

Utilization of Non-Destructive Tools for In-Situ Determination of Hydrogen Content in Advanced Materials

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G2MT



Report Documentation Page

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Outline

- Introduction
- Common Factor Between Electronic Tools and Hydrogen Measurements
- Non-Destructive Hydrogen Content Sensors
 - Thermoelectric Power Measurements
 - Low Frequency Impedance Measurements
- Results
- Summary



NDE Progress and Challenges

The NDE Community has made impressive advancements in assessment of material defects and increased structural integrity

New Challenges:

- To Assess Material Health
 - - Aging - Properties - Specifications
 - - Stability - Strain State
- To non-destructively characterize material in technical assemblies with electronic, magnetic, and elastic metallography techniques
- To be integrated in agile vertical manufacturing systems
- To rapidly perform real-time testing, data acquisition, and assessment, to qualify materials during manufacturing

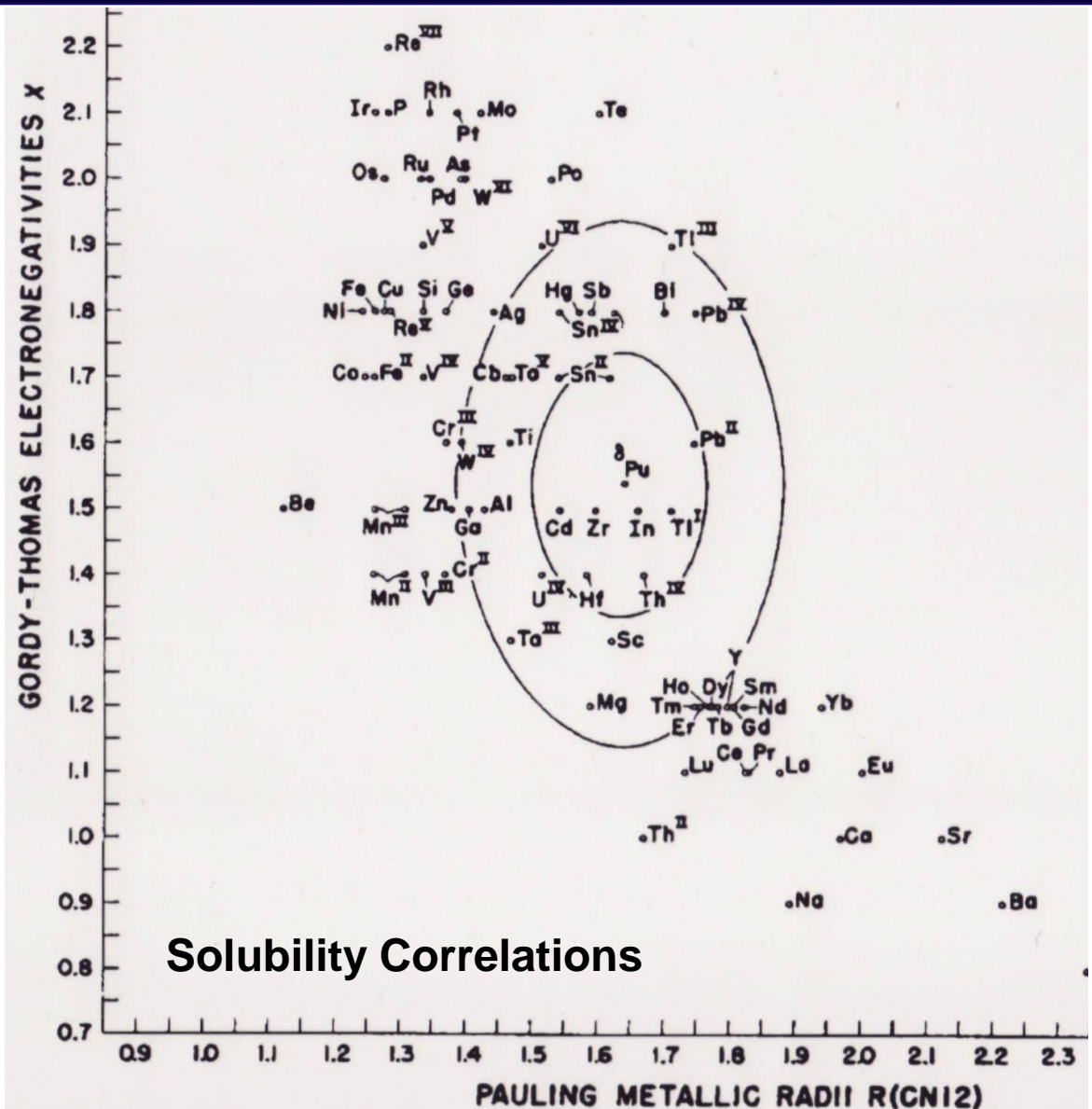
Early Metallurgical Approach

*Hume-
Rothery*

*Darken-
Gurry*

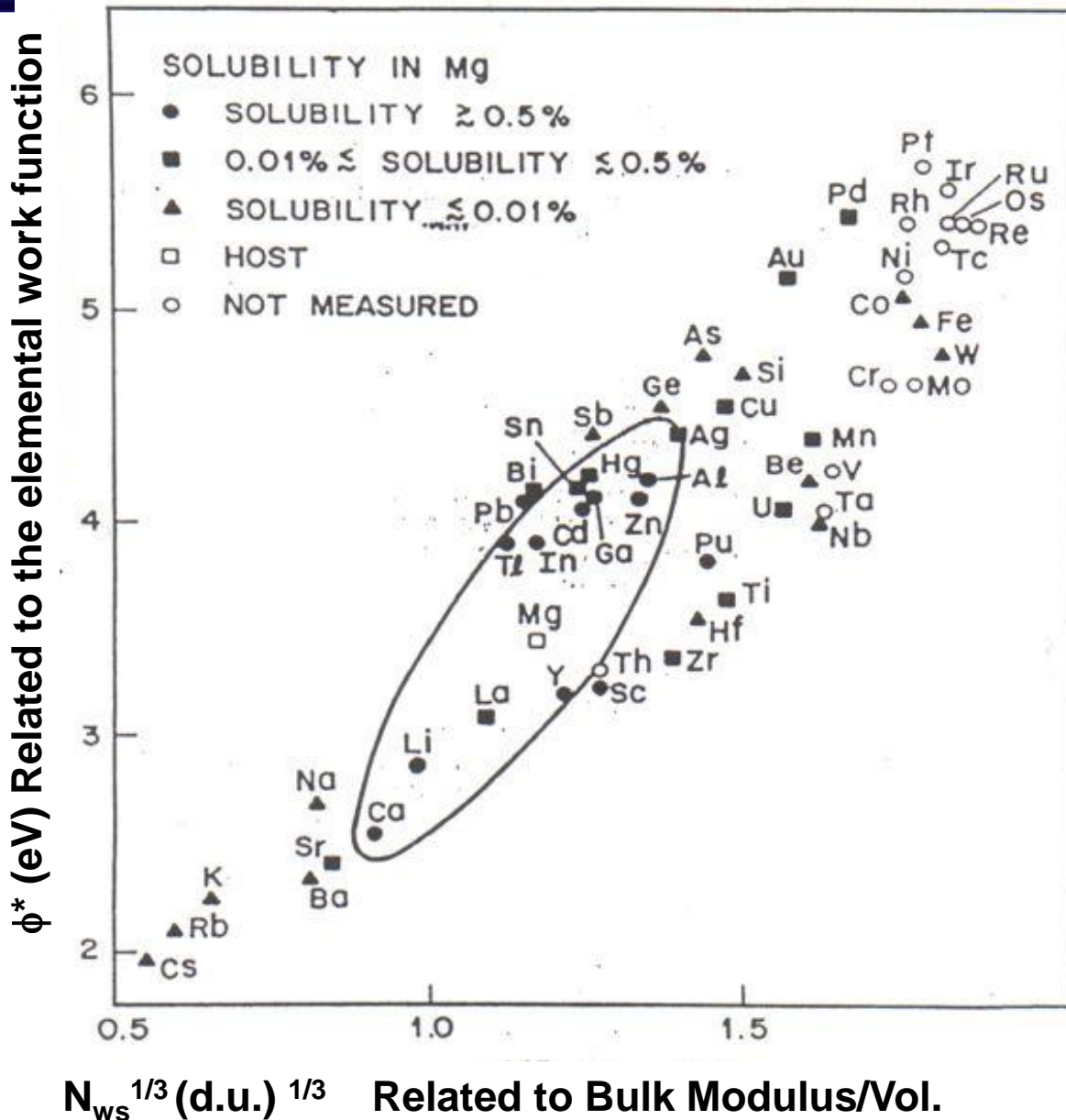
Gschneider

Waber

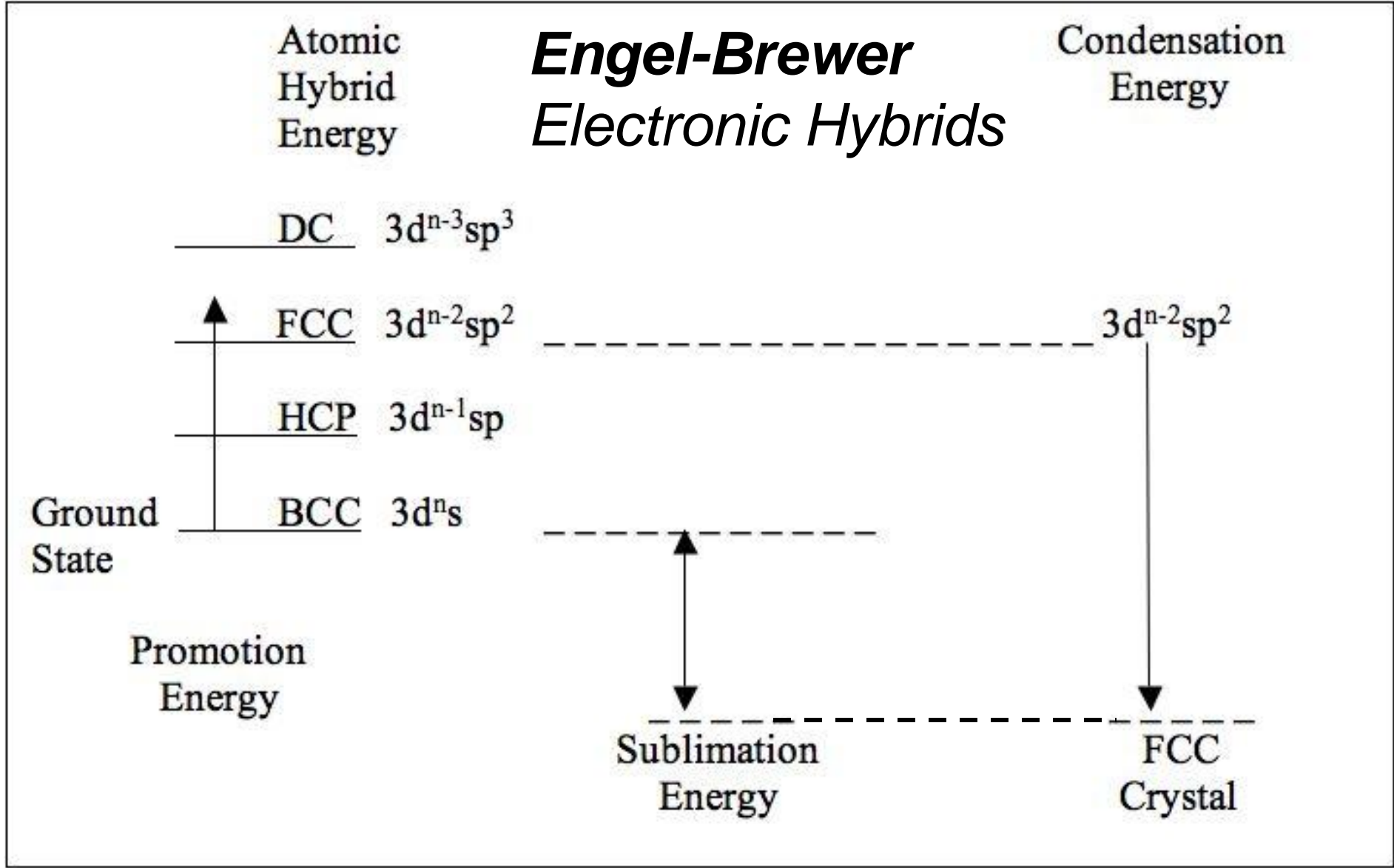


Early Metallurgical Approach

Miedema-Chelikowsky



Early Metallurgical Approach



Electronic Property Crystal Structure Correlation

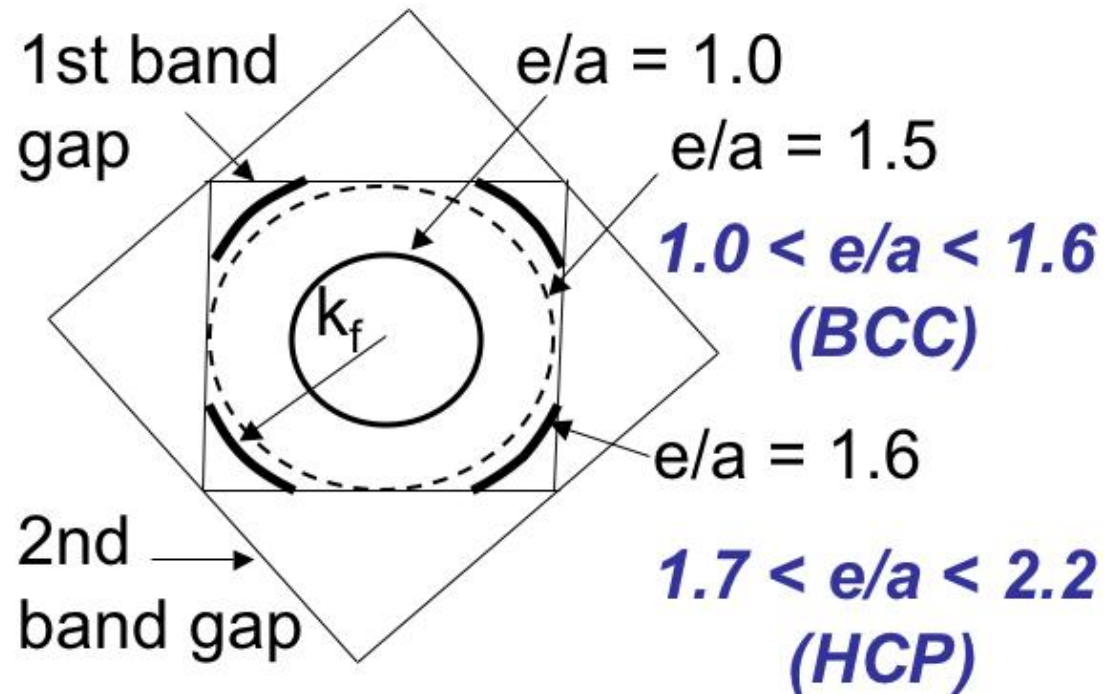
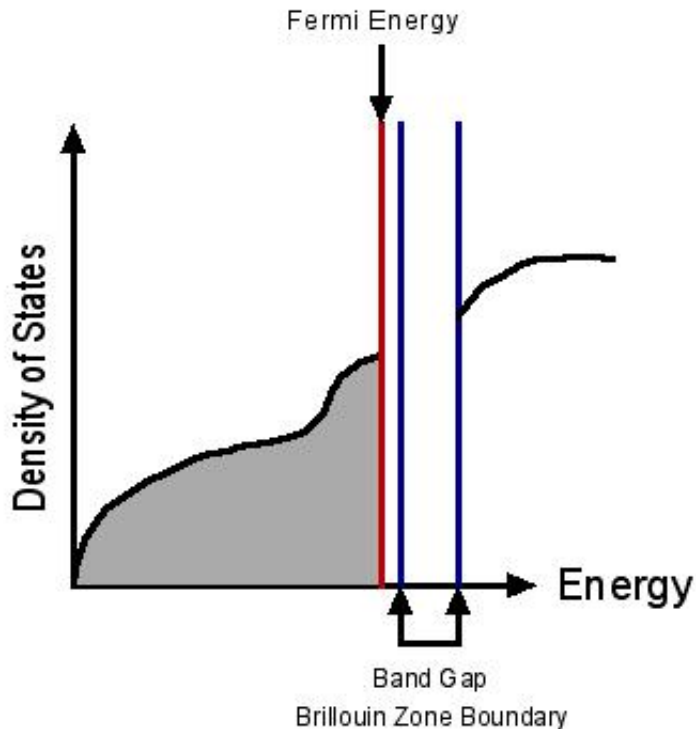
Brewer

Crystal Structure	Elements e/a ratio	Alloys e/a ratio	Electronic Configuration
BCC	1	< 1.5	$d^n s$
HCP	2	1.7 to 2.1	$d^n sp$
FCC	3	2.5 to 3.0	$d^n sp^2$
Diamond	4	>3.5	$d^n sp^3$

- Consider the number of unpaired s and p electrons

Early Physics Approach

Intro to Wave Mechanics - *Mott and Jones* - *Ziman*



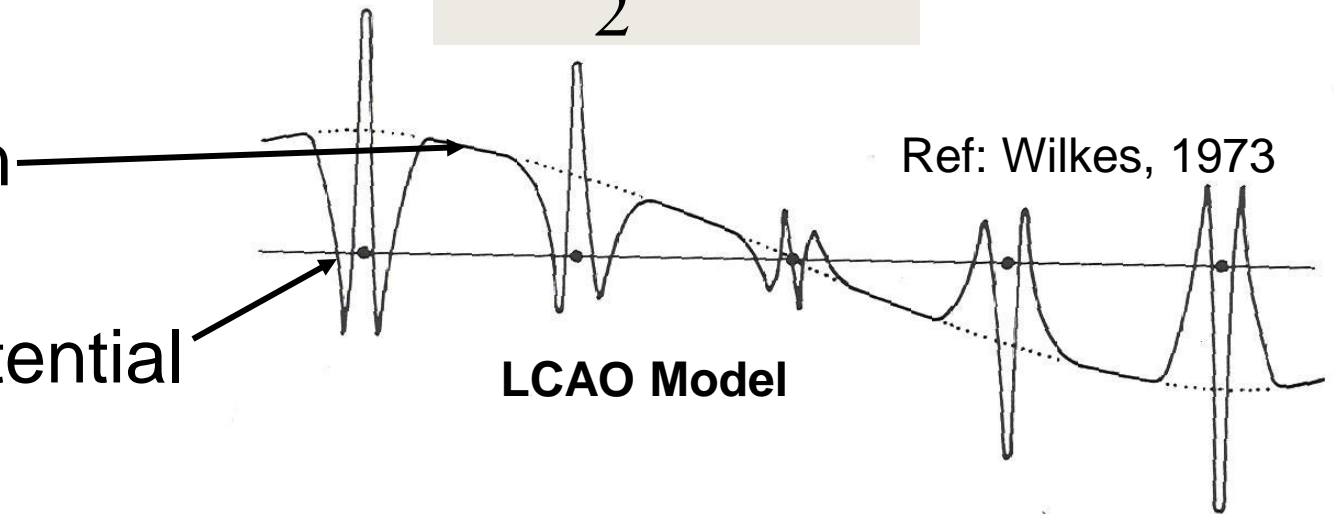
Effective Mass

- Electron wave function is modified by localized potentials

$$E = \frac{1}{2} mv^2 + V$$

- Free Electron Wave (-----)

