These PowerPoint slides accompany a lecture on the topics described in the paper:

Hubler, G.K., *Anomalous Effects in Hydrogen-Charged Palladium - A review*. Surf. Coatings Technol., 2007.

The paper is available here: <a href="http://dx.doi.org/10.1016/j.surfcoat.2006.03.062">http://dx.doi.org/10.1016/j.surfcoat.2006.03.062</a>

#### **Abstract**

There are more than 10 groups world wide that have reported the measurement of excess heat in 1/3 of their experiments in open and/or closed electrochemical cells with a Pd solid metal cathode and Deuterium containing electrolyte, or  $D_2$  gas loading of Pd powders (see Table 1). Most of these groups have occasionally experienced significant events lasting for time periods of hours to days with 50-200% excess heat measured as the ratio between electrical input energy and heat output energy. Moreover, these experimenters have improved their methods over time and it is to be noted that the reported excess heat effect has not diminished in frequency or magnitude. This paper cites selected data generated over the past 15 years to briefly summarize what has been reported about the production of excess heat in Pd cathodes charged with Deuterium. A set of new materials experiments is suggested that, if performed, may help to reveal the underlying mechanism(s) responsible for the reported excess heat.

#### The Status of Anomalous Effects in Hydrogen Loaded Palladium

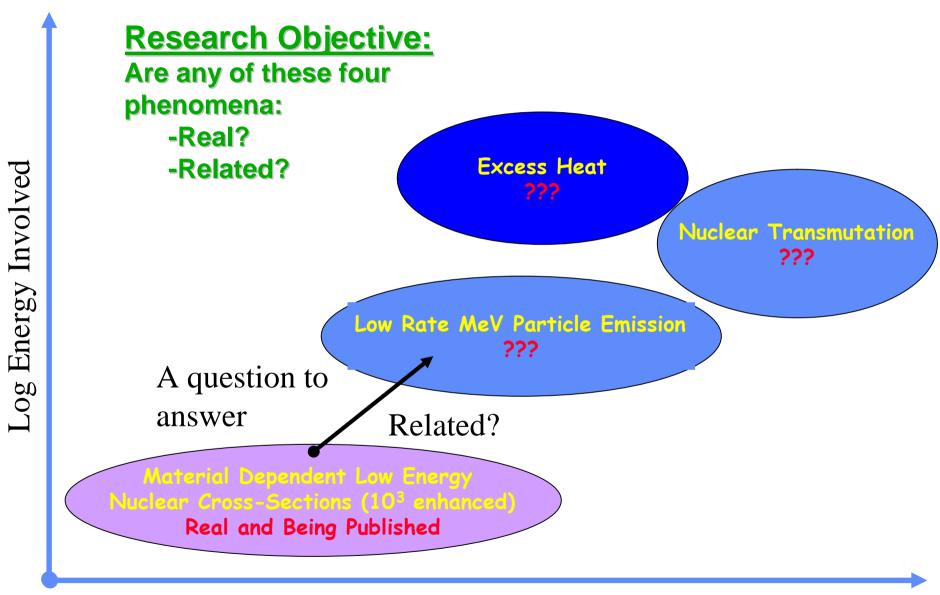


#### Graham Hubler

US Naval Research Laboratory Washington, DC 200375 USA

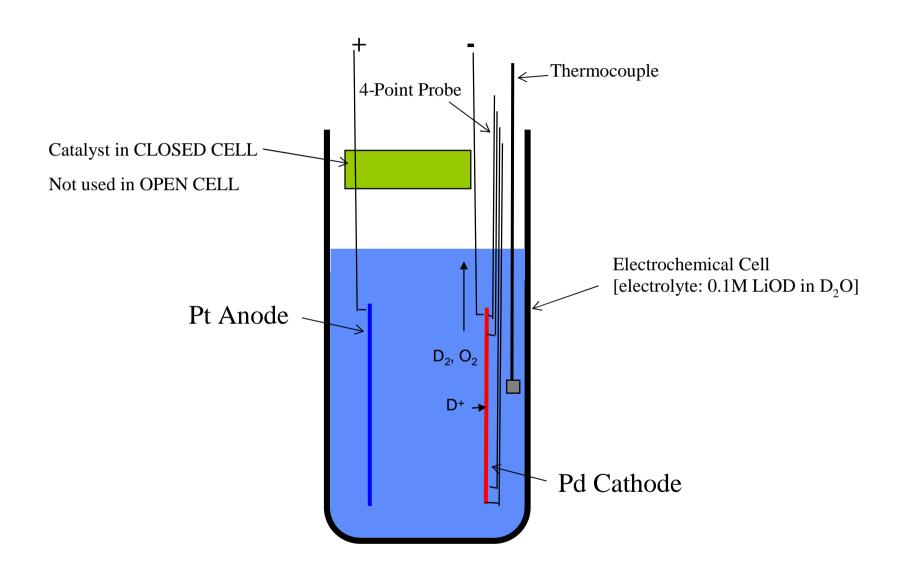
#### **Outline**

- 1-vu-graph summary of activity in anomalous effects in Pd-H, Pd-D materials systems
- History and facts of excess heat
- Suggested experiments
- Summary



Implausibility to Community

## **Schematic of Fleishmann-Pons Cell**



## Conditions developed over a period of 15 years

#### Facts:

>10 groups can produce ~20% excess heat ~30% of time in open and/or closed cells Many groups have occasional runs with 100-2500% excess heat

