

Emergence of a High Temperature Superconductivity in Hydrogen Cycled Pd compounds as an Evidence of a Superstoichiometric H/D Sites

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Introduction

- Dislocations in H-cycled Pd can absorb large amount of hydrogen (5-6 at. H/1Å of dislocation line)

B.J. Heuser and S.J. King Met. & Material Transaction A29, 1594 (1998)

Under electrochemical cathode-anodic and/or H₂ gas cycling the dislocation density in Pd foils reaches $(2-5) \times 10^{11} \text{ cm}^{-2}$.

Hydrogen trapped inside deep dislocation cores may show specific behavior.

Binding energy of this H should exceed 0.7 eV/H-atom.

Dislocations in the H-cycled Pd single crystal (x 80K)





What is expected ?

If hydrogen trapped inside deep dislocation cores:

- Very high loading ratio $x=H/Pd \geq 3$ or metallic H_n , $(H_2)_n$,
- Compression $P \sim 120$ GPa,
- High optic phonon frequency ($h\omega \sim 120$ meV).
- Strong electron-phonon coupling ($\lambda_{e-ph} > 1.0$).
- Strong Pd-H(D) band overlapping (N. Ashcroft, PRL, 92, 187002, (2004)).
- Excellent Conditions to achieve High Temperature Superconductivity ?
- Triggering of LENR.



Objectives

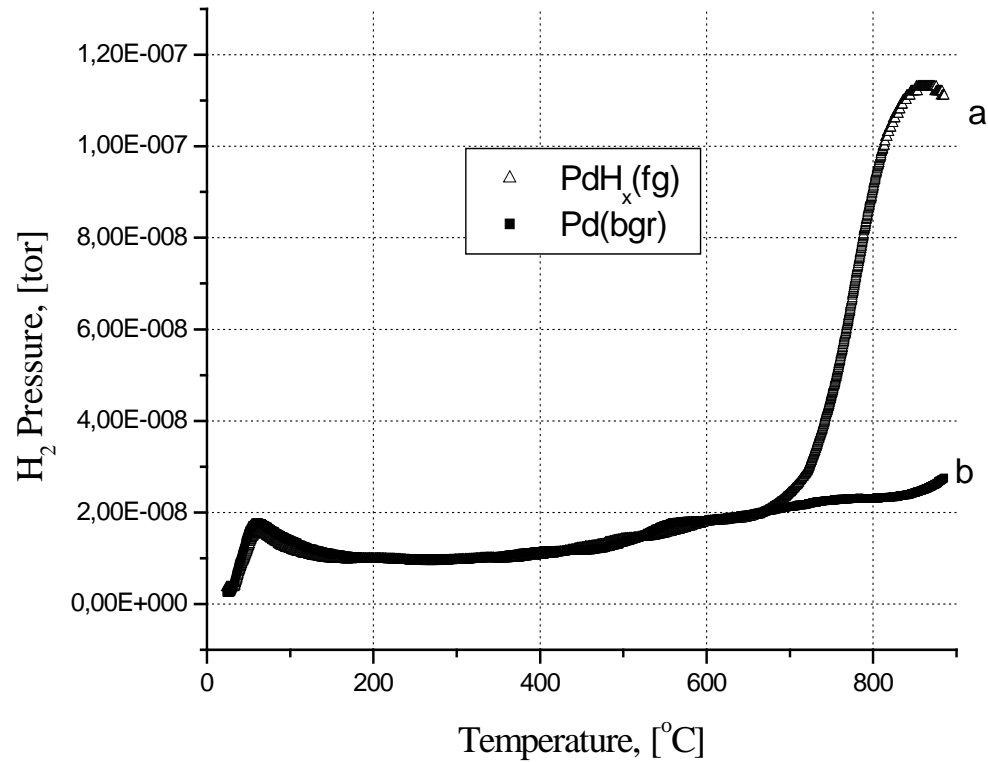
- Study of hydrogen localization inside dislocation cores in Pd and Pd/PdO by vacuum thermal desorption technique.
- Study magnetic and transport properties of condensed hydrogen phase inside dislocation nanotubes in Pd/PdO foils and Pd single crystal (1.8-300K).
- Define a role of these superstoichiometric D-sites in LENR to trigger various DD-reactions, including multibody type (3D).