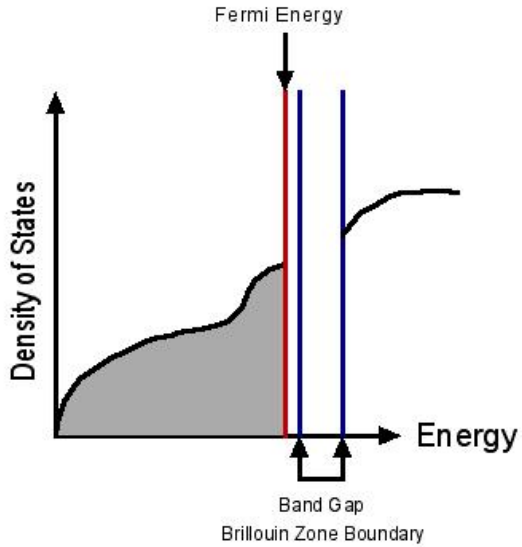
+ Localized Potential



$$E = \frac{1}{2} mv^2 = \frac{P^2}{2m} = \frac{\hbar^2 k^2}{2m_e}$$

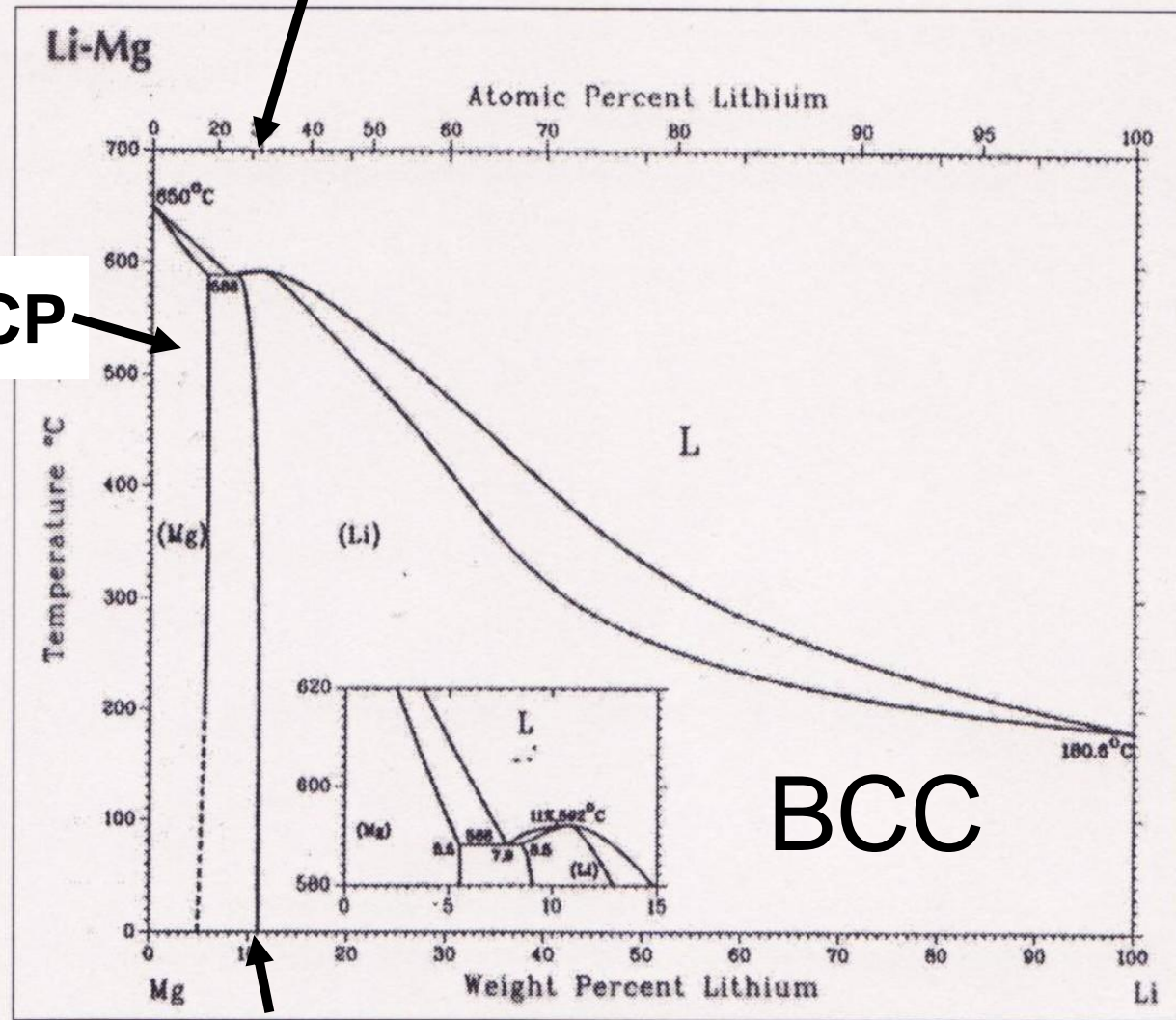
$$m_e = \frac{\hbar^2}{(d^2 E / dk^2)}$$

Correlation of Phase Diagram to Electronic Filling of Band Structure

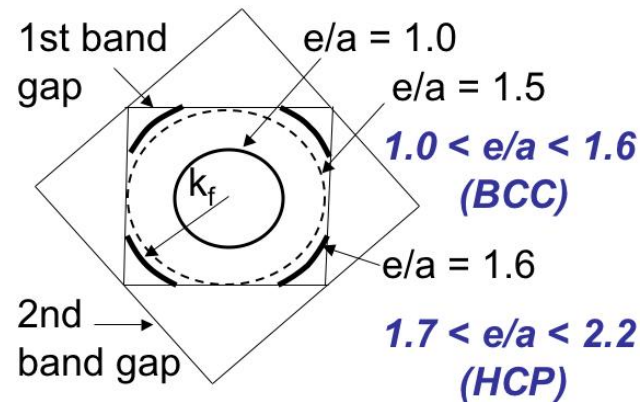


$e/a \sim 1.4$

HCP

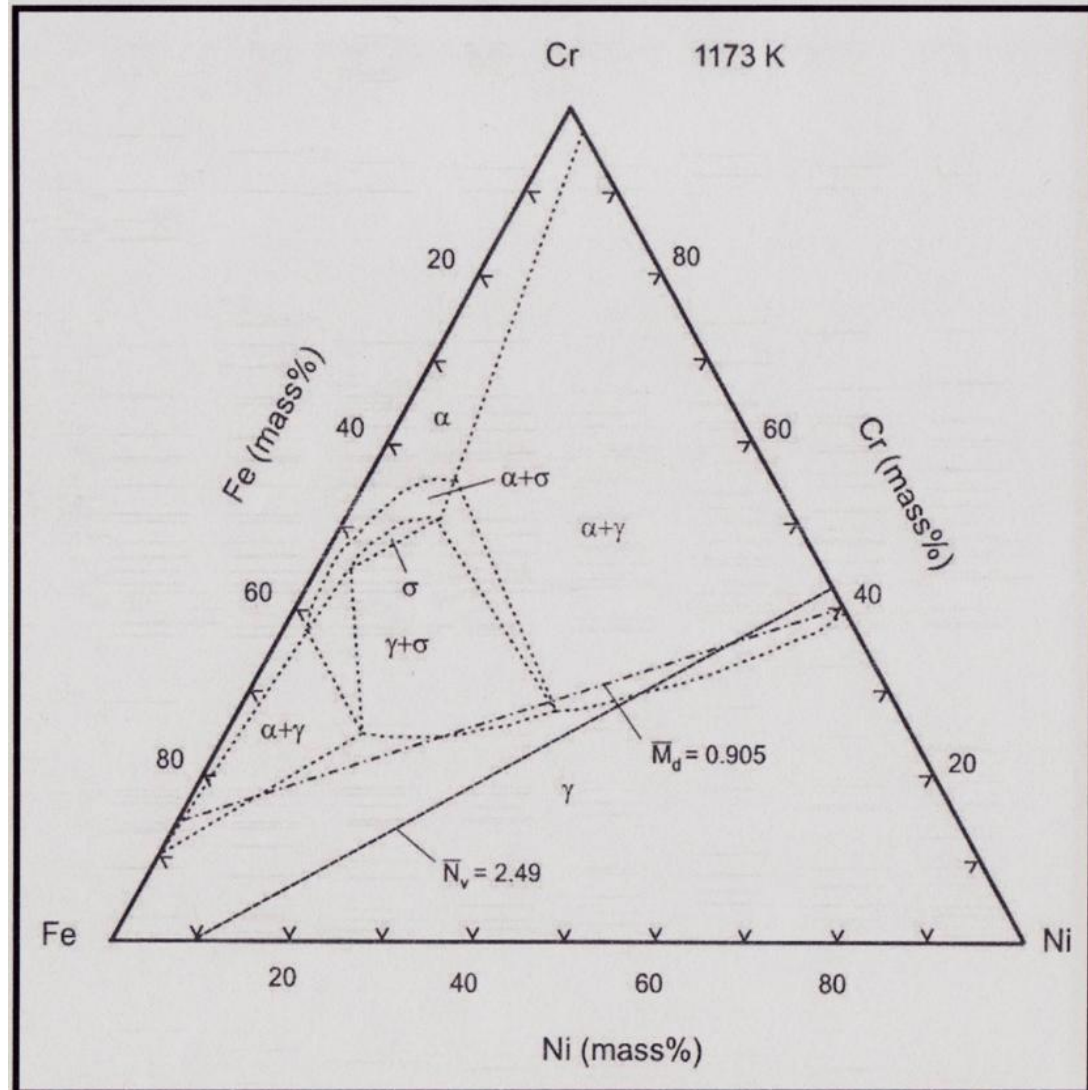


BCC



QUESTION:

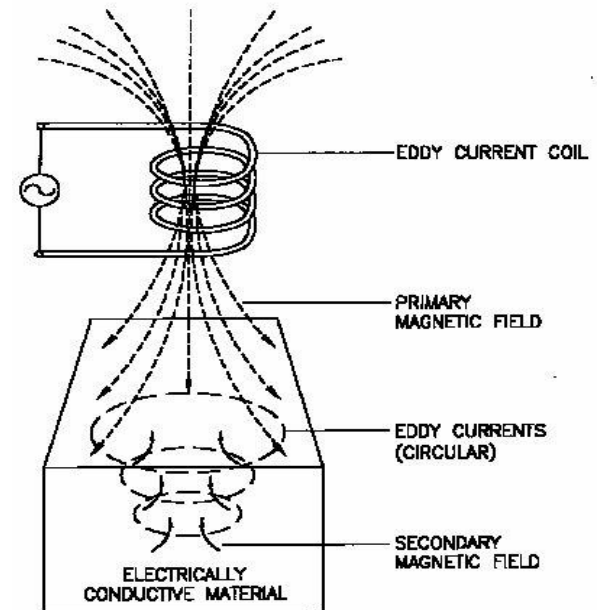
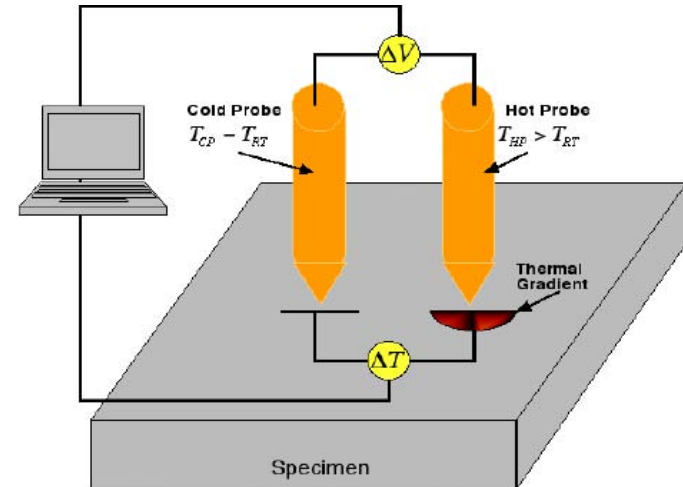
If you can
calculate e/a ,
why can't you
measure it?



Electronic Property Measurement Tools

- Thermoelectric Power
 - Contact Technique

- Low Frequency Impedance Measurements
 - Non-Contact Technique

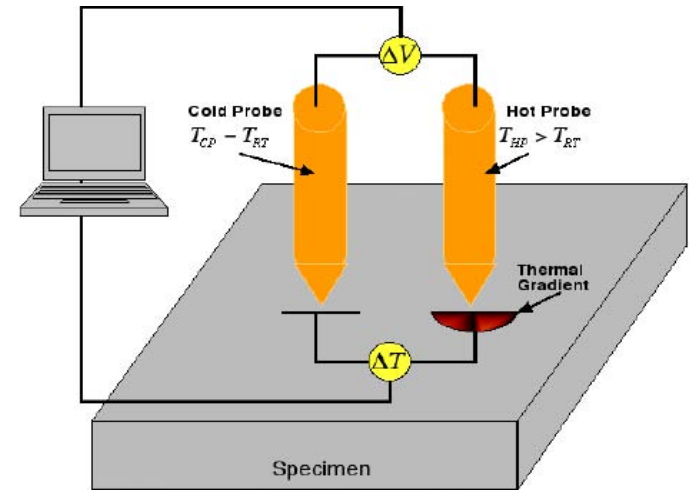


Quantum Mechanical Principle of Thermoelectric Power

$$S = \left(\pm \frac{k}{e} \right) (27.1) \left(r + \frac{3}{2} \right) \left(\frac{m_e}{h^2} \right) \left(kTn \right)^{\left(-\frac{2}{3} \right)}$$

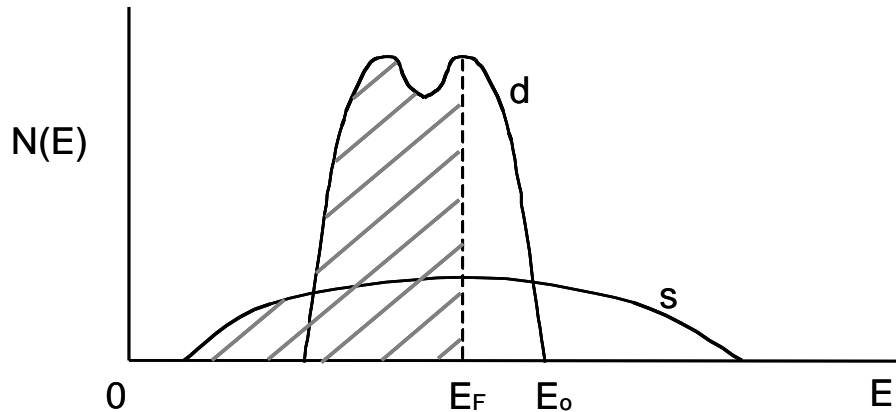
$$S = S(n, m_e, r)$$

- S Thermoelectric power
- r Scattering parameter
- h Planck constant
- k Boltzmann's constant
- n Free electron concentration
- m_e Effective mass (m^*)

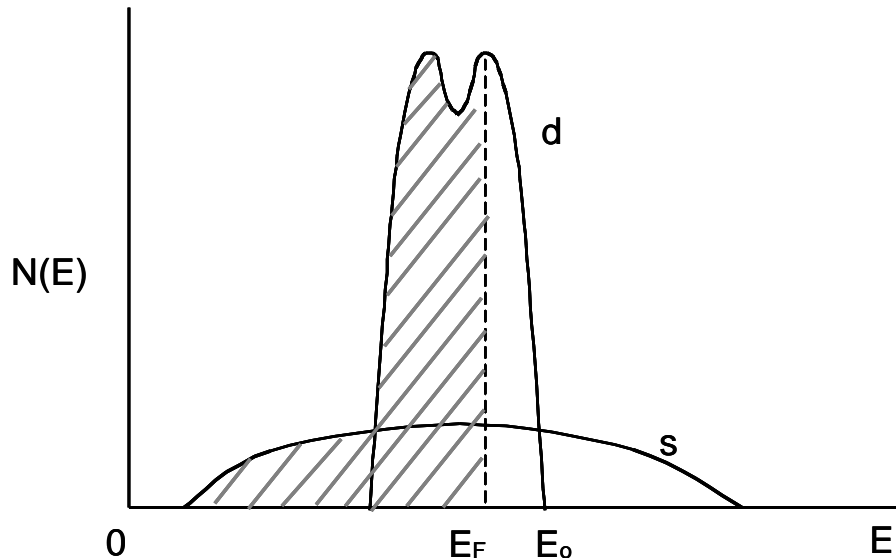
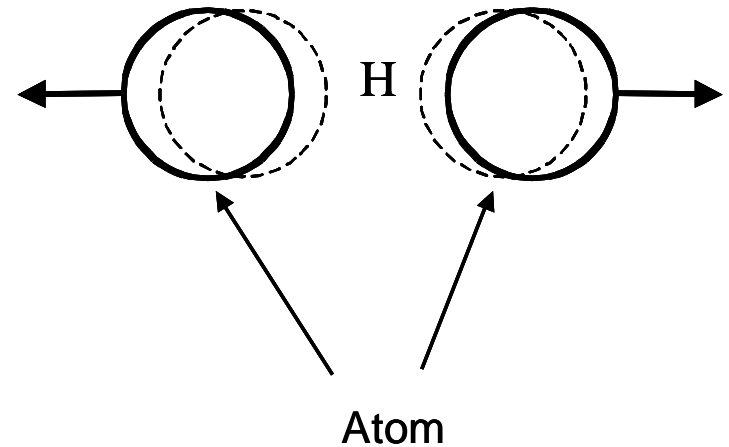