Conditions (necessary but not sufficient): High D Loading (x > 0.90;  $PdD_x$ ) High electrical current (~250 mA/cm²) Imposed D flux Dynamic trigger ( $\Delta T$ ,  $\Delta I$ ,  $\Delta D$  flow, laser)

## **Additional Emerging Systematic Trends**

Incubation time decreases as cathode volume decreases

Increased surface area increases heat production

Heat production may be a surface or near surface phenomenon

### **History - What Happened in 1989-1990?**

[after Fleishmann & Pons announcement]

Most researchers could not observe any excess heat so field was labeled as bad science and it went underground Why no heat?

- -Now known that Pd cracks under loading (3% vol. change), especially pure, annealed Pd, and surface area of cracks are recombination sites for hydrogen and deloading occurs
- -Pd must be strengthened and toughened by alloying or mechanical treatment
- -Must have the optimum grain size to load
- -Must be loaded gently (gradually or cycled in current) or will crack anyway (loading time >> diffusion time)
- -Dealing with catalytic surface is non-trivial

Most researchers never were able to achieve one of the necessary conditions to observe excess heat, that is, D loading of >0.90-0.95

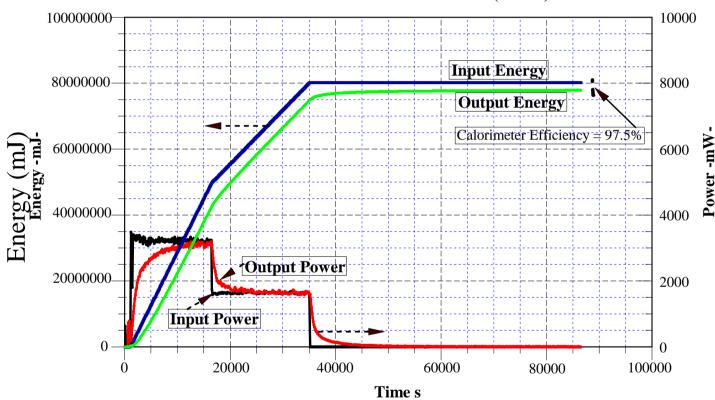
## **Selected List of Laboratories Producing Excess Heat**

Arata - University of Osaka, Japan
Violante - ENEA, Rome, Italy
Mizuno, Hokkaido University, Japan
Lesin, Energetic Technology, LLC, Israel
Dash - Portland State University, USA
Swartz - Jet thermal Products, Inc., USA
McKubre - SRI, USA
Storms - Lattice Energy, Inc., USA
LabA - Institutional censorship prevents disclosure
LabB - Company decision not to disclose

### Reference Experiments with Hydrogen at ENEA

V. Violante et al.

#### C ALIBRATION ENERGY (LiOH)



- •Input and output energy & input power plot for a calibration experiment with H<sub>2</sub>O 0.1 M LiOH.
- •Calorimeter Sensitivity =  $\pm$  40 mW
- •Calorimeter Accuracy = ± 25 mW

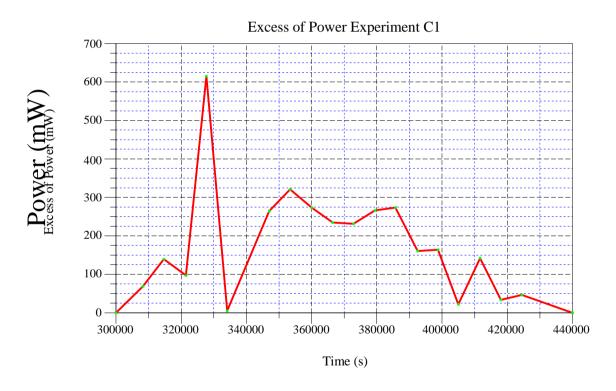
V. Violante et al.

