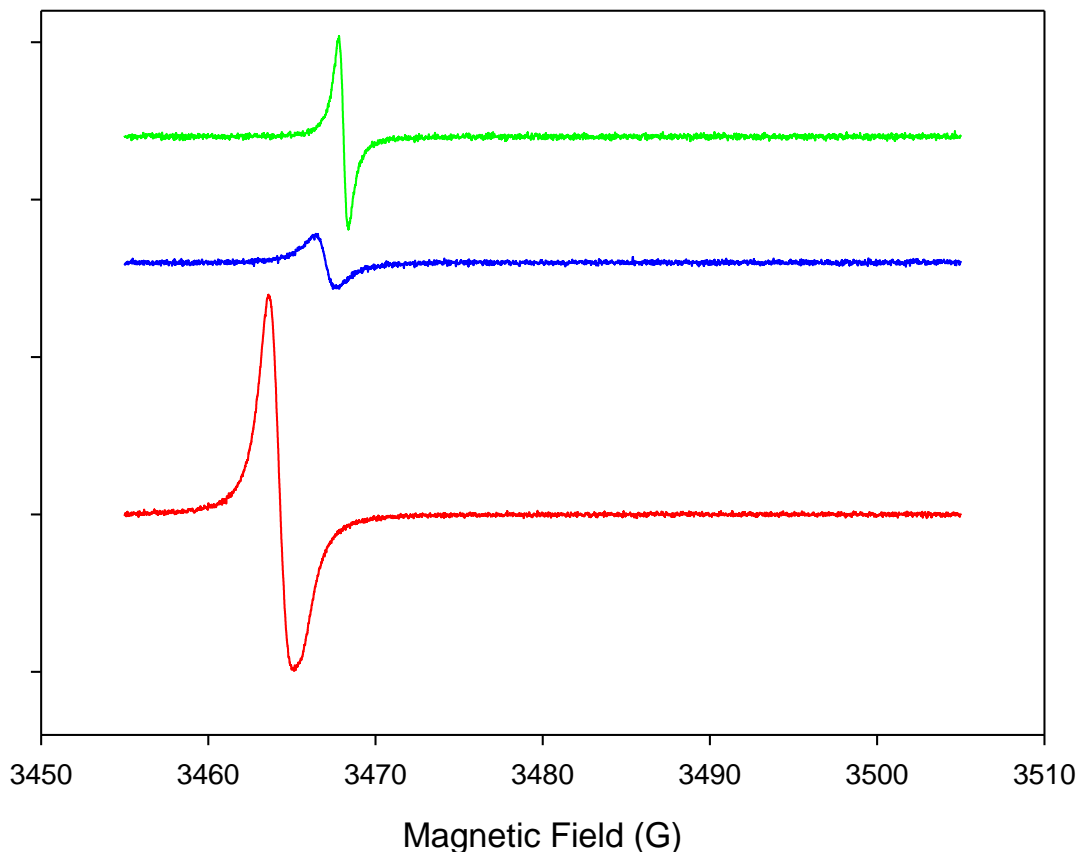
SDB = 0.4% Nitrogen

SDC = 2.5% Nitrogen

SDD = 5% Nitrogen



EPR Data of Selected Samples

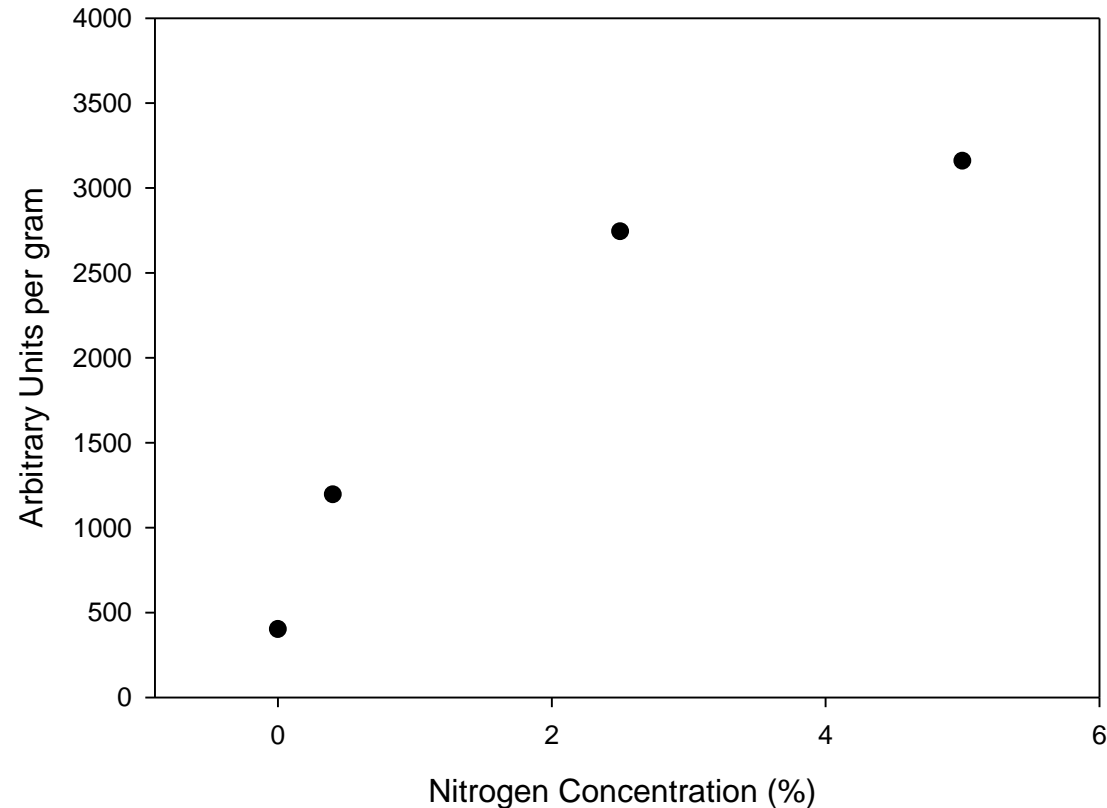


EPR spectra of three N-doped CNS obtained. Signal