$$m_e = \frac{\hbar^2}{(d^2 E / dk^2)}$$

Electronic Nature of Hydrogen

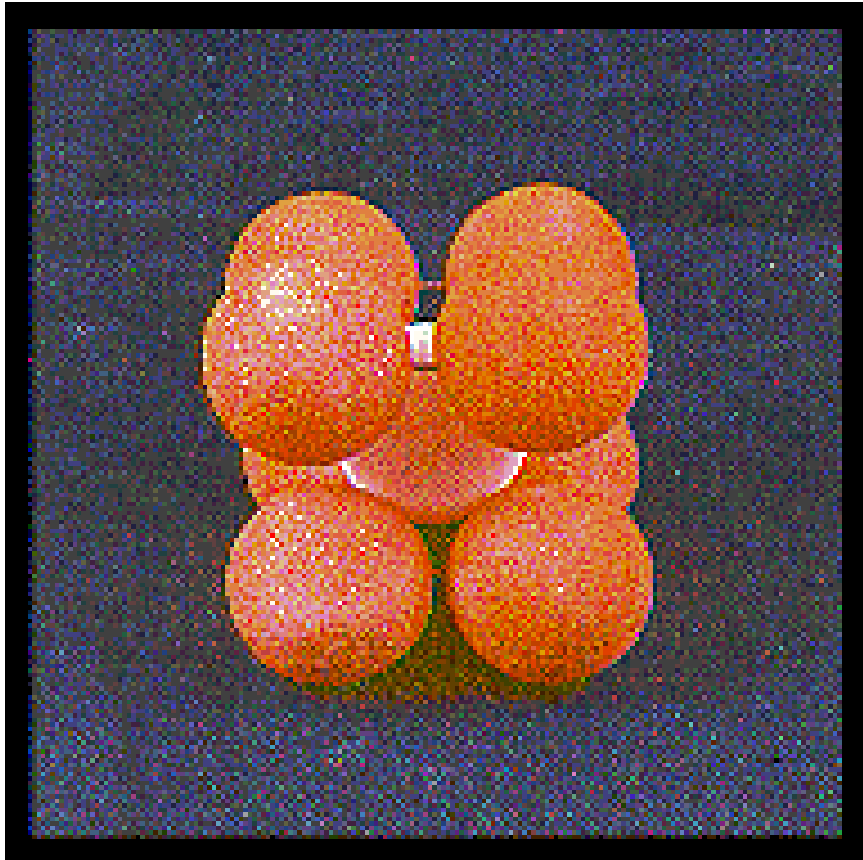


$$E_F = \frac{\hbar^2 k^2}{2m_e}$$



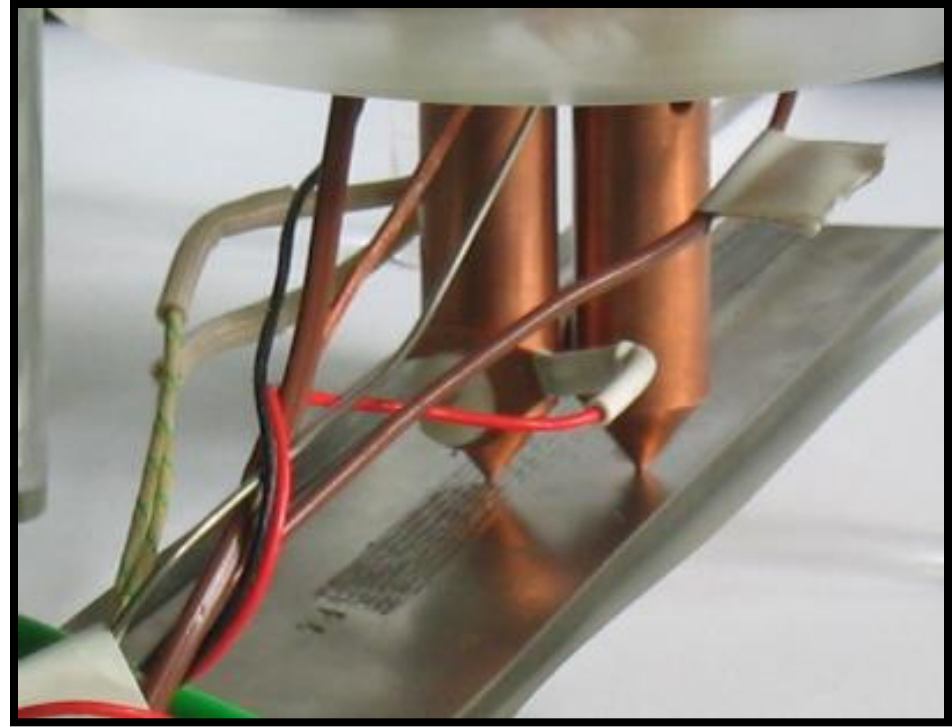
$$m_e \propto \frac{1}{(d^2 E / dk^2)}$$

Hydrogen in BCC Interstitial Sites



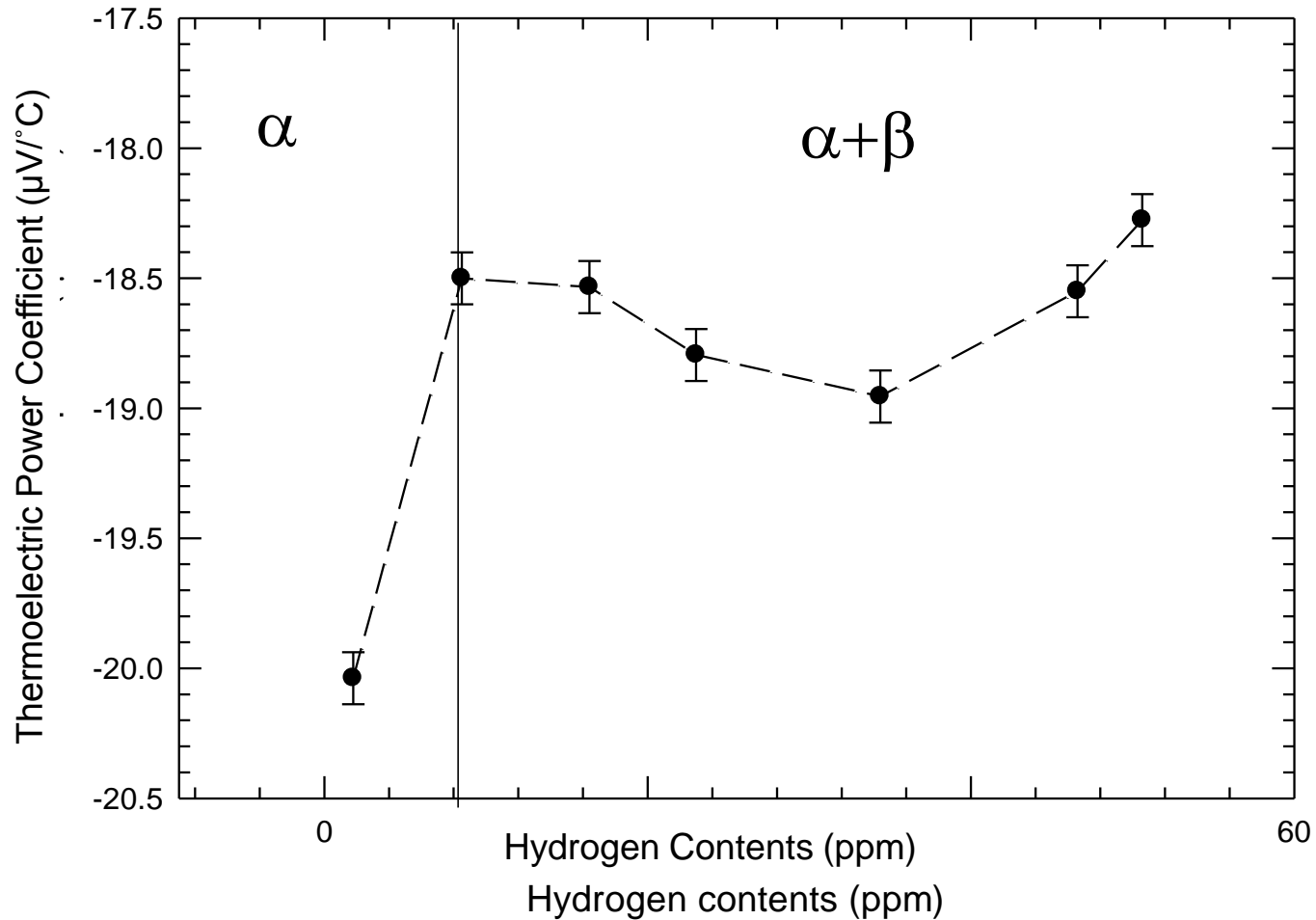
	Tetra- hedral	Octa- hedral
BCC-Iron Interstitial Hole Size	0.36 Å	0.19 Å
H-Filled Interstitial Hole Size	0.87 Å	0.66 Å

Thermoelectric Power Surface Probe

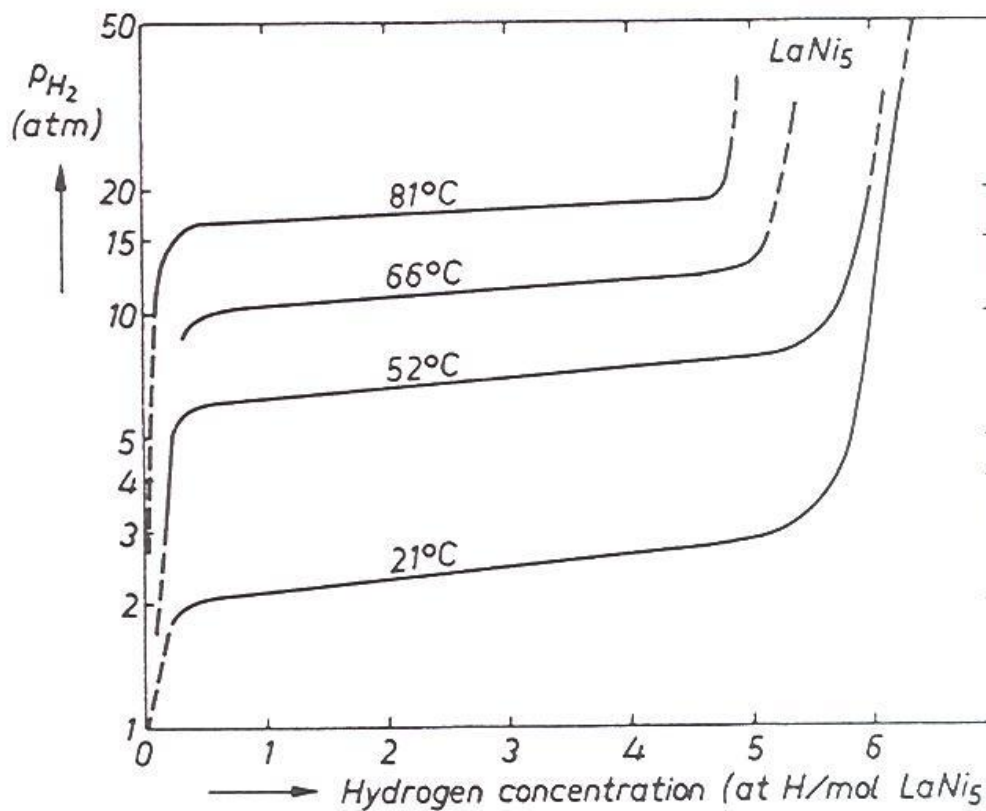
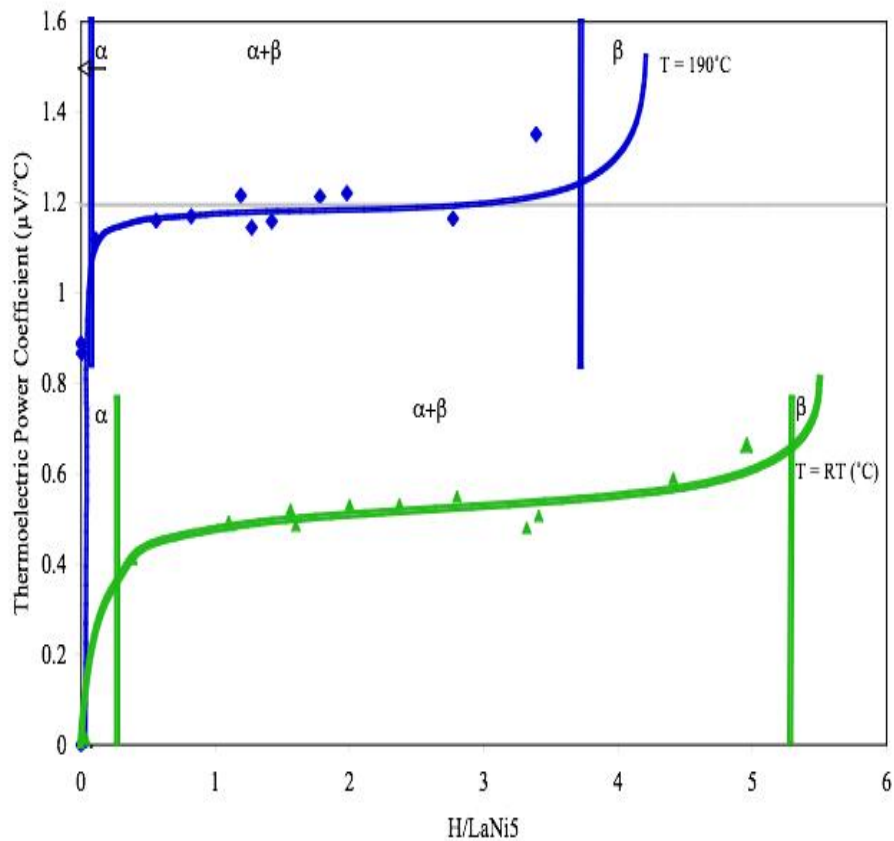