**ENEA** 

Rome, Italy

# Excess of Power: Recent Experiments at ENEA (V. Violante, unpublished results)

#### **Experiment C1**

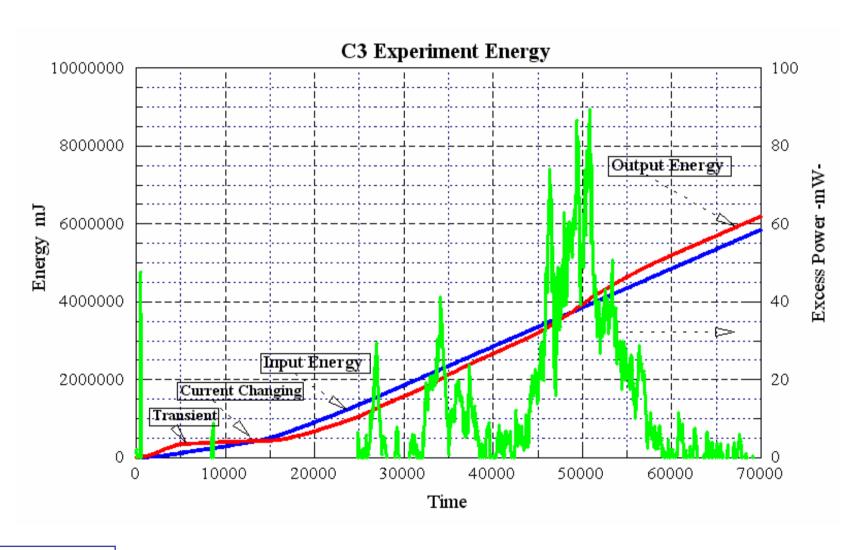


Experiment C1: excess of power vs time (45 KJ of produced energy)

V. Violante et al. ENEA

Rome, Italy

### Experiment C3

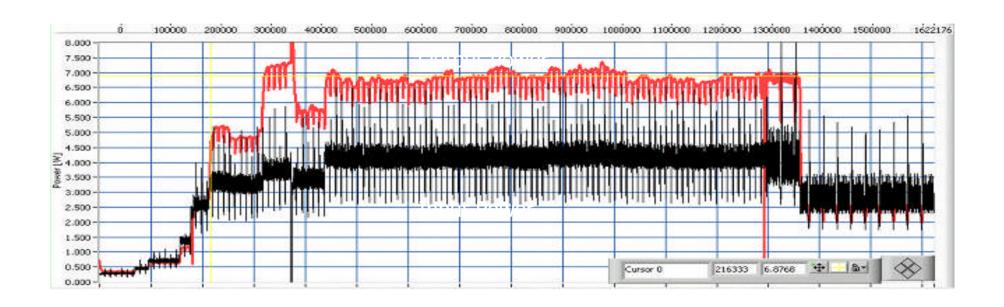


V. Violante et al. ENEA

Rome, Italy

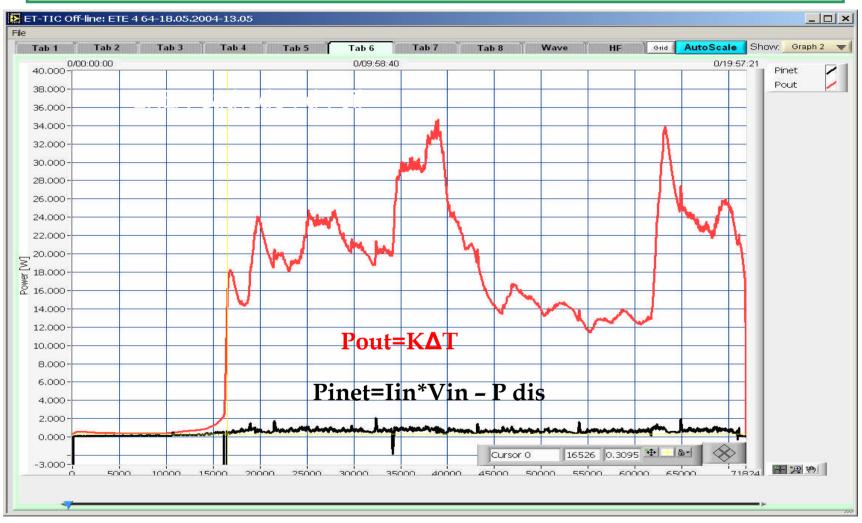
Excess of power and energy gain in C3 experiment.