strength is based on many parameters, both of the spectrometer and the samples.

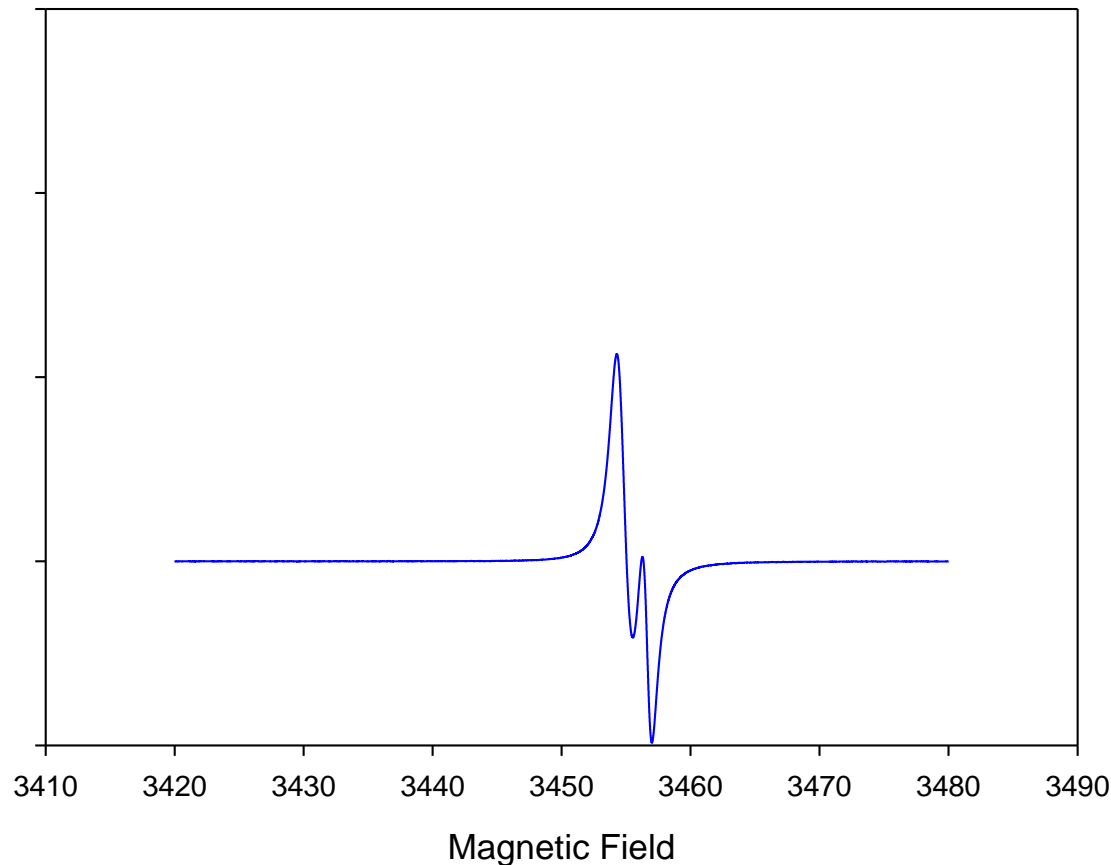
Samples are carefully weighed to calculate the number of paramagnetic sites present per gram of sample.