Contact diameter of probe tip: 0.015 inches (381 μ)

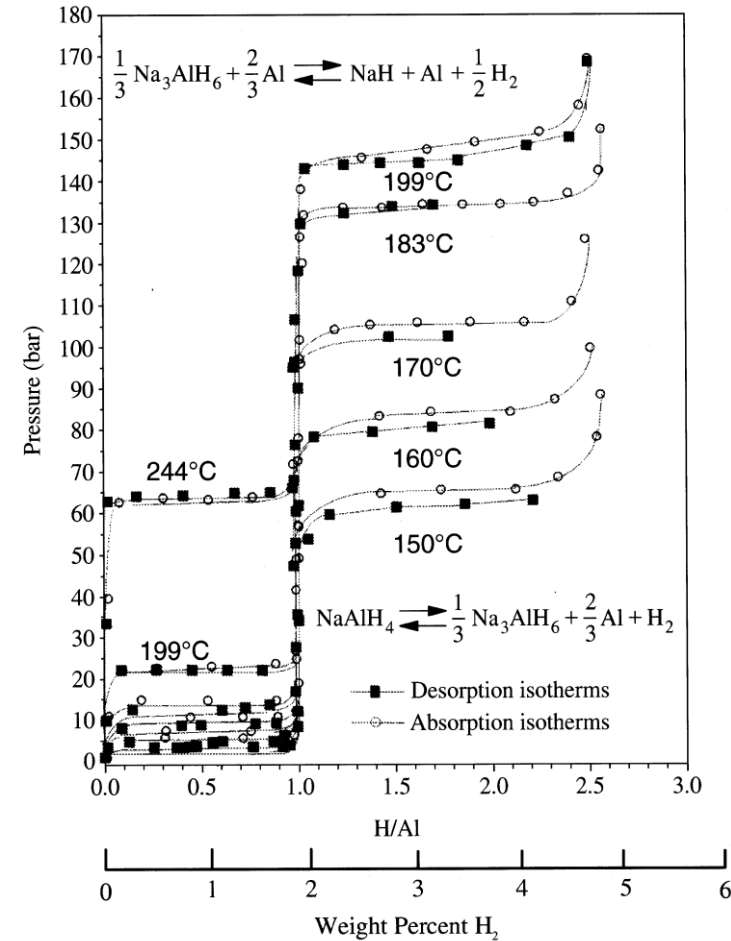
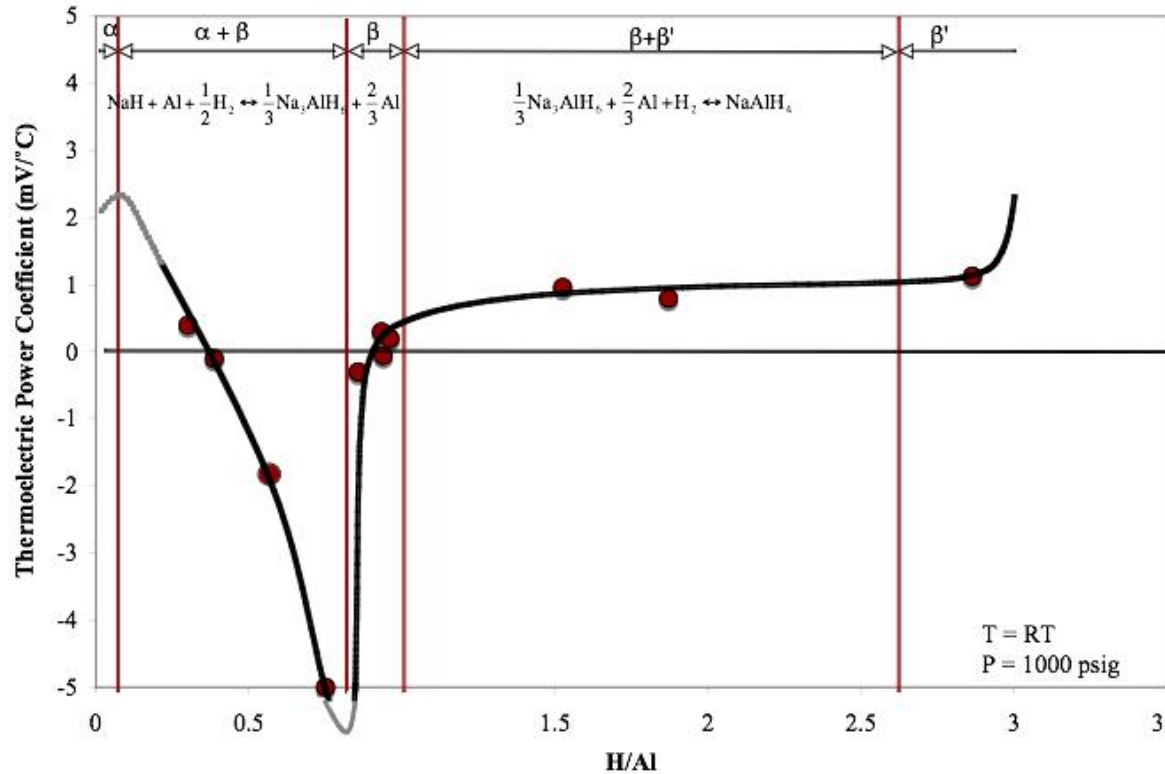
Thermoelectric Power as a Function of Hydrogen in Monel K-500



Thermoelectric Power as a Function of H/LaNi₅



Thermoelectric Power as a Function of H/Al



Eddy Current Analysis

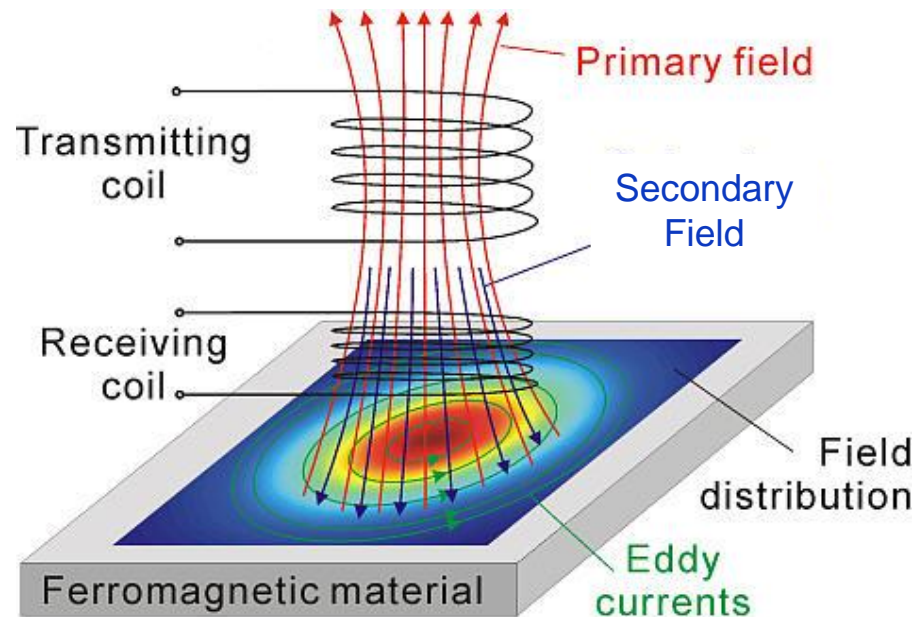
- Eddy current non-destructively measure:
 - Plate and coating thickness
 - Conductivity
 - Differences in composition, microstructure, and properties
 - Cracks, defect, flaws
 - Hardness and physical conditions