# Energetics Technologies Ltd.



Tanya Zilov, Shaoul Lesin, Irving Dardik ET, Ltd. Omer, Israel Excess of Power at Energetics Ltd with ENEA Pd Foil [~2.25 Watts (+53%) for ~12 days, ~3 day incubation time]

# Energetics Technologies Ltd.



Tanya Zilov, Shaoul Lesin, Irving Dardik ET, Ltd. Omer, Israel Excess Power production from cell ETE-4-64 during 16 h (ENEA Pd foil)

## Primary Criticisms of Excess Heat Results

- -Energy stored by some as yet unknown but straightforward mechanism during long incubation times and then released (battery)
- -Excess heat due to recombination of oxygen and hydrogen in cell (battery)
- -Calorimeter is not calibrated correctly (experimental error)
- -Energy inventory not measured correctly (experimental error)

Data on previous slides appear to refute these criticisms

#### Is There a Sensible Pathway to Solve this Interesting Puzzle?

## Needed:

- -Some materials science to get to:
  - Increase in repeatability (main reason this work is not published)
  - Increase in power production
  - Theory

New, more innovative experiments might help

#### **What Types of Experiments?**

- EX SITU Experiment
- -Detailed characterization of cathodes before and after excess heat is produced
- <u>In situ</u> experiments are to be considered since electrochemical loading is the primary means to obtain this material in the highly loaded state

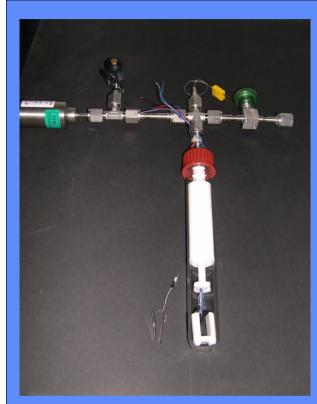
#### IN SITU Experiment Requirements

- Probes that can penetrate glass cell walls, the  $D_2\mathrm{O}$  in cell the and get to  $PdD_x$  target
- Signal carriers that can emerge from the  $PdD_x$  target, the  $D_2O$  and cell walls and be detected
- This in situ requirement leaves us with the choices of:
  - -x-rays
  - -gamma rays
  - -neutrons
  - -certain lightwaves
  - -sensors in proximity to cathode

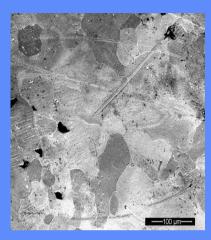
#### Some Experiments Suggested by In-Situ Requirement