EPR Spectrometer Calibration

EPR Spectrometer Calibration Curve



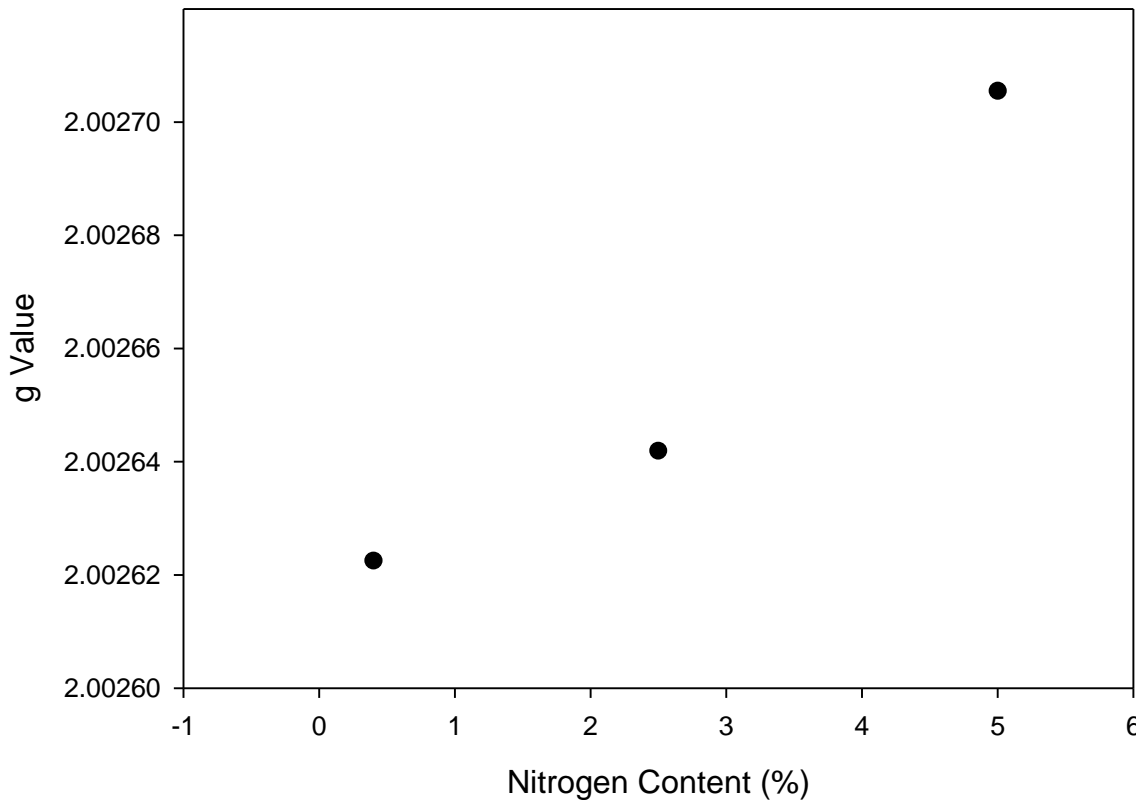
Composite EPR Spectrum of SDD and DPPH



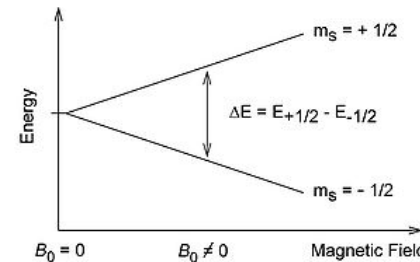
A Composite EPR spectrum of N-doped CNS sample (SDD) and the EPR reference standard (DPPH) that we used to determine the g-factor. Deconvolution of the two spectra allows one to determine the g value of the original signal.

EPR Spectrometer Calibration

Electron g Value vs Sample Nitrogen Concentration



- The g Value increases with increased nitrogen concentration.



$$h\nu = g\mu B$$

Unknown Sample Characterisation

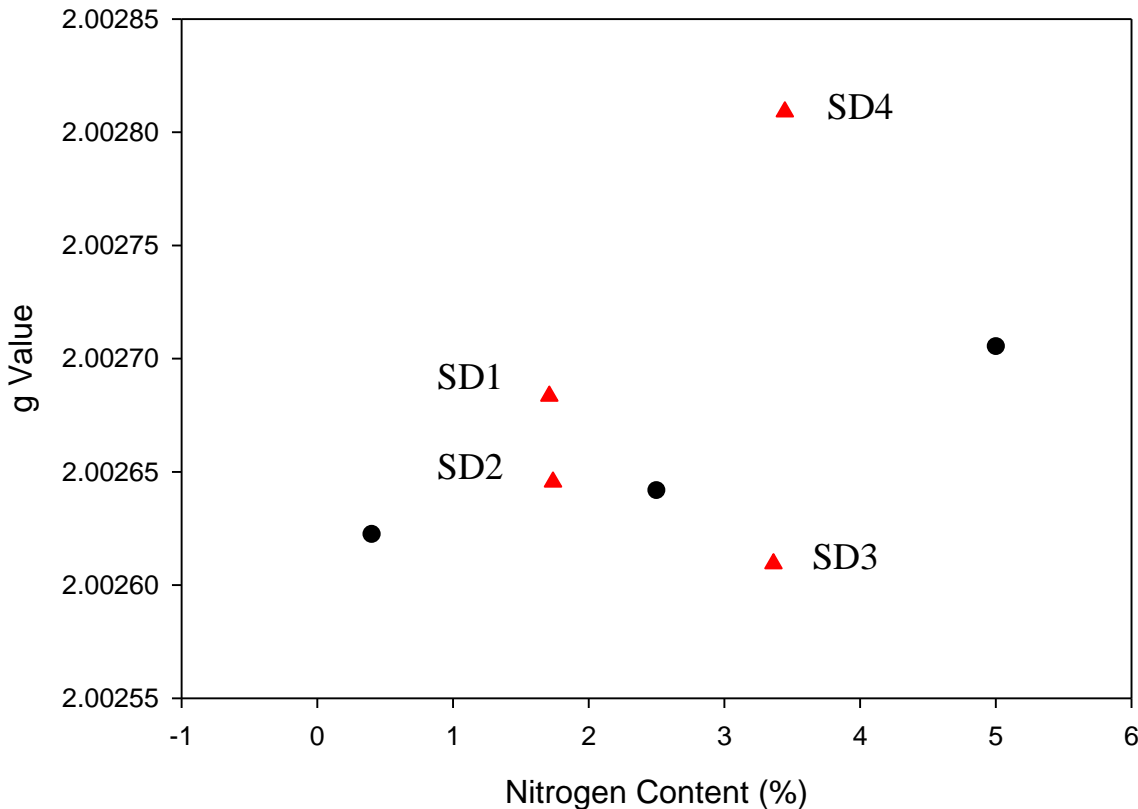
- SD1 = C₂H₂ at 900°C for 2 hours.
- SD2 = C₂H₂ at 900°C for 1.5 hours.