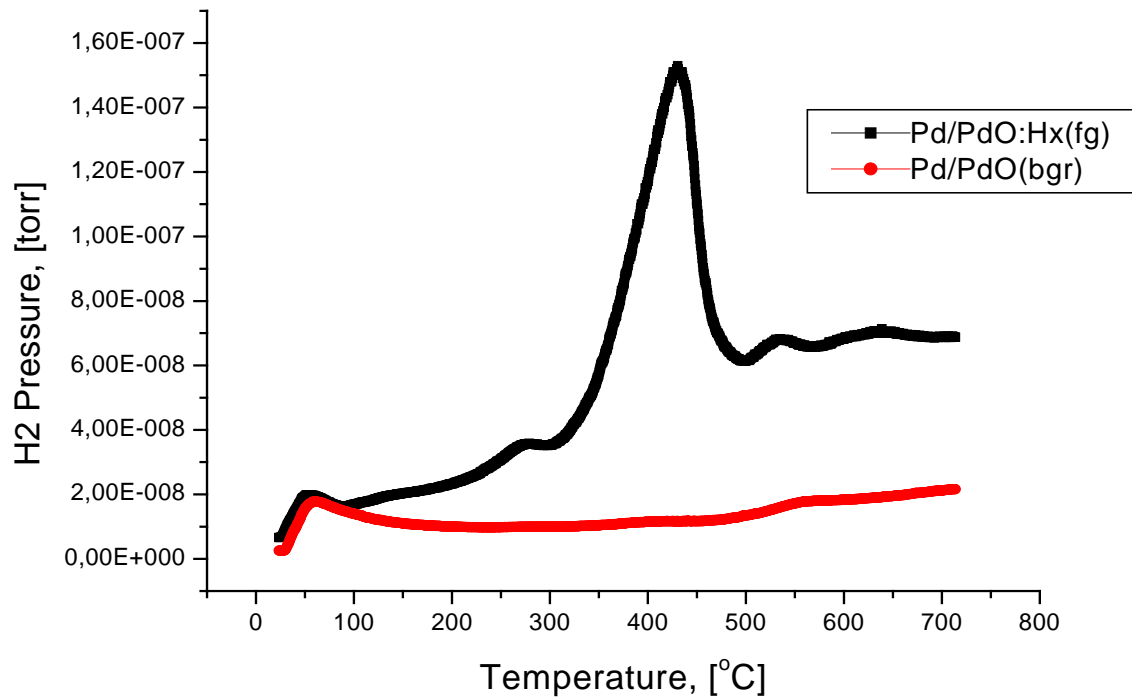
Sample Preparation

- Pd single crystal ingot (99.999%); samples $m=62$ mg, H₂ gas loading-unloading cycles and annealing at $T=573$ K for 2 hr.
- Pd/PdO cold worked heterostructure. $h=12.5$ μm , (PdO ~ 40 nm), 99.95 %, Nilaco Co., Japan, Fe 10 ppm. Electrochemical cycling (cathode loading-anodic unloading); $j=5.0$ mA/cm², 1M Li₂SO₄/H₂O, annealing at $T=573$ K.
- Measurements: TDA with mass-spectrometer; 1T-SQUID "Quantum Design" DC and AC modes, $M(T)$, $M(H)$, $X'(T)$; 4 and 2 -probe resistance: $R(T)$, $R(I)$

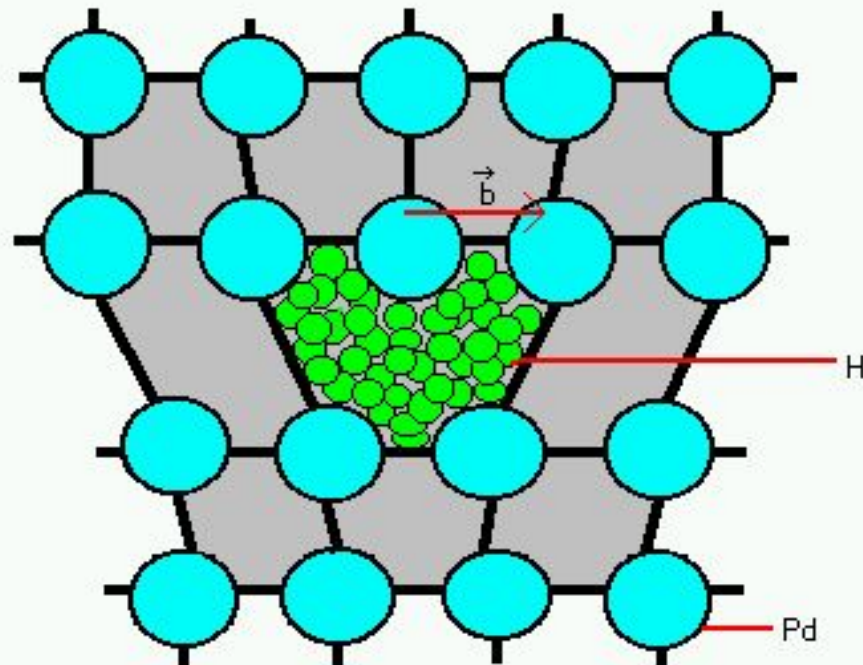
Thermal desorption of Hydrogen from Pd:Hx
($x=H/Pd=3.8 \times 10^{-4}$). $T_{\max}=870 \text{ }^{\circ}\text{C}$, $\varepsilon_H = 1.6 \pm 0.2 \text{ eV}$



H-Thermal Desorption from Pd/PdO:Hx ($x \sim 5.5 \times 10^{-4}$), $T_m = 440 \text{ }^\circ\text{C}$, $\varepsilon_H = 0.65 \pm 0.10 \text{ eV}$