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-In situ tensile stress
-In situ high energy x-ray scattering
    (Pd structure)
-In-situ inelastic neutron scattering
    (phonons, Pd, D structure)
-In situ radioactive isotope spectroscopy
    (nuclear environment)
-In situ Mössbauer spectroscopy
    (hyperfine field characterization)
-In situ Perturbed Angular Correlations (PAC)
    (hyperfine magnetic, electric fields)
-In situ Nuclear Acoustic Resonance (NAR)
    (phonon-lattice coupling)
```

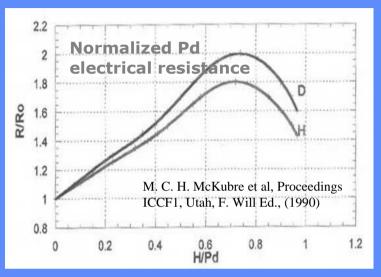
#### A Platform for Reproducible Experiments has Emerged



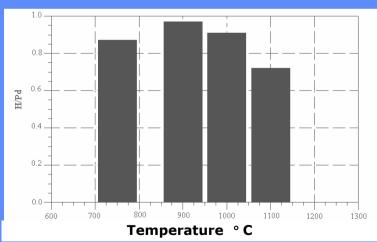
**ENEA Cell** 



Cold worked and annealed at 850 °C for 1 hr.



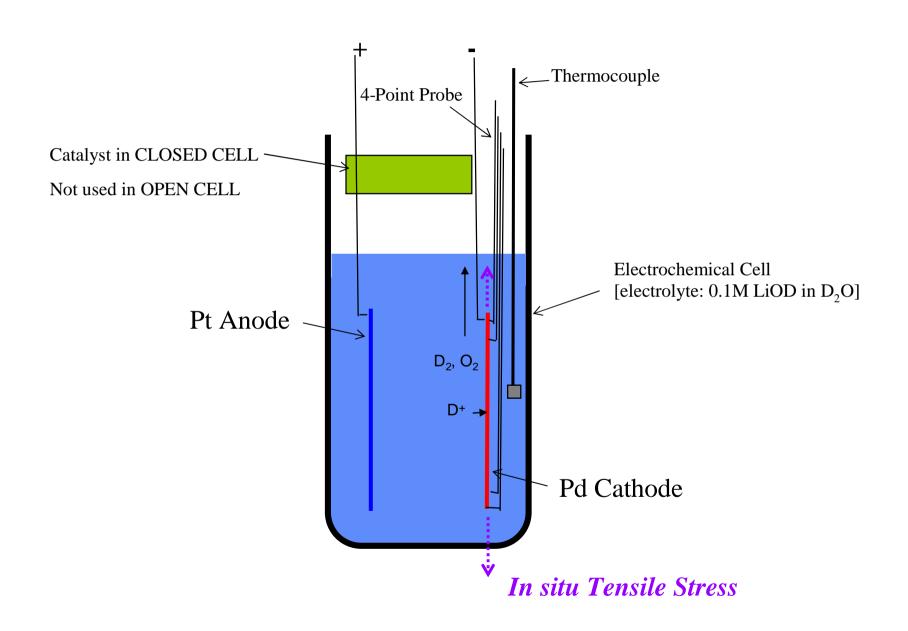
4-point probe Pd electrical resistivity with loading.



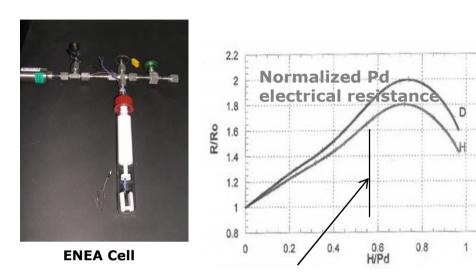
Annealing temperature effect on H loading in Pd

Work of Vittorio Violante, ENEA, Frascati, Italy

## **Schematic of Fleishmann-Pons Cell**



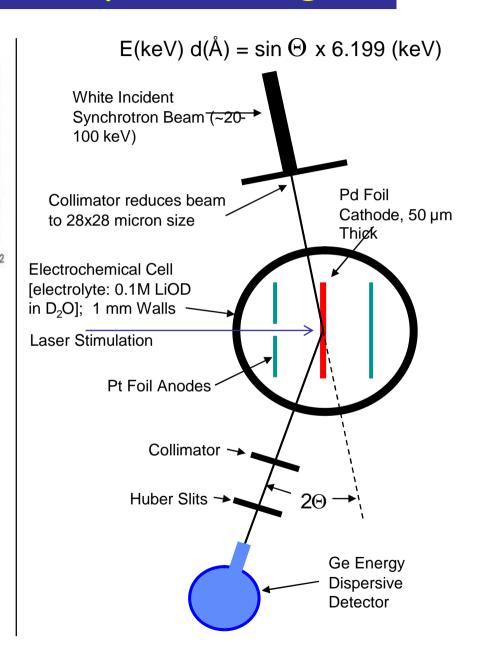
## In situ high energy x-ray scattering



Existing data only extends to H/Pd = 0.55

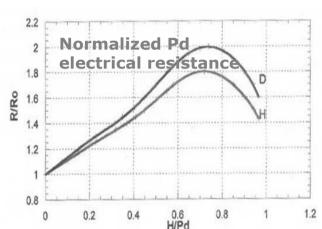
Goals: versus D loading,

- Characterize Pd sublattice structure of this highly unique material
- Stimulate with HeNe laser impingement to possibly stimulate excess heat condition



## In situ neutron scattering

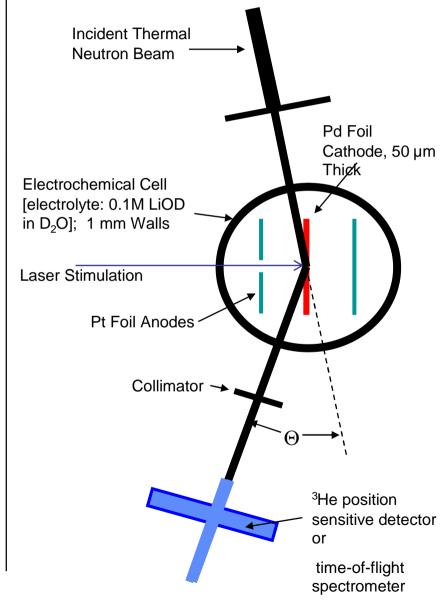




\*Neutron moderation - Neutron scattering length is 9X larger in D<sub>2</sub>O than in H<sub>2</sub>O