- SD3 = Collected from quartz tube
- SD4 = Collected from quartz boat

- SD1 = 1.710% ± 0.503%
- SD2 = 1.737% ± 0.509%
- SD3 = 3.362% ± 1.101%
- SD4 = 3.446% ± 0.986%

Unknown Sample Characterisation

Electron g Value vs Sample Nitrogen Concentration



SD1 = 2 hours.

SD2 = 1.5 hours.

SD3 = quartz tube

SD4 = quartz boat

Introduction Part 2

- Determination of Electronic Transport Properties of CNS are critical to their deployment in industry.
- Doping contributes significantly to the transport properties of these materials.
- Resistivity of the bulk CNS samples was determined using the Van der Pauw technique.
- Sample chambers were designed and built in-house with the use of open source communities.



Open Source Technologies

- Open Source technologies were extensively deployed in the production of these results.

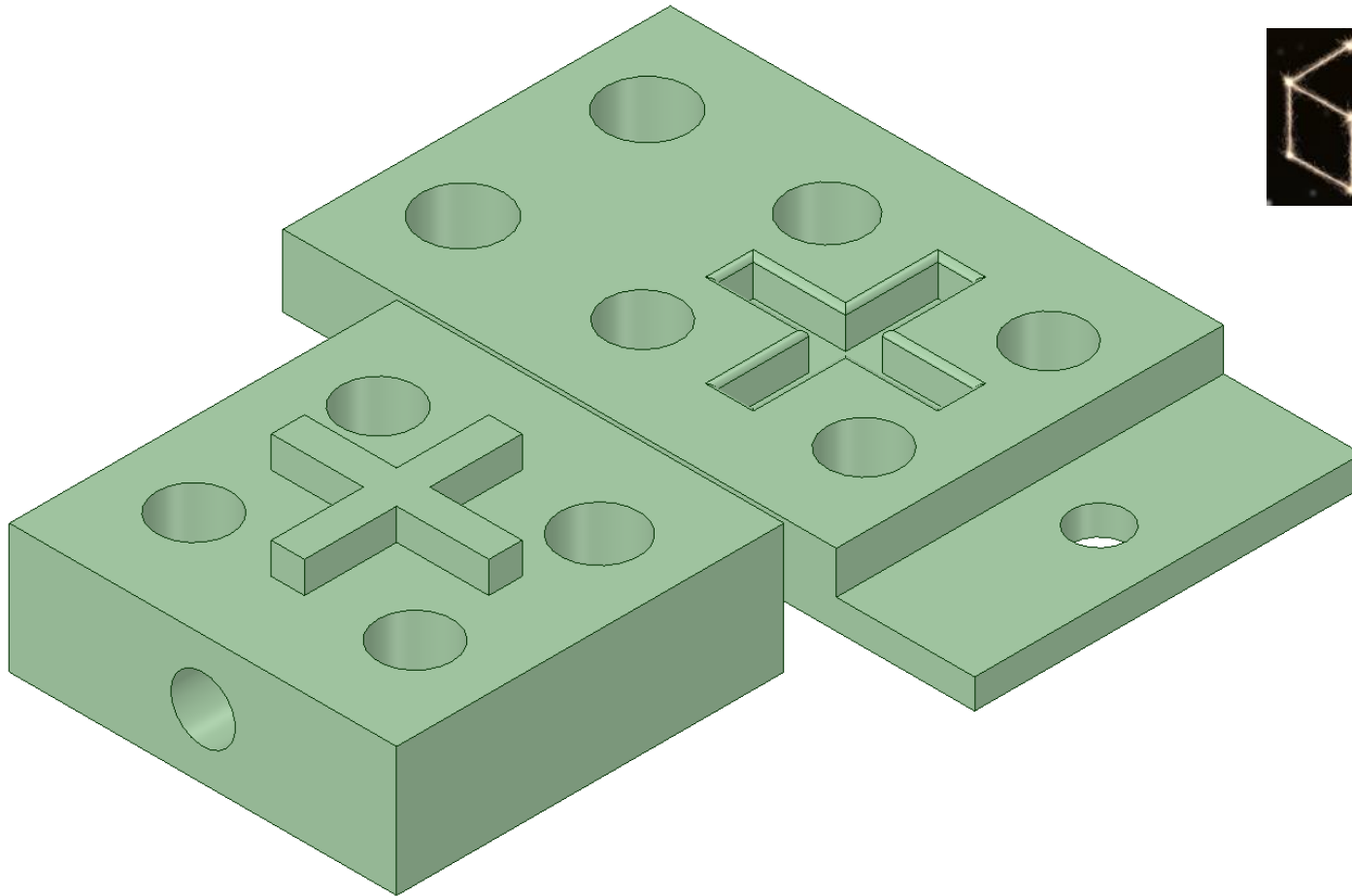


Slic3r

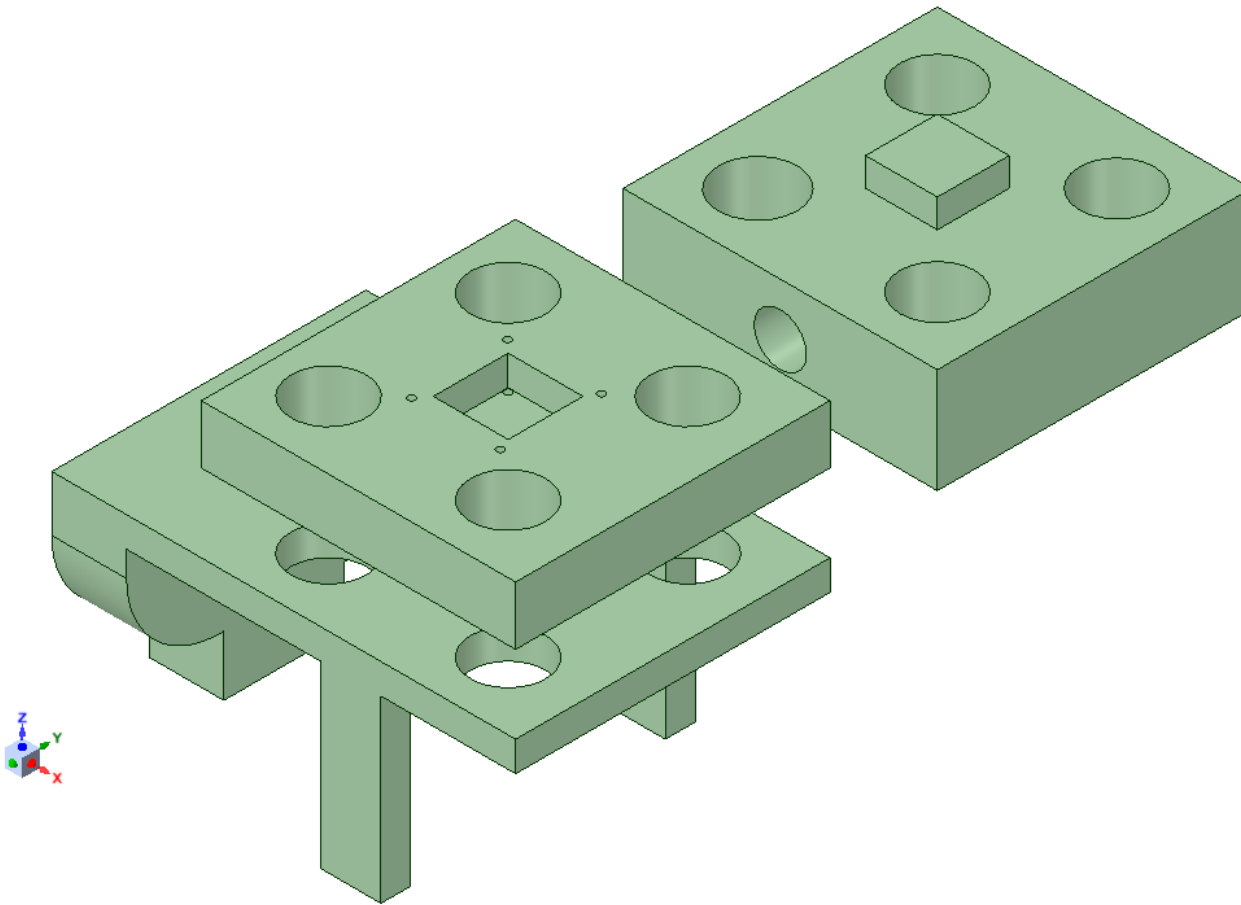


- The experiments were conducted with significant time and cost saving.