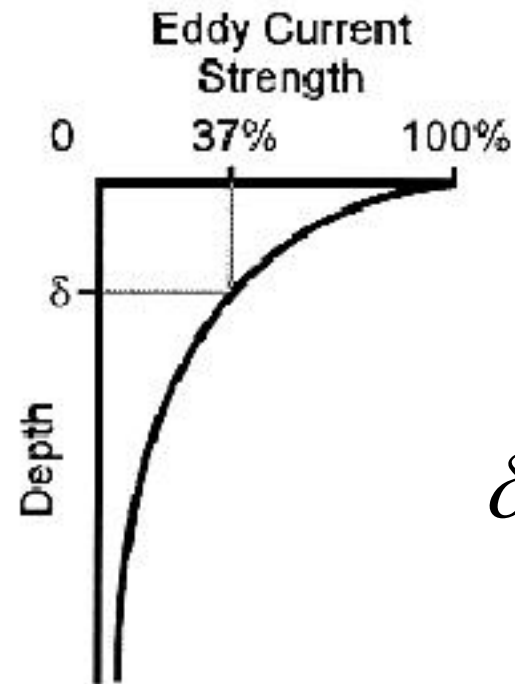
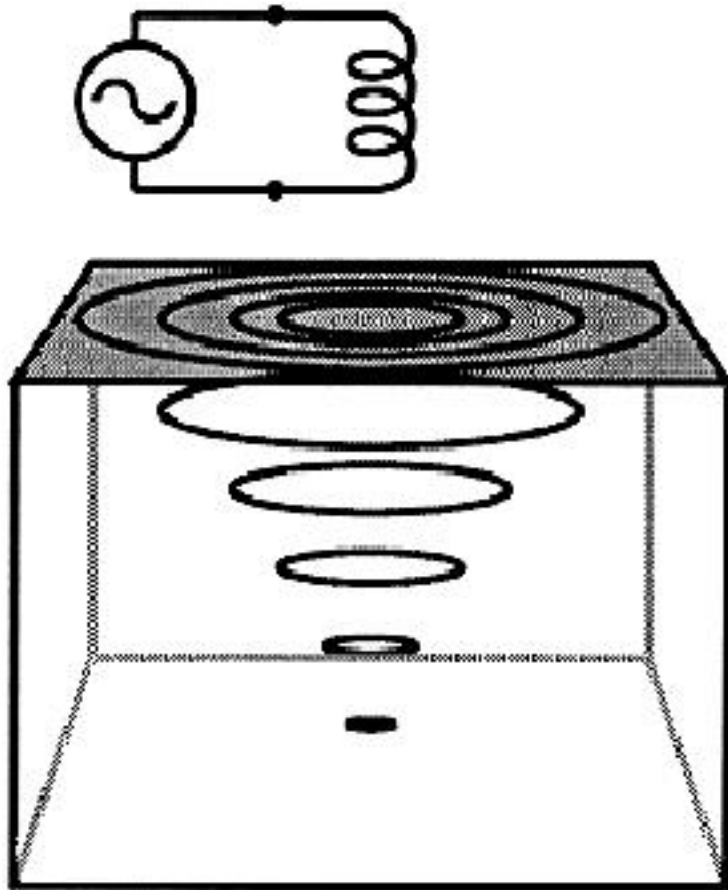
Eddy Current Analysis

- Eddy Currents
 - Spread out into the specimen
 - Will naturally be constrained by the specimen boundaries
 - Circulating currents produce their own secondary flux, Φ_S
 - This secondary flux is in opposition to Φ_P
- The coil now senses an equilibrium flux:

$$\Phi_E = \Phi_P - \Phi_S$$

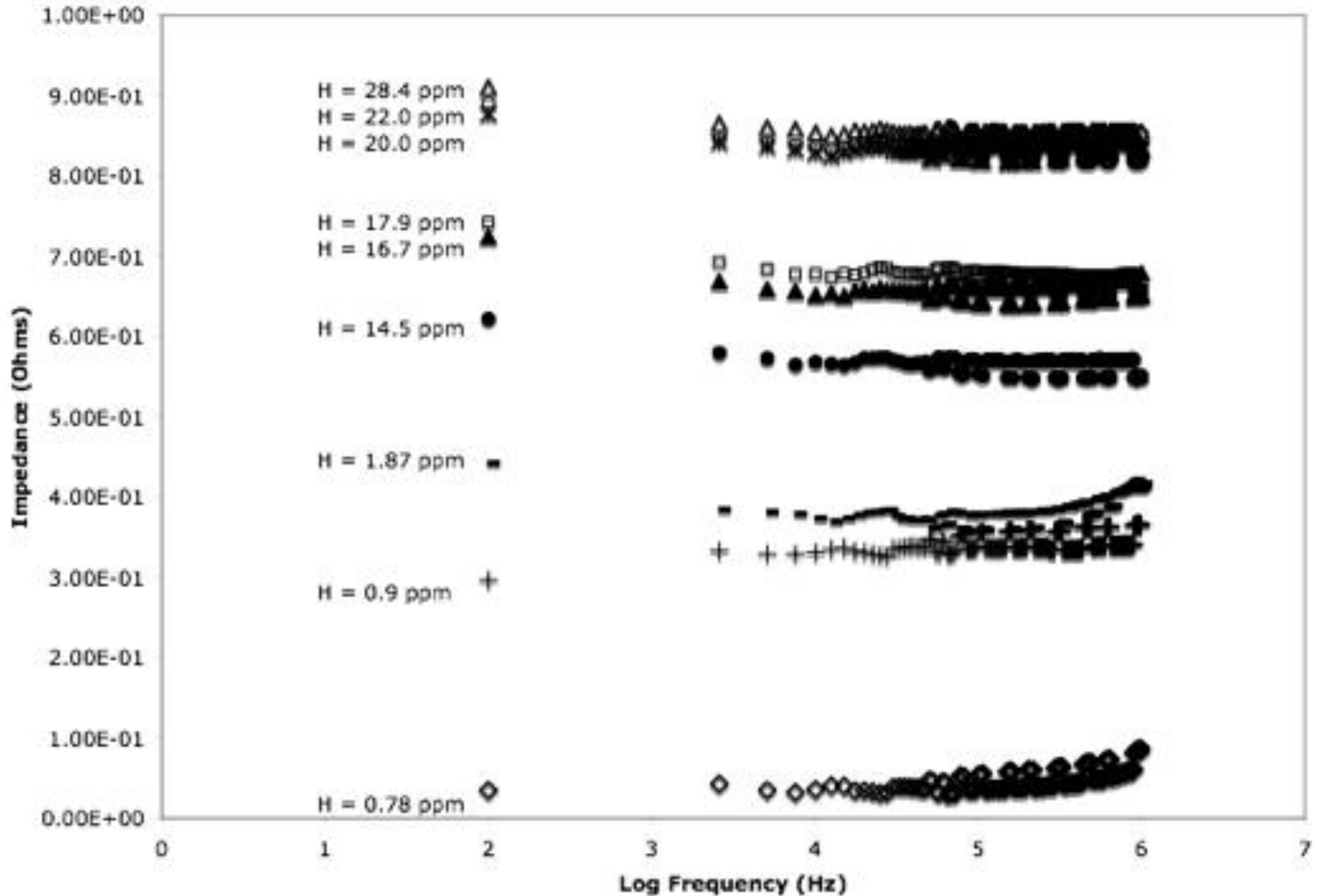


Eddy Current Theory

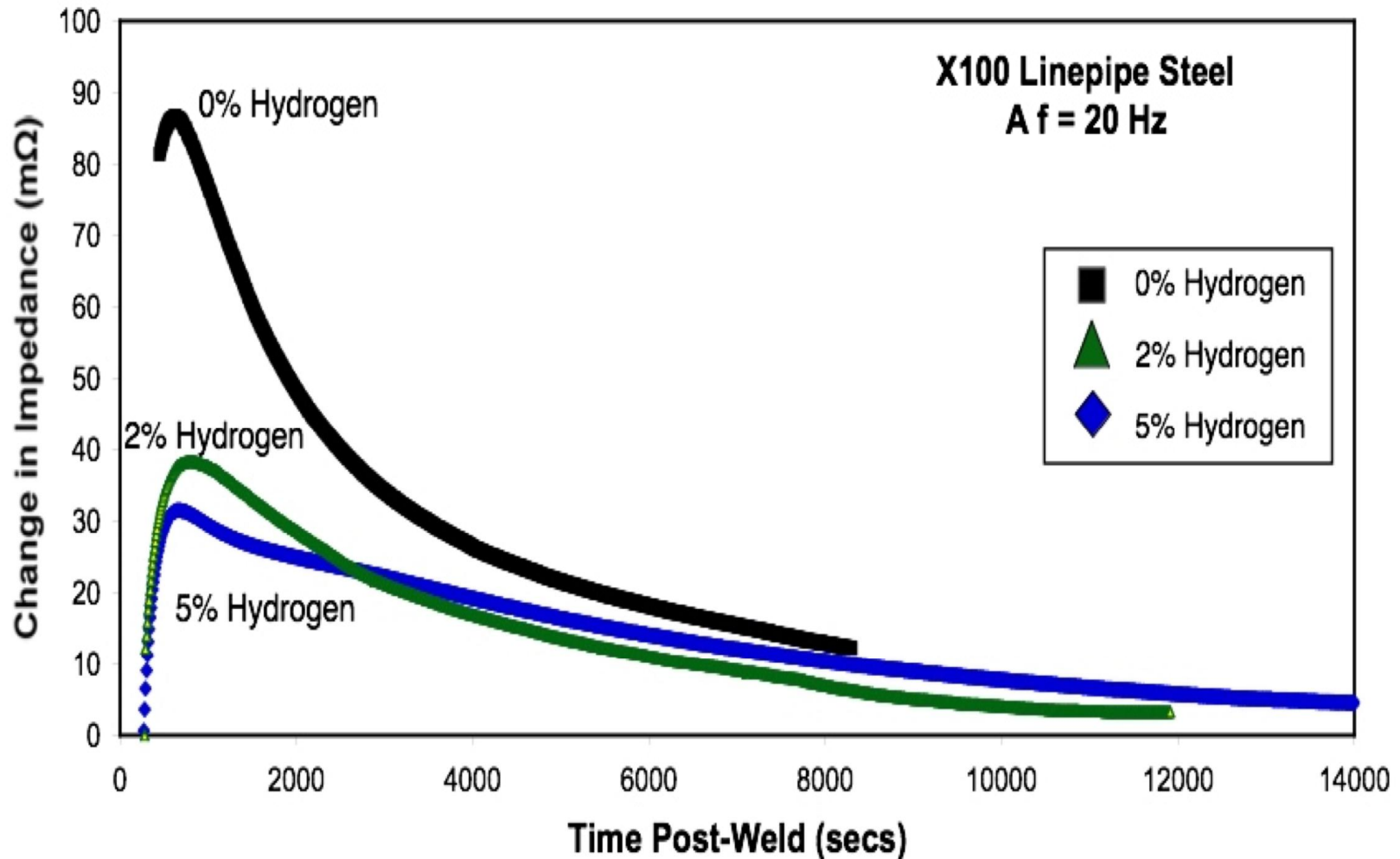


$$\delta \propto \frac{1}{f^{1/2}}$$

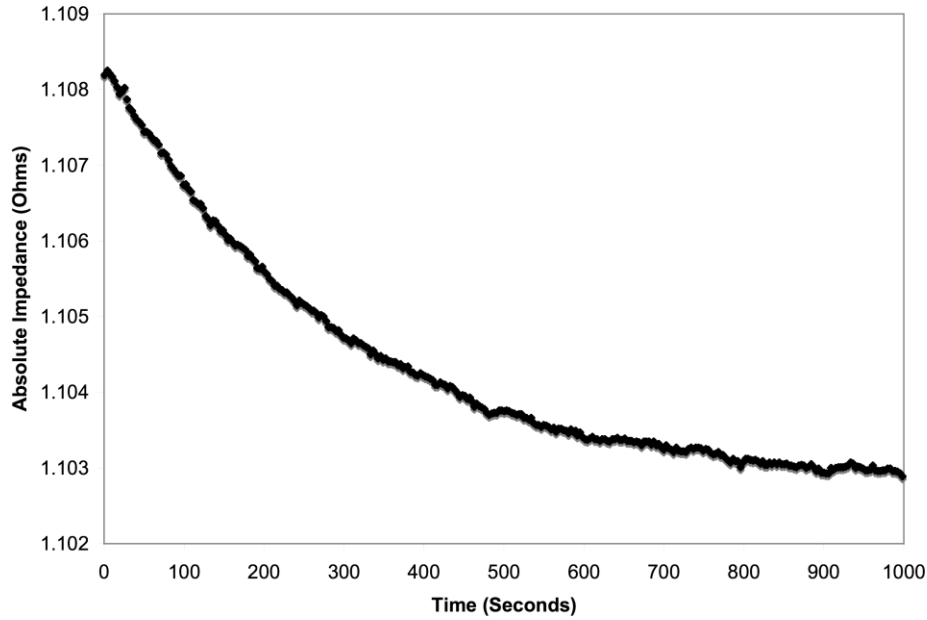
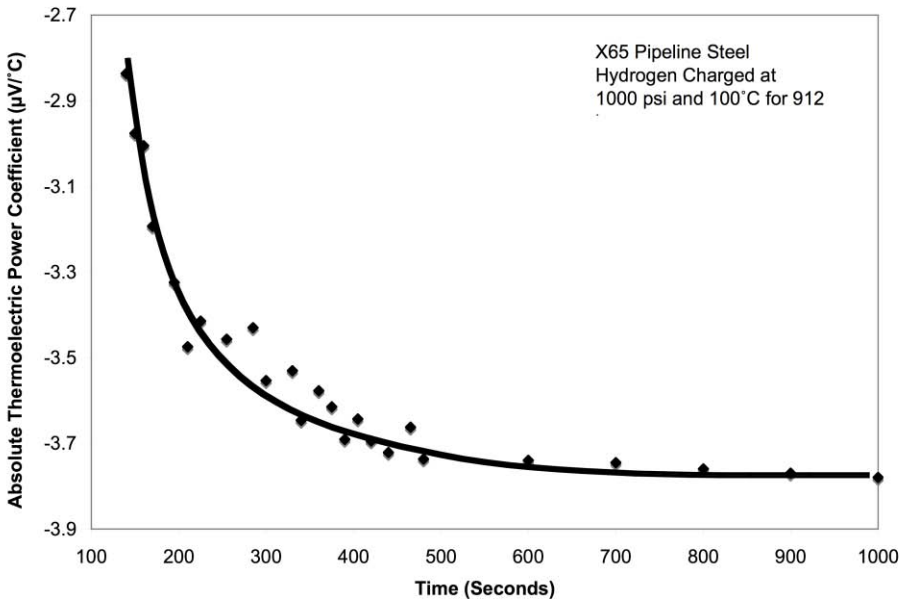
Low Frequency Impedance Measurements



Impedance as a Function of Time as Hydrogen Diffuses out of Steel Weld Metal



Thermoelectric Power and Impedance as a Function of Time



Additional Non-Destructive Tools

- **Electronic**
 - Conductivity
 - TEP
 - Hall Effect
- **Magnetic**
 - Susceptibility
- **Electromagnetic**
 - Eddy Current
 - EM-Acoustic -> Barkhausen Noise
 - Electromagnetic Radiation (X-ray, gamma, and tera)
- **Elastic**
 - Acoustic Emission – Kaiser Effect
- **Thermal Analysis**
 - Infrared

Summary

- Both thermoelectric power and low frequency impedance measurements successfully provide real-time, non-destructive, hydrogen content measurements in advanced materials.

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- Agilent Technologies
- Holland Consulting
- TMR Exploration
- Blade Energy



Diffusion Coefficient of Hydrogen in Steel

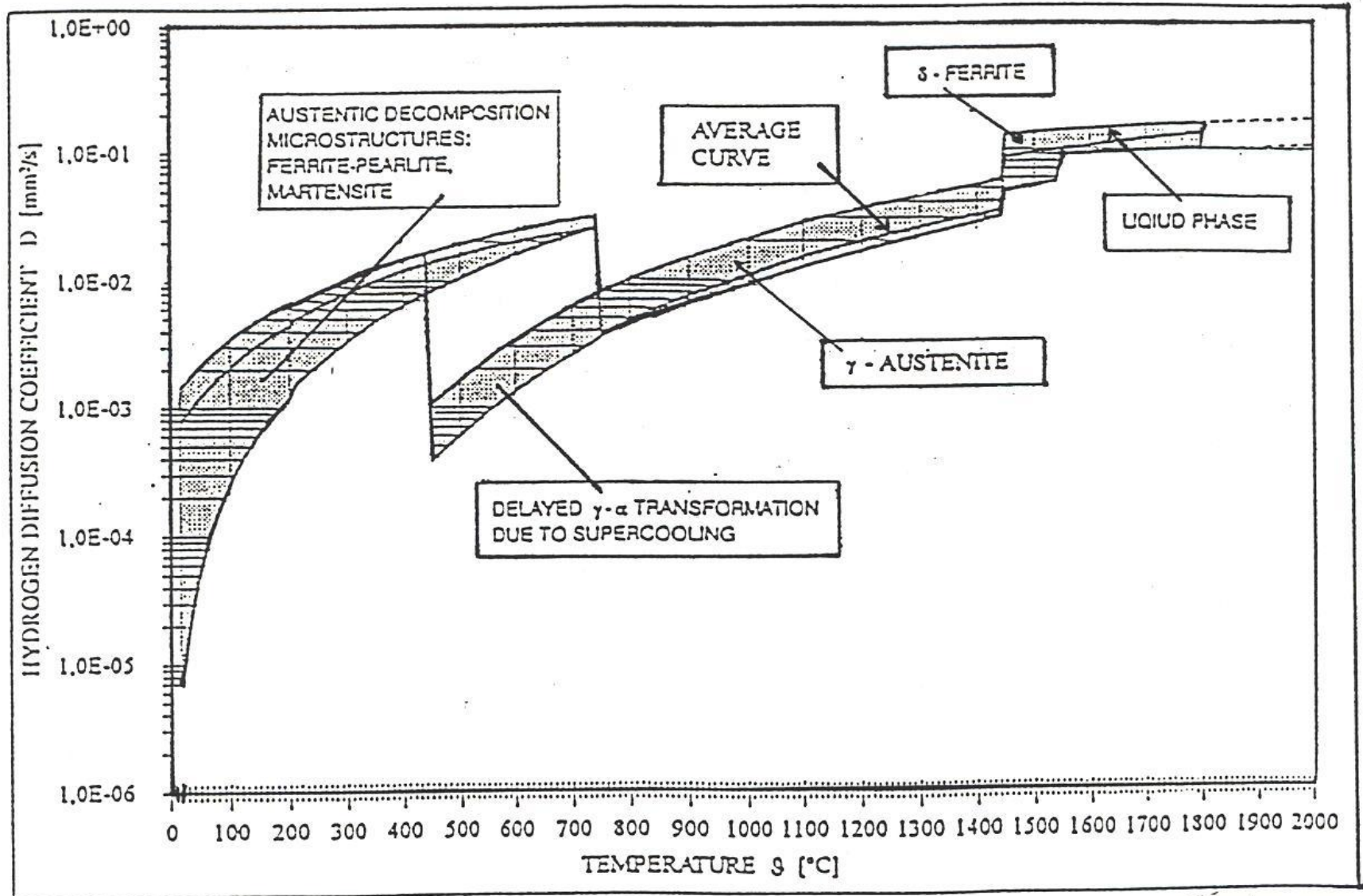


Fig. 13: Scatterband for hydrogen diffusion coefficients in micro alloyed and low carbon structural steels

Current Methods for Hydrogen Measurement

- Laser Ablation/Gas Chromatography
- Laser Ablation/Mass Spectrometer
- Electrochemical
- Opto-electronic diffusible hydrogen sensor
- AWS Volumetric Displacement