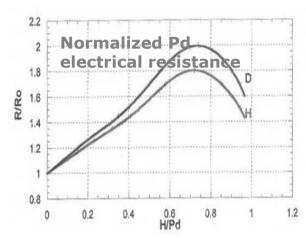
Goals: versus D loading,

- Characterize Pd and D sublattice structures of this highly unique material
- Characterize phonons in Pd, D sublattices
- Stimulate with HeNe laser impingement to possibly stimulate excess heat condition

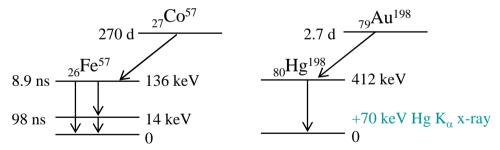


## In situ radioactive isotope spectroscopy



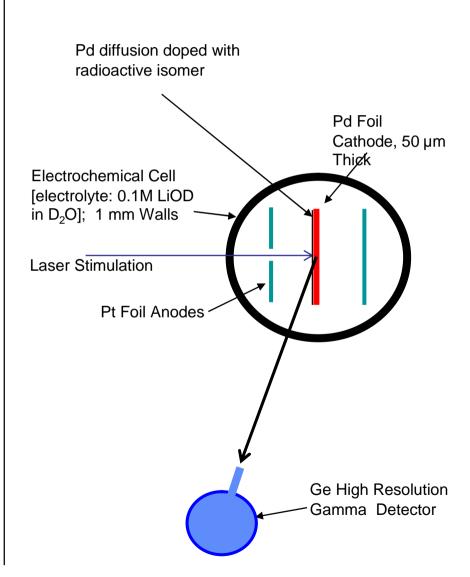


<u>Isotopes:</u> Co (Electron Capture)& Au (Beta Decay) are soluble in Pd



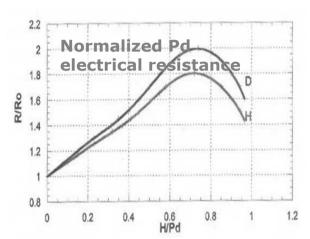
Goals: versus D loading & laser stimulation,

- Provide simple, gross experiment that might uncover influence of chemistry or lattice effects on:
  - Nuclear decay rate
  - Energy of emitted gamma rays
  - Energy of emitted x-rays

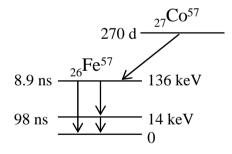


## In situ Mössbauer spectroscopy



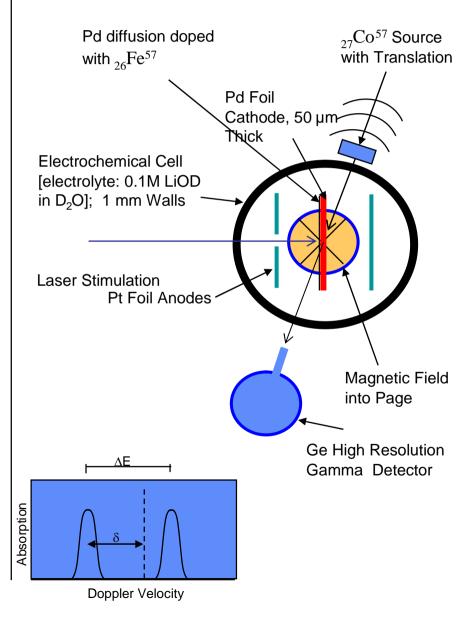


**Isotope:** 



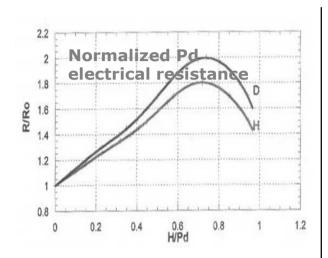
Goals: versus D loading & laser stimulation,

- Survey effect of H environment on magnetic and/or electric quadrupole hyperfine fields caused by distortion of electron cloud in ns time resolution
- Isomer shift  $\delta$  s-electron distortion
- Magnitude of electric and/or magnetic field at Fe nucleus (ΔΕ)

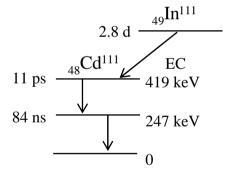


## In situ Perturbed Angular Correlations (PAC)



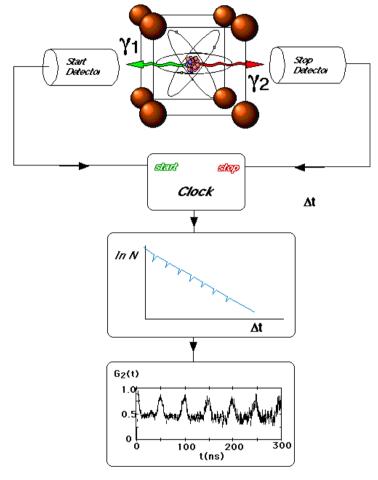


Isotope:

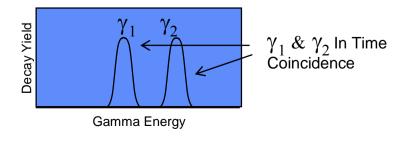


Goals: versus D loading & laser stimulation,