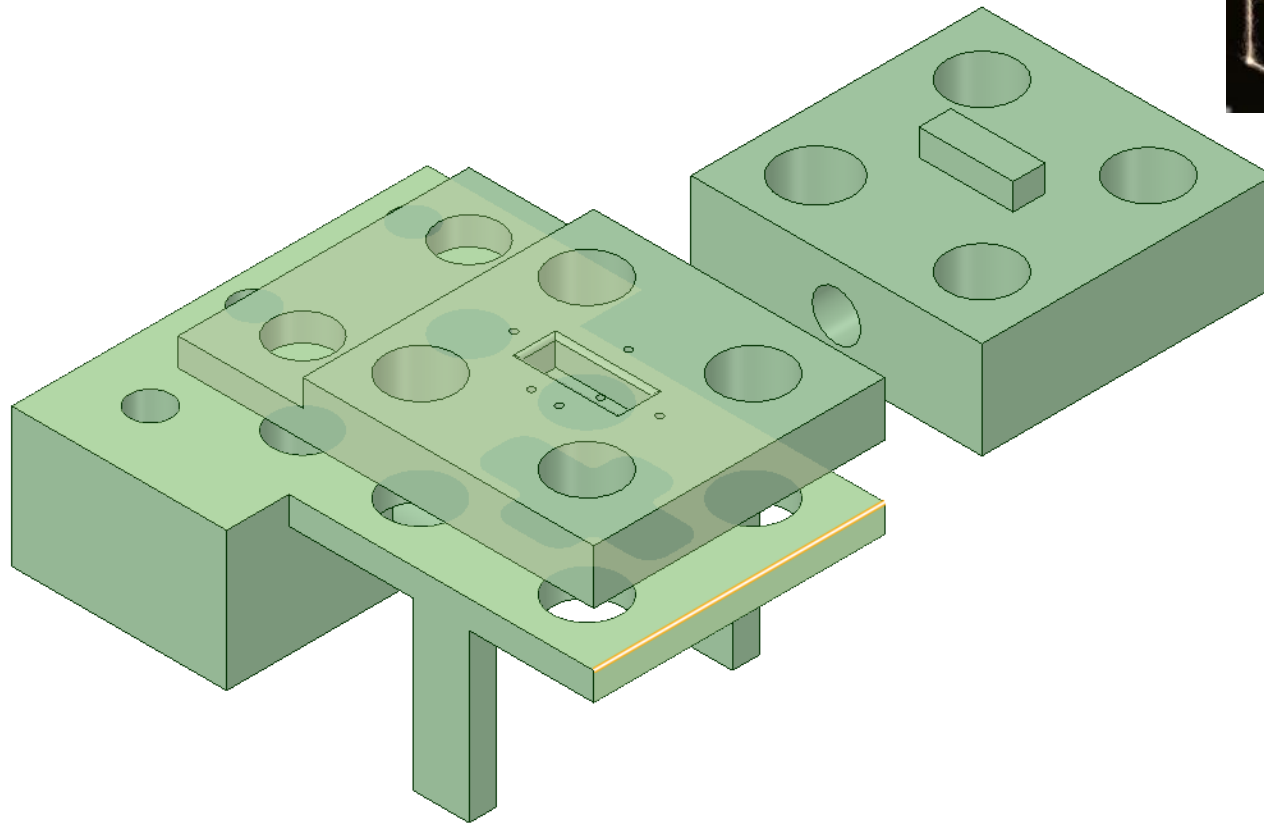
Van der Pauw Greek Cross Cell



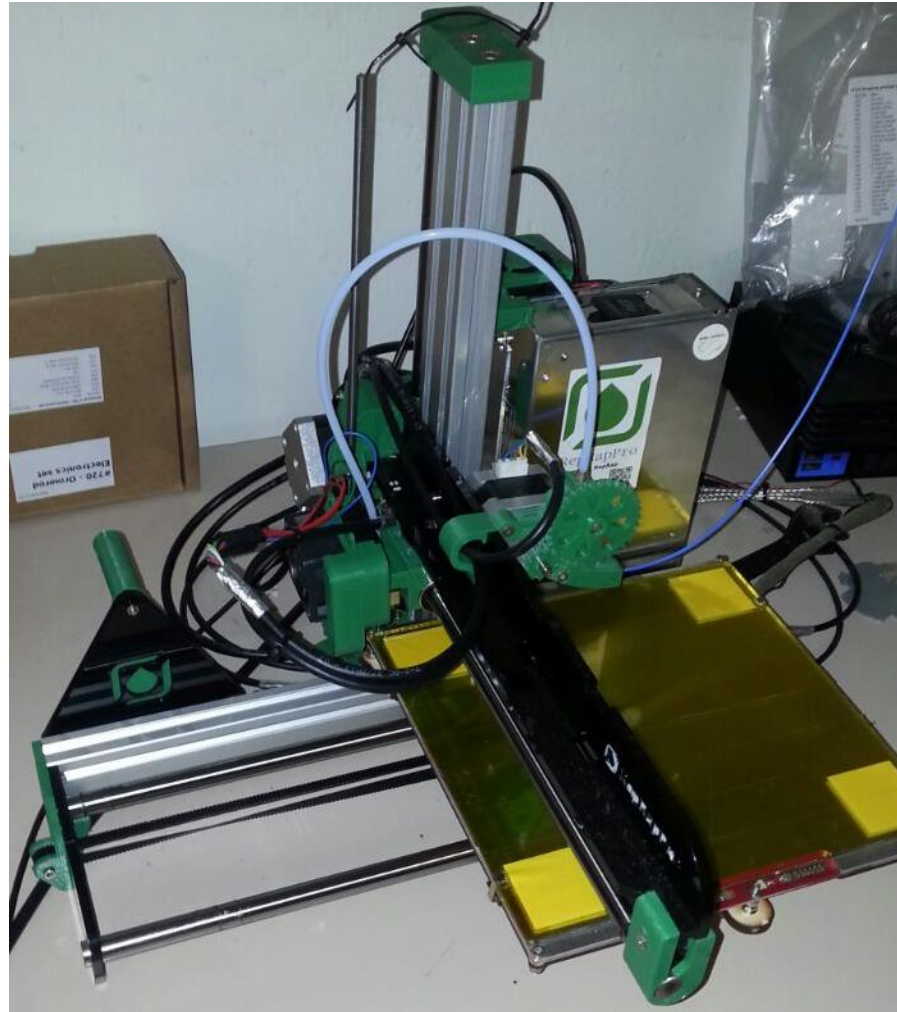
Van der Pauw Square Cell



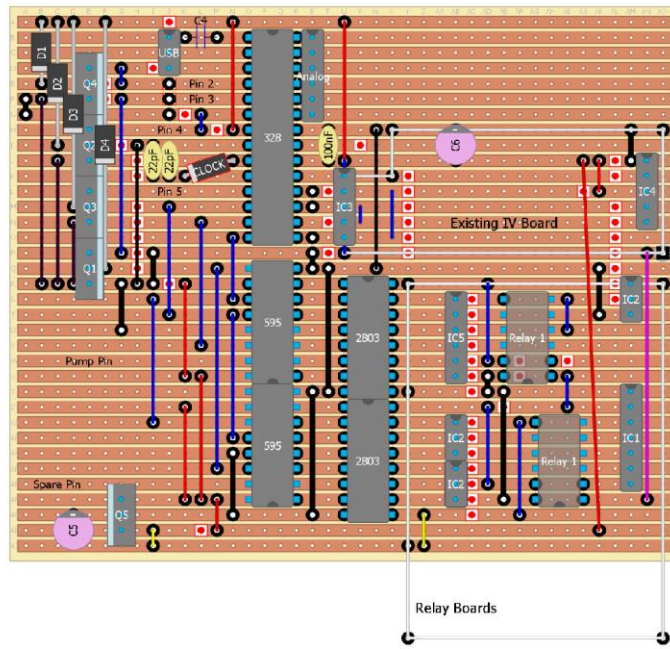
Hall Effect Cell



RepRap Ormerod Printer



Computer Controlled 4 Channel Physical Relay Multiplexer