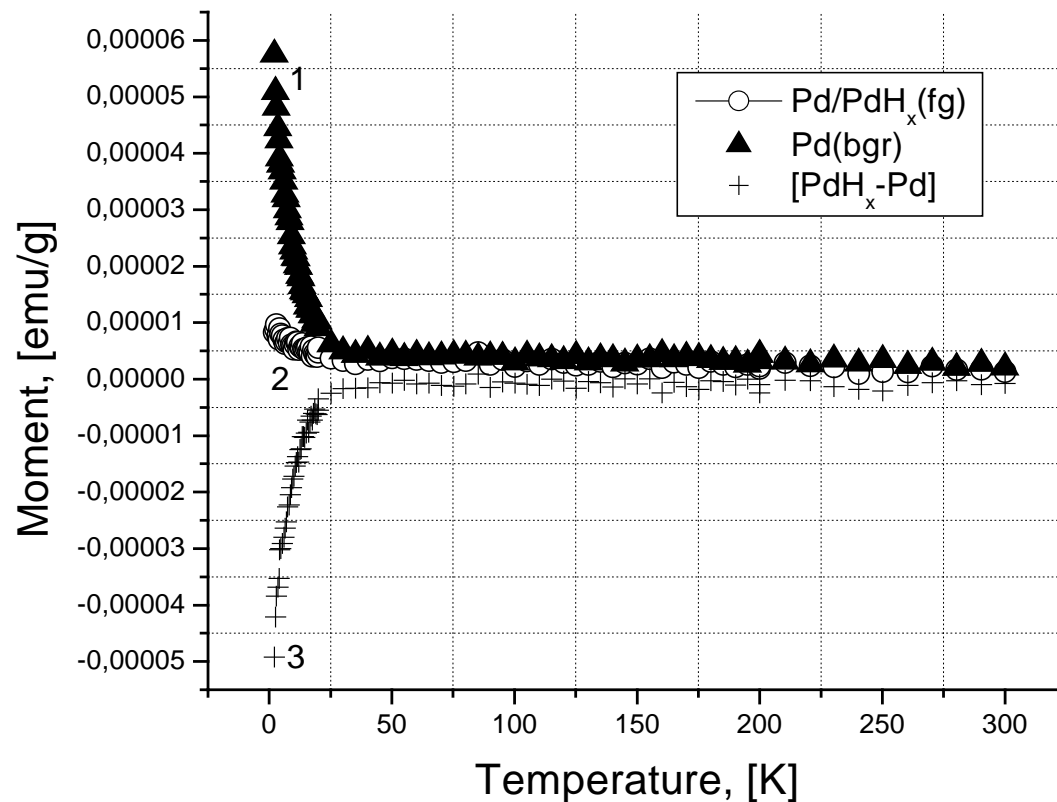


Edge dislocation core in Pd with H_n-condensed hydrogen phase

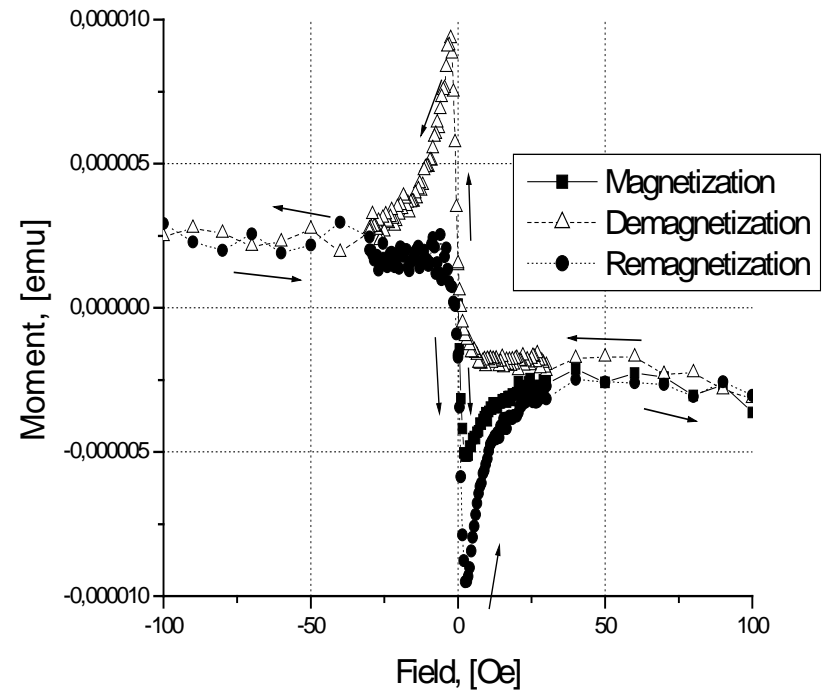
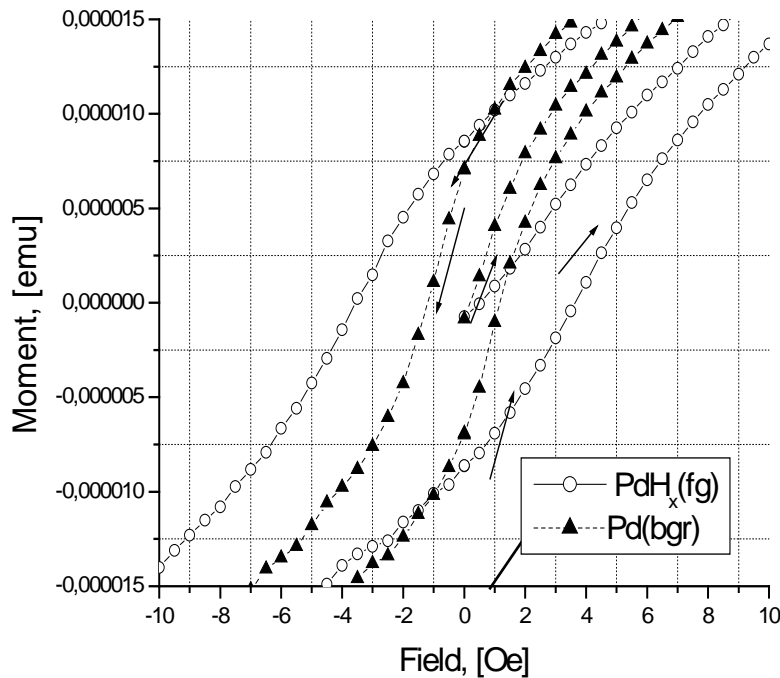