- Get lattice site information on Cd impurity
- Measure Electric field at Cd site

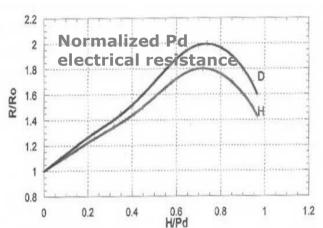


Univ. Wash, Web site



## In situ Nuclear Acoustic Resonance (NAR)



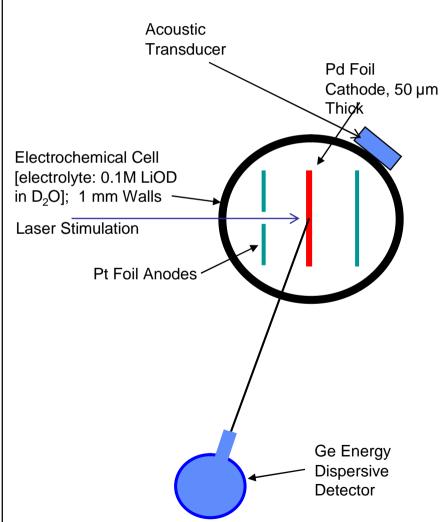


#### **Experiment:**

Vary acoustic frequency to natural vibration frequencies of Pd foil and internal friction defect resonances

Goals: versus D loading & laser stimulation,

- Measure any of the previous 5
   experimental parameters in and out of
   acoustic resonance
- Assess interplay of acoustic mode phonons and nuclear alignment/ environment



## **Summary**

•These suggested experiments can not be conducted by individuals acting alone or in their garage or basement

- These experiments require:
  - · Sophisticated experimental infrastructure
  - Interested participants acting as a team
  - Research funding

•There is a severe mismatch between the researchers wanting to perform experiments, infrastructure and expertise, and funding availability

## **Summary**

- Some anomalous effects in the materials system of highly hydrogen-loaded Pd were highlighted
- Based upon a new reproducible Pd-H materials system, a case was made that there is a new opportunity for exploration of this material with a variety of sophisticated materials science techniques
- There are currently few options for carrying out such experiments. The most critical need is genuine interest in the anomalous effects by the appropriate scientific community coupled to an appropriate level of support