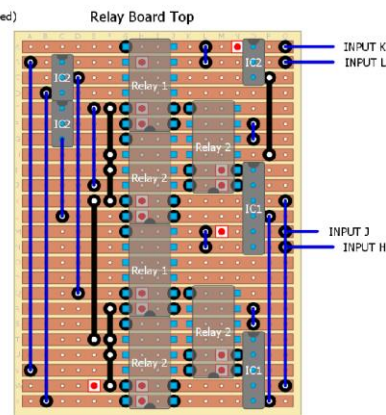
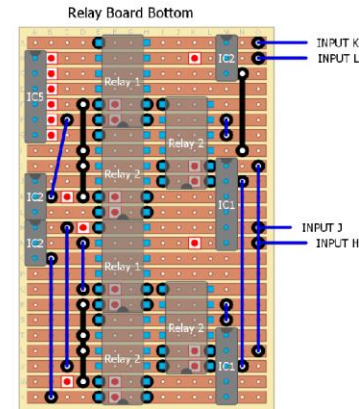


INPUT K
INPUT L

Pin 4 (Sense BLACK)

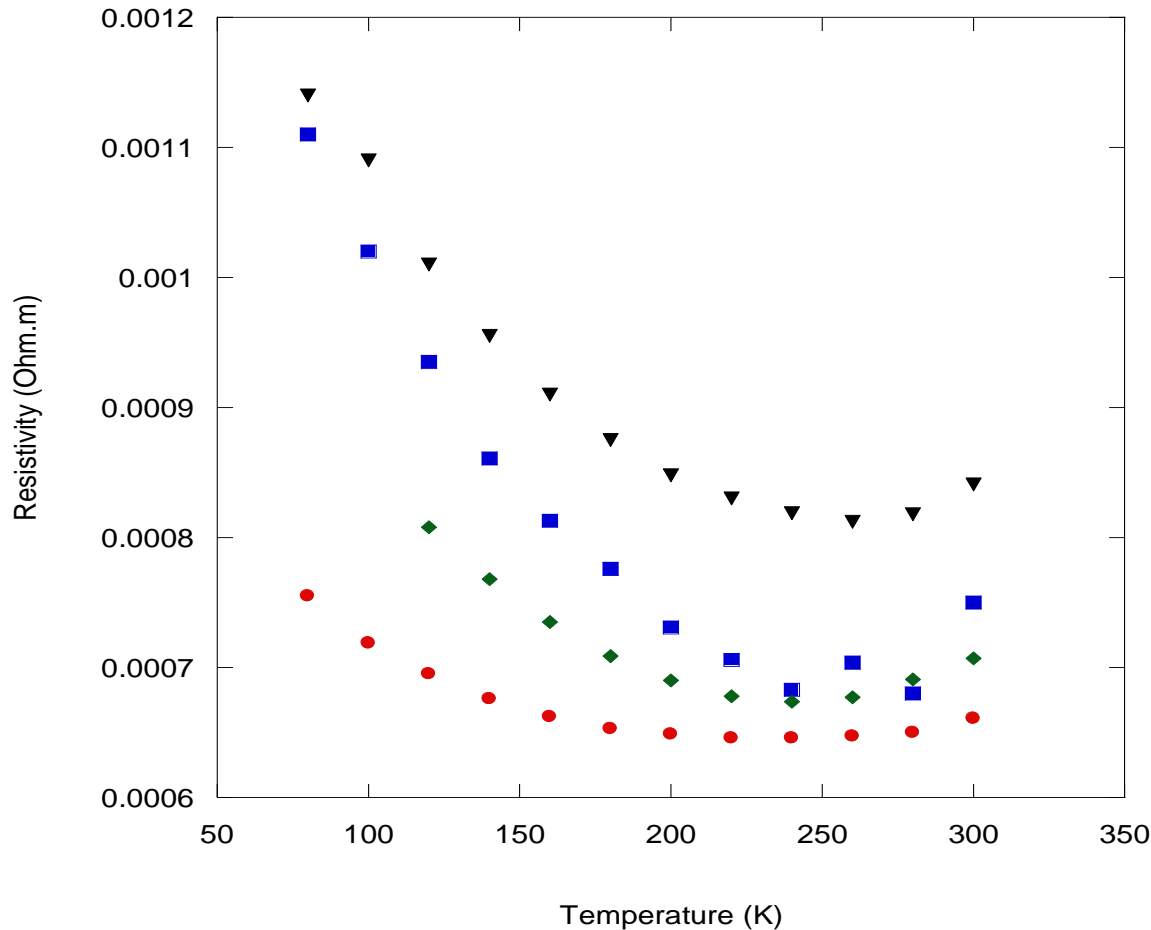
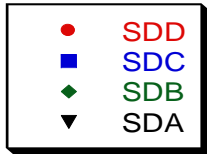
Pin 3 (Sense RED)
Pin 2 (Input Black)
Pin 1 (Input RED)
INPUT J
INPUT H (Switched)

INPUT H



VDP Results

Temperature Dependence of the Resistivity of Carbon
Microspheres of Varying Nitrogen Dopant
Concentrations Determined with a Greek Cross Cell

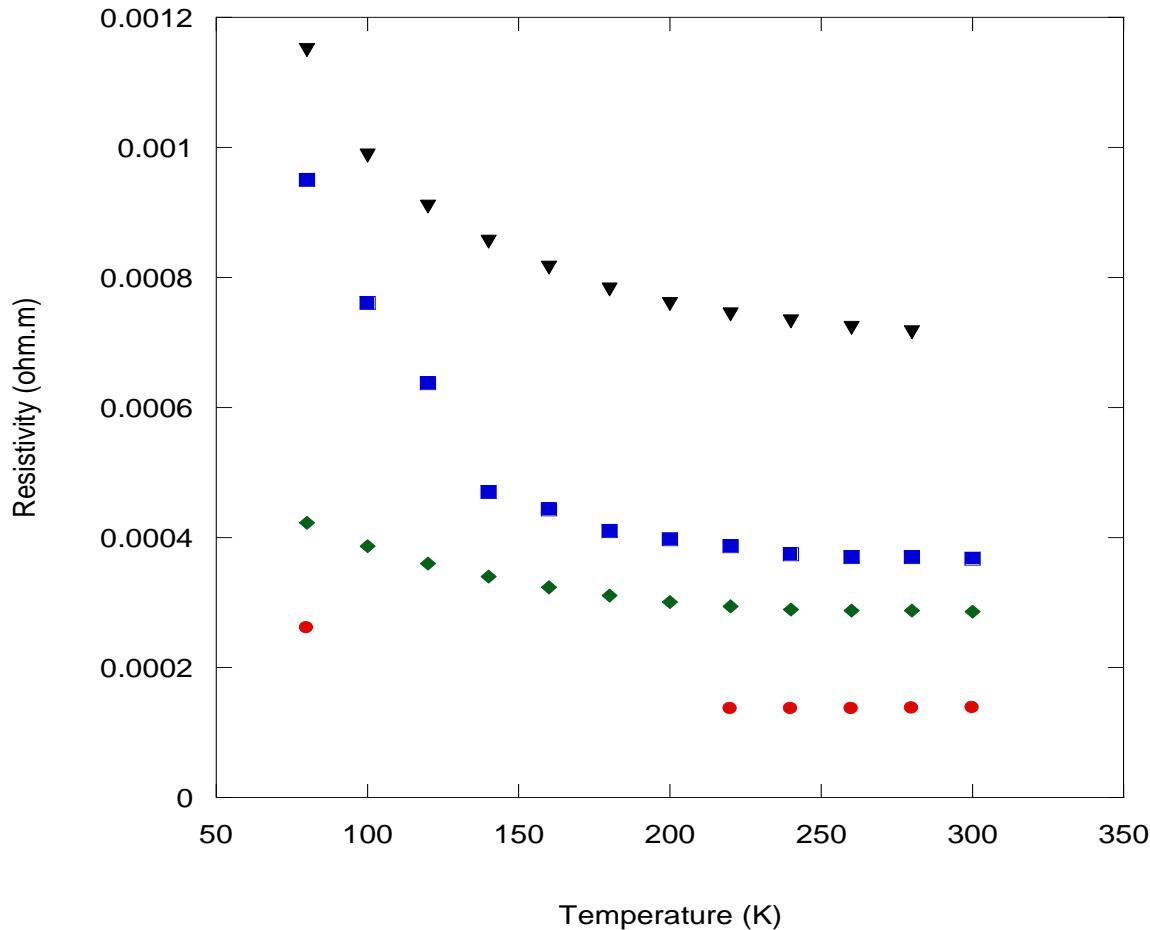
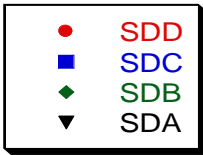


Resistivity decreases
with Nitrogen
Dopant level.

Metal-Insulator
transition likely caused
by contacts.

VDP Results

Temperature Dependence of the Resistivity of Carbon
Microspheres of Varying Nitrogen Dopant
Concentrations Determined with a Square Cell



Increasing dopant level increases the semiconducting behaviour.

VRH and FIT likely models for conduction.

$$R_H \approx -1.1537e-07$$



Conclusions

- N-doped CNS have been successfully produced using a horizontal CVD reactor.
- The nitrogen is strongly paramagnetic indicating that the nitrogen is in substitutional sites
- EPR can be used as a characterisation technique for determining dopant level.
- Open source technologies allow for substantial cost savings in producing research.
- Developing an MPRI “open source” community to improve sharing of ideas and technologies is imperative to increase research output.



Acknowledgements

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