Pd ($\bar{1}21$)
→
 $b[\bar{1}01] = 2.75 \text{ \AA}$

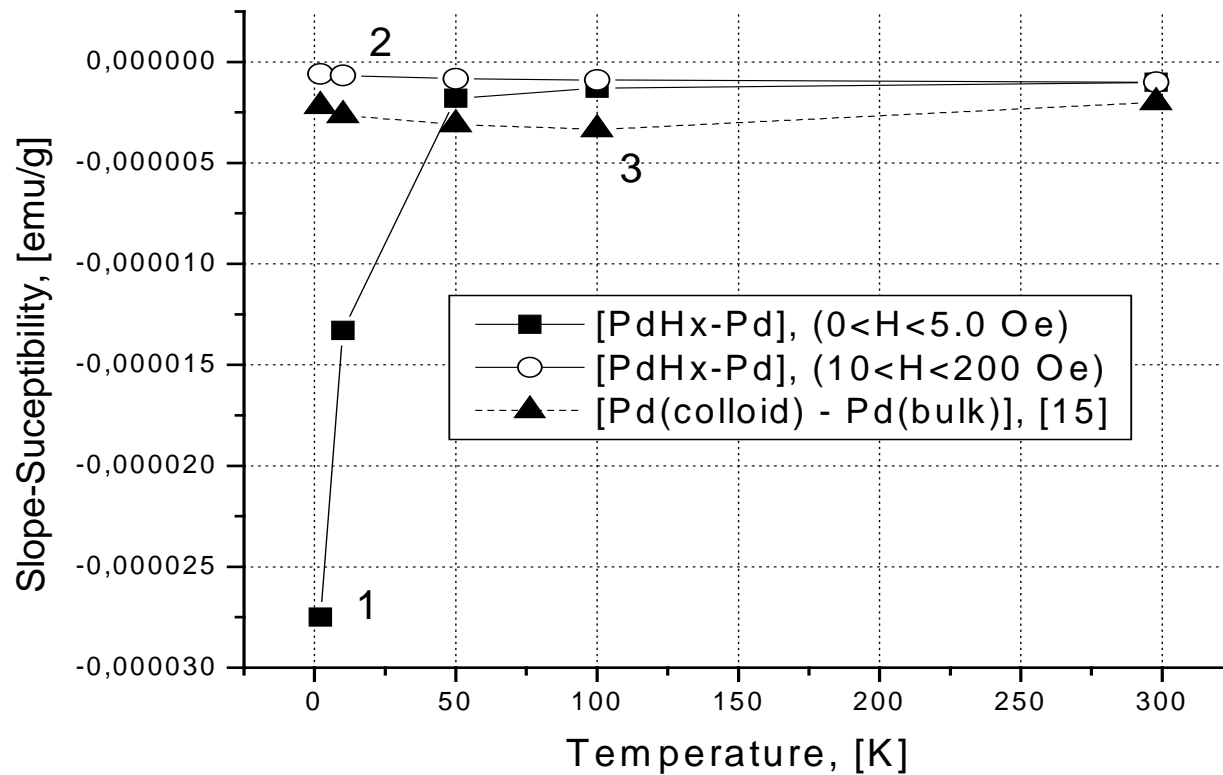
DC Magnetic SQUID measurement with PdH_x and Pd single crystal: M(T), H=1.0 Oe



