Surface Morphology of Metal Deuterides Upon e-Beam Excitation of Their Deuterium Subsystem
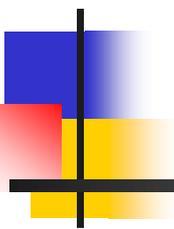
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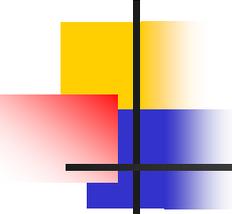
Introduction

- Our last reported experiments on electron-beam bombardment of metal deuterides with high affinity to deuterium (Pd/PdO:Dx and TiDx) showed reproducible charged particle emissions, including 3 MeV protons from DD-reaction and energetic alpha particles (with energy in the range of 11-20 MeV)

- To explain obseved effects we suggested that the deuteron acceleration mechanism should be involved, which alongside with a possible large electron screening (occurring in the metal targets with enhanced hydrogen diffusivity) could potentially strongly enhance the yield of DD-reaction in metal deuterides, even at extremely low energy of their excitation.

- To support this suggested mechanism we performed study of the surface morphology changes prior and after e-beam bombardment in the Pd/PdO:Dx and TiDx targets.

Objectives

- To verify the hypothesis on the role of electron excitation of the hydrogen subsystem in metal deuterides to enhance DD- yield.

- To search for changes in oxide layers and deuterium distributions in the targets under e-beam irradiation.

- To study morphologycal changes at the surface of the targets, involving creation of nanochannels and microcraters that could witness release of high energy via some specific spots at the surface.

Surface Analysis Techniques

- Rutherford Back Scattering/Elastic Recoil Detection (alphas E_α = 2.2 MeV, depth 2 µm)

- SEM

- STM

- Deuterium Thermal Desorption in Vacuum (t = 400 C)

Pd/PdO electrolysis in 0.3 M LiOD/D2O at t = 4°C (the cell with separated cathode and anodic spaces is cooled with LN$_2$)
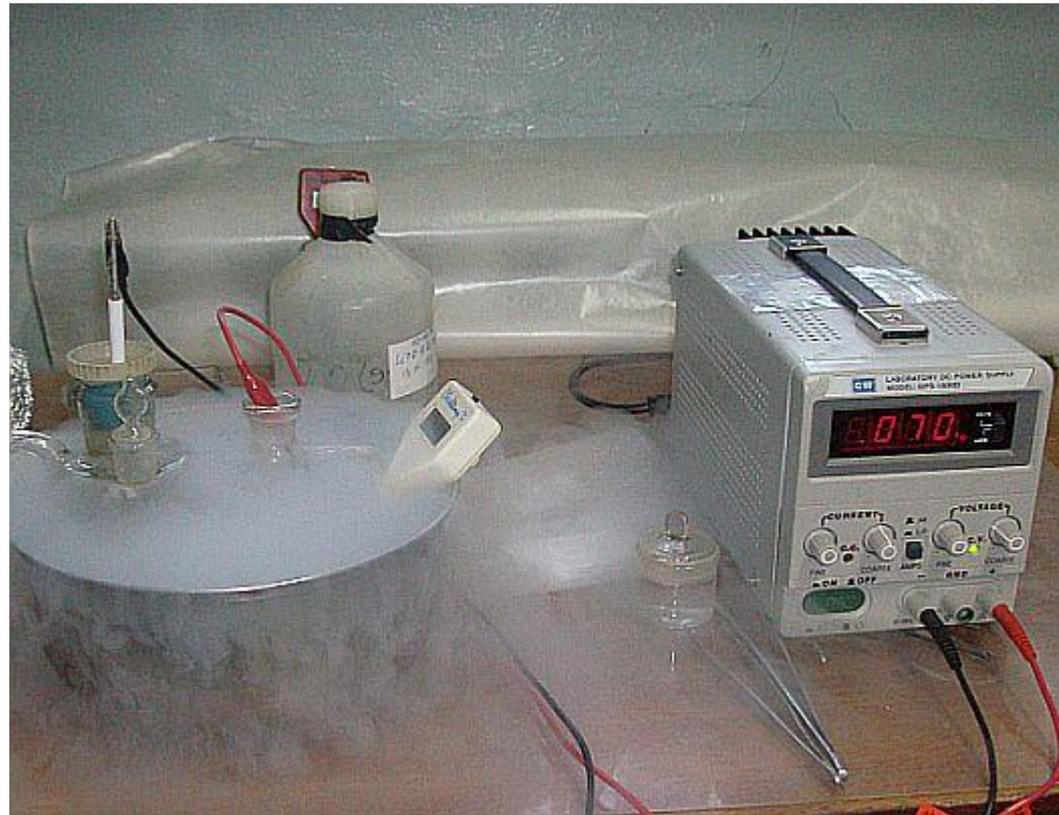
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Pd/PdO:Dx Sample preparation