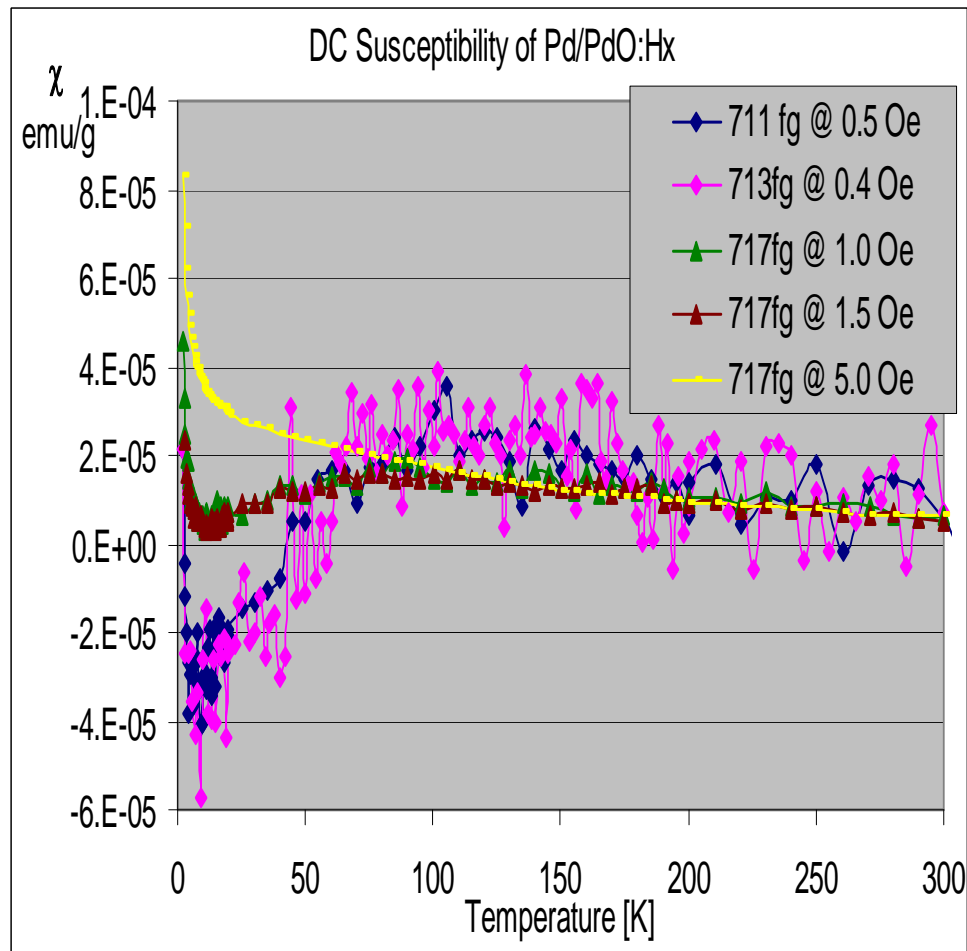
PdH_x, Pd single crystal and [PdHx-Pd] phase: M(H), T=2.0 K



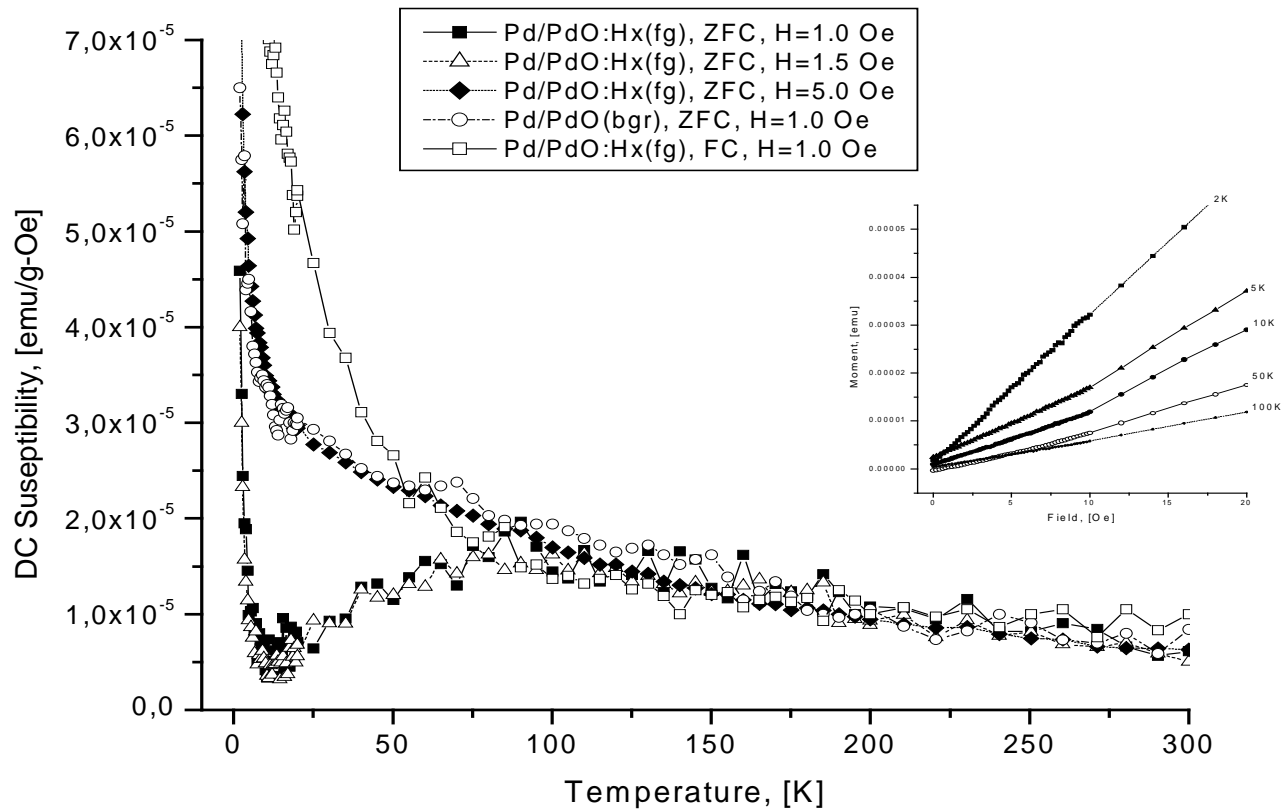
DC $\chi(T)$ for [Pd:Hx-Pd] phase



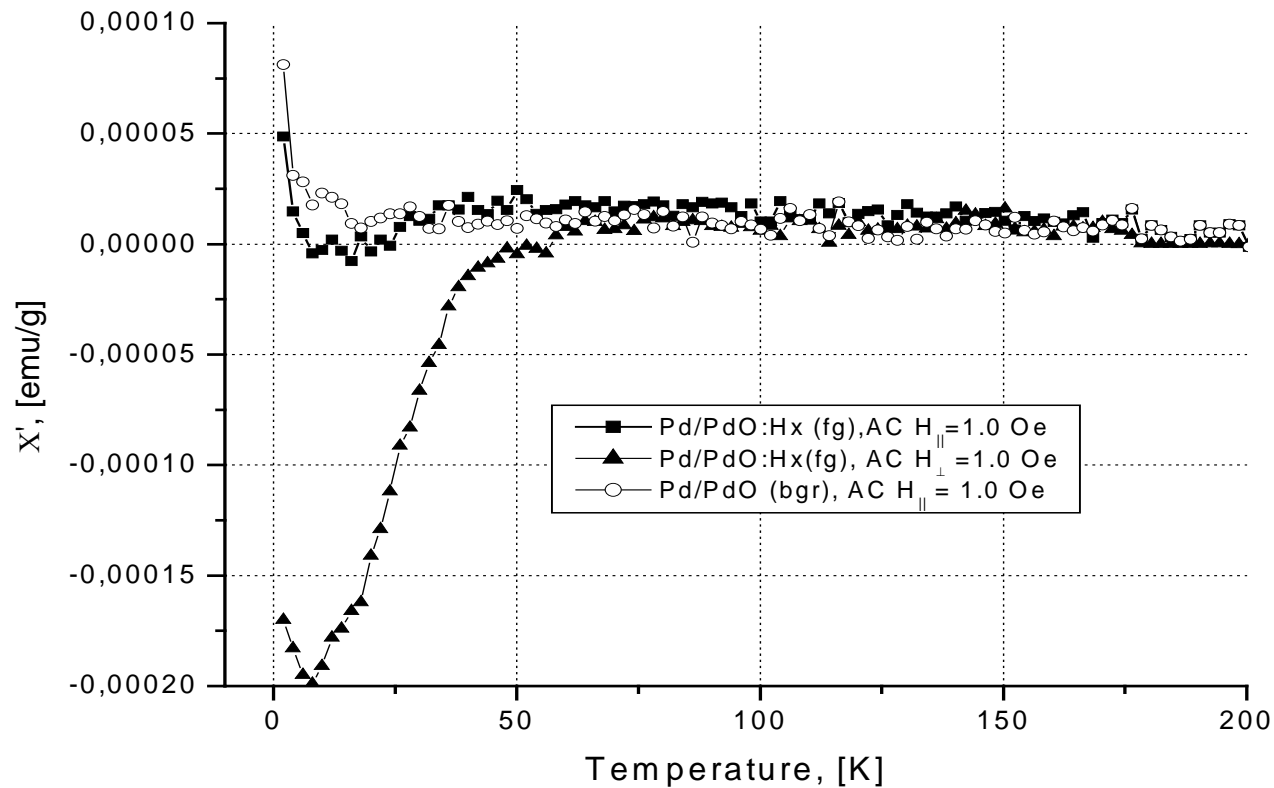
Low magnetic field DC-susceptibility for Pd/PdO:H_x sample



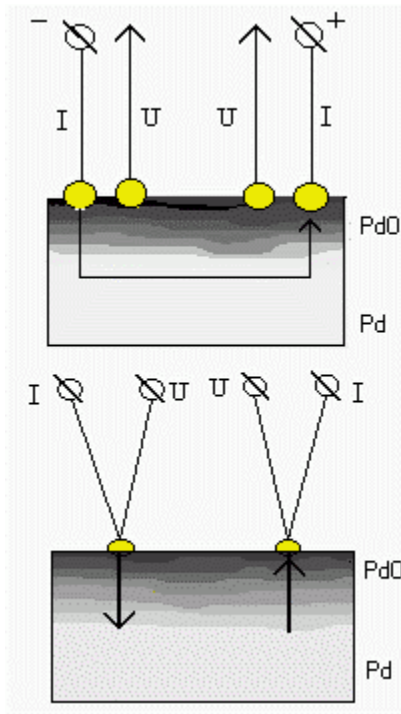
Pd/PdO:Hx and Pd/PdO blank: M(T) and M(H)



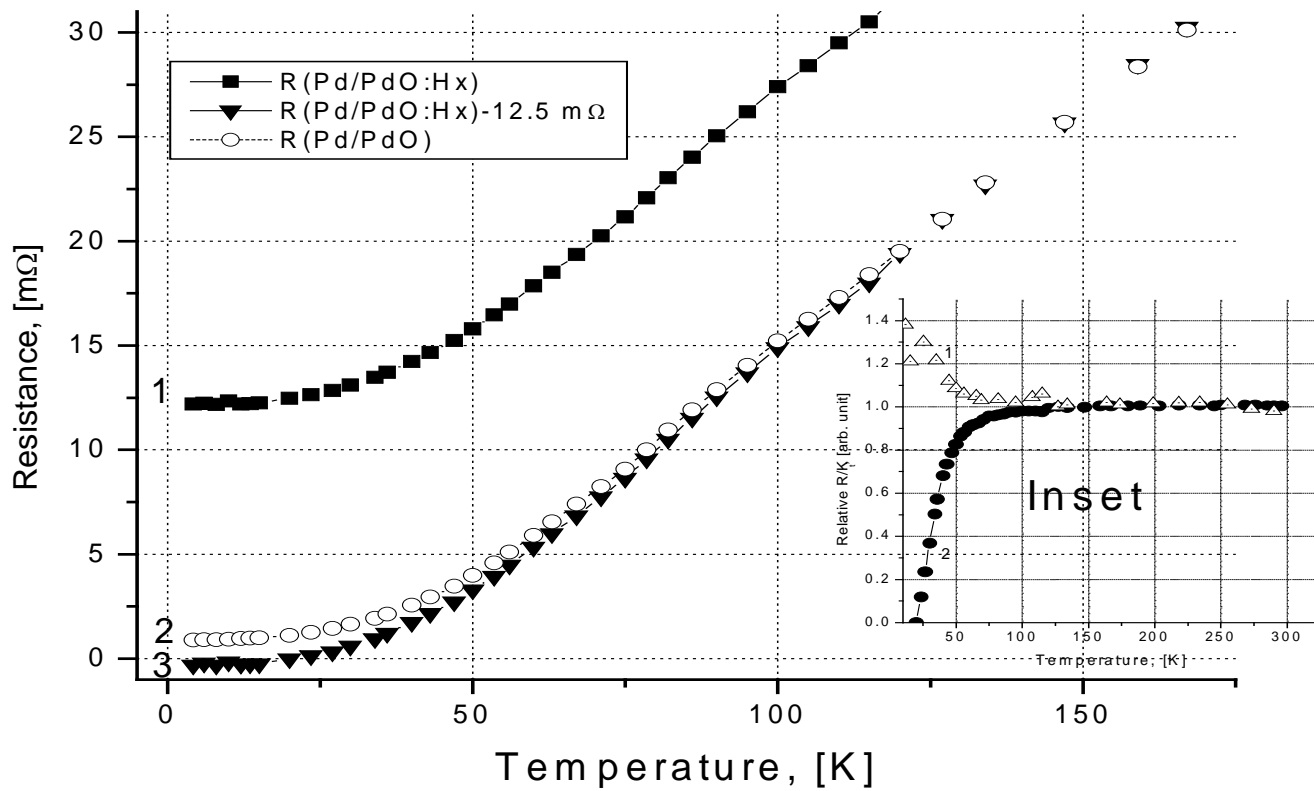
X' AC susceptibility of Pd/PdO:Hx and Pd/PdO-blank samples vs. T for H_{\perp} and H_{\parallel} with respect to the sample



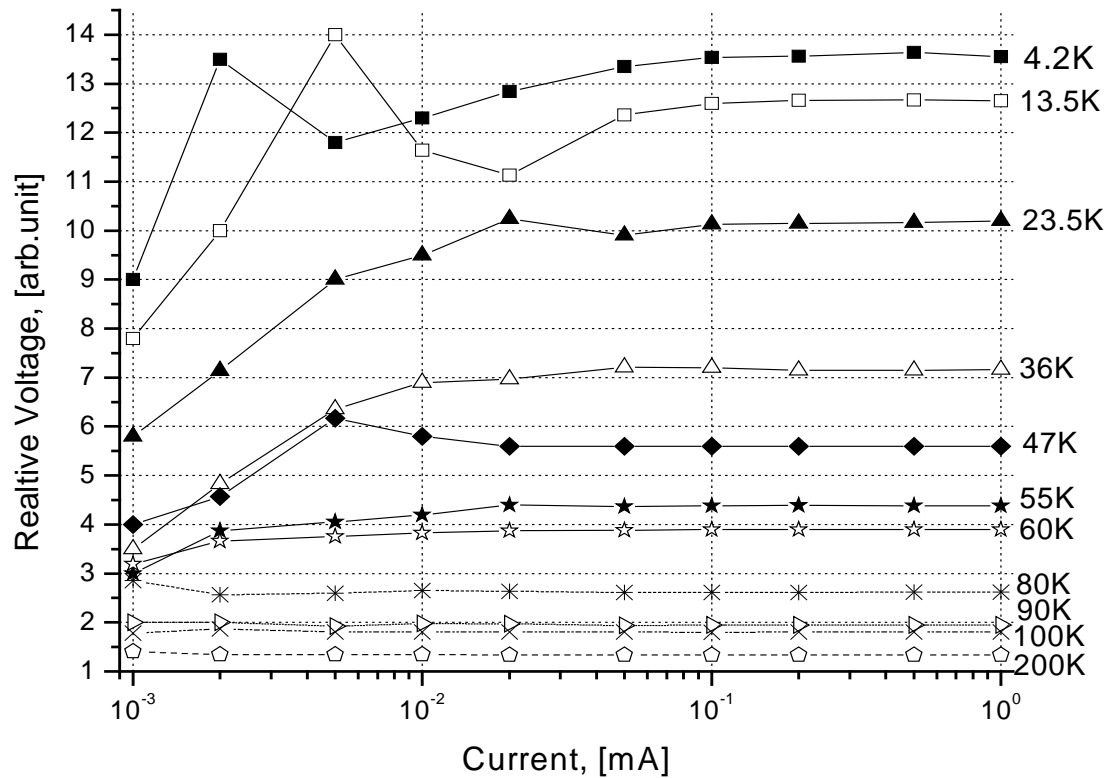
Diagrams of 4-probe and 2-probe(pseudo-four probe) resistance measurements