- The PdO/Pd/PdO samples have been prepared by thermal oxidation from Nilaco (Japan) Pd foil (99.95 % purity) of 50 µm thick with dimensions S = 30 x 10 mm2.

- Electrochemical loading in 0.3M-LiOD solution in D2O with Pt anode; j = 10 mA/cm2 T ~ 280 K (below room temperature) in special electrolytic cell with splitted cathode and anodic spaces. x =D/Pd ~ 0.73 (about 40 min required).

- The samples have been rinsed in pure D_2O and then were put in the Dewar glass to cool them down to T =77 K.

- The cooled samples then were rapidly mounted (during 1 min) in sample holder in front of CR-39 detectors set and irradiated by e-beam (E = 30 keV, J=0.2-0.6 µA/cm2).

- In reference experiments without e-beam stimulation two CR-39 detectors (open and covered with 25 µm Cu) were used.

RBS/ERD profiles of the Pd/PdO samples prior to D-loading –left; after electrochemical loading and D-desorption in vacuum during 50 min with e –beam (J=0.6 μA/cm2 U = 30 kV) - right

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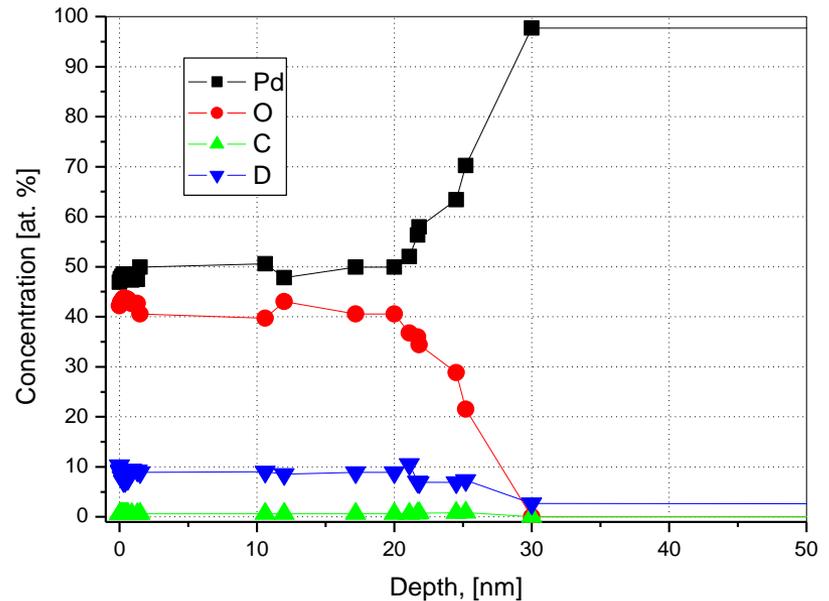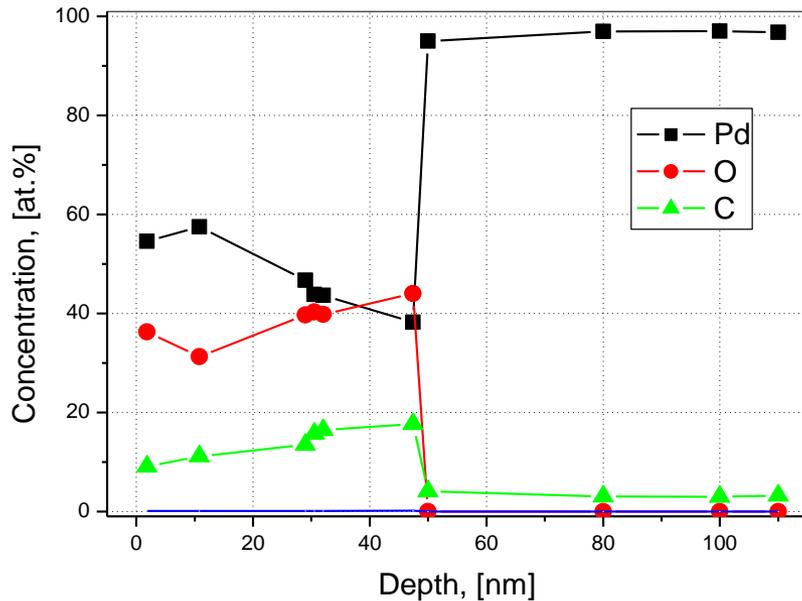
D-desorption from the Pd/PdODx sample in vacuum without e-beam excitation and in air. Under e-beam the desorption rate is J_D ~ 3.3 x 10^{15} D/s-cm^2. This number of desorbed deuterons is