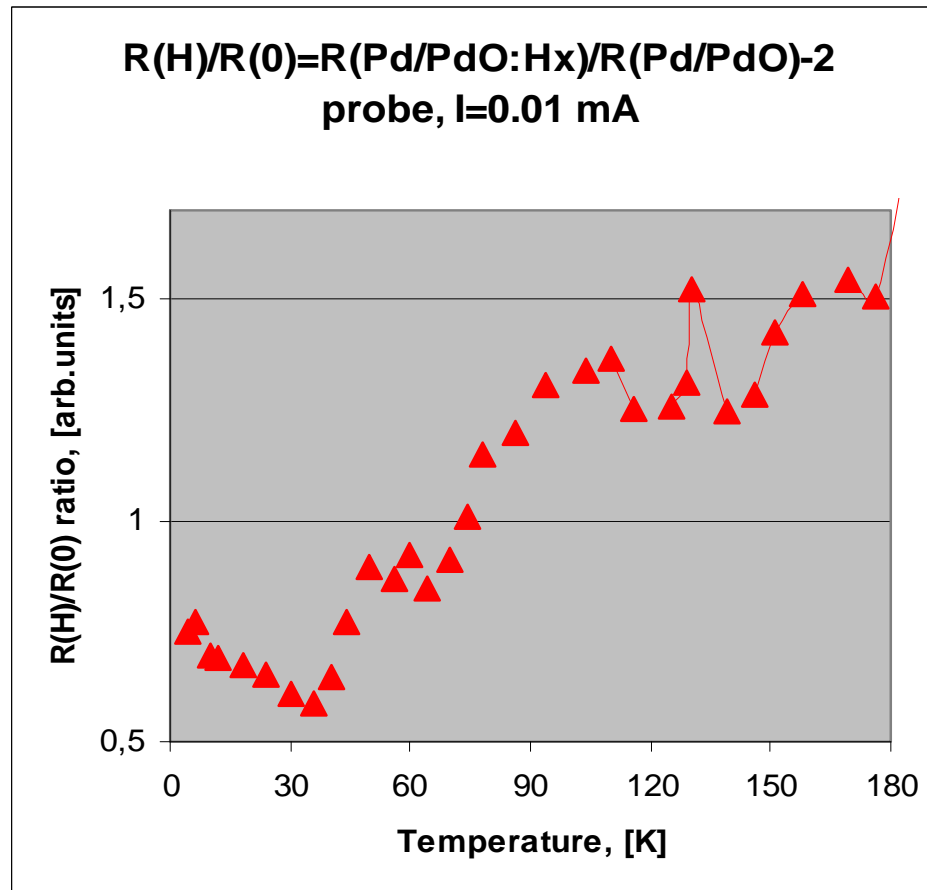
$R(T)$ for Pd/PdO:H_x and blank Pd/PdO. Inset: - Relative resistance (black circles) $R = R(\text{Pd/PdO:H}_x - R_i) / R(\text{Pd/PdO})$ and temperature coefficient of resistivity (triangles) $K = K(\text{Pd/PdO:H}_x) / K(\text{Pd/PdO})$



Relative I-V characteristics for (Pd/PdOH_x)/(Pd/PdO) ratio vs. T. Resistance decrease with decrease in I below 60 K.



2-probe resistance measurement: Relative resistance of Pd-PdO:H_x boundary $[R(\text{Pd}/\text{PdO:H}_x)/R(\text{Pd}/\text{PdO})]$ vs. T. $R(\text{Pd}/\text{PdO:H}_x) < R(\text{Pd}/\text{PdO})$ at $T < 75$ K





Summary of results

- Pd:H_x and Pd/PdO:H_x samples after H-cycling and annealing at T=573 K contain only condensed hydrogen phase inside deep dislocation cores: $x=H/Pd = (3.8-5.5) \times 10^{-4}$ with respect to the sample. Inside dislocation nanotube $x=H/Pd \sim 3.0$.
- Accordingly to SQUID measurements the H₂-cycled PdH_x single crystal sample demonstrates signature of a weak type II superconductivity, involving condensed hydrogen phase in deep dislocation cores [PdH_x-Pd] below 30 K.
- Results of both magnetic and transport measurements in Pd/PdO:H_x are suggested superconducting transition below 70 K. Reproducible Meissner-effect was obtained at $H \leq 1.0$ Oe in AC field ($f = 1$ kHz).



Conclusions and LENR connection

- Deep dislocation cores in Pd could be considered as a H-dominant Pd hydride ($x = \text{H/Pd} \geq 3.0$) sites suggesting HTS properties.
- Triggering of LENR at such sites will be appreciated due to:
 - - shortest DD-distance (close to Bohr radius)
 - - highest D-loading and lattice compression
 - - effective electron screening
 - - large optic phonon energy ($\hbar\omega_D \geq 120$ meV) resulting in a most effective lattice-nuclei energy transfer.