consistent with the rate of D-desorption in air at ambient conditions
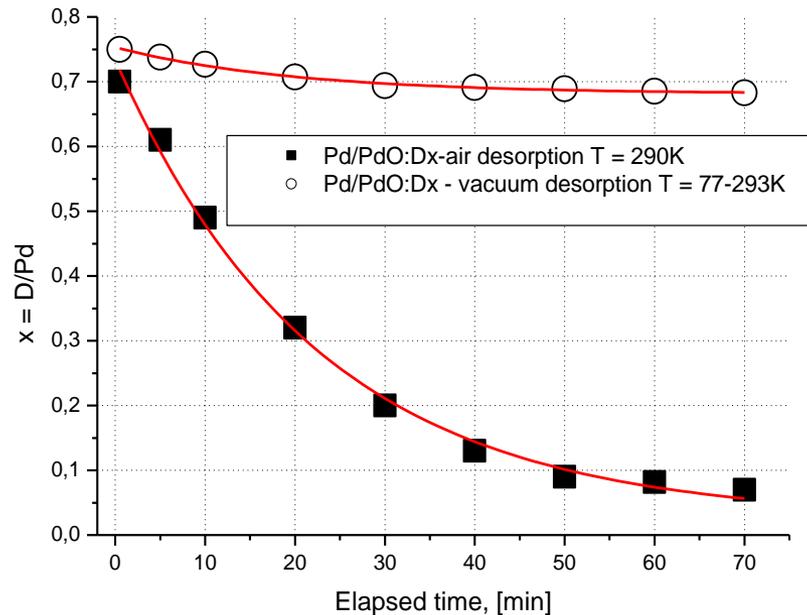
D-desorption rate from the Pd/PdO:Dx samples in vacuum (electrolysis at T=280 K with cooling down to T = 77K after electrolysis termination)) and in air at ambient conditions (electrolysis at T=290K). Notice very low D-desorption rate in vacuum

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TiD$_x$ sample preparation

- The Ti foils of 30 and 300 μm thick have been loaded in 1M solution of D_2SO_4 in D2O during t = 35 hr at J = 30 mA/cm^2, in order to dissolve the TiO_2 oxide layer at the Ti-surface and to provide D-penetration.

- The average loading (x = D/Ti = 1.0 at depth of 2-3 μm) has been determined by weight balance.

- It was found (by weight balance before and after e-beam irradiation) that the D-desorption rate in case of TiD$_1$ irradiation is consistent with the $J_D \sim 1.0$ x 10^{14} D/s-cm^2.

- All desorbed deuterons in TiD has been caused only by e-beam irradiation because the compound is absolutely stable at T = 300 K.

RBS/ERD profiles of the TiDx samples prior to e-beam bombardment –left; after D-desorption in vacuum during 60 min with e –beam (J=0,6 µA/cm2 U = 30 kV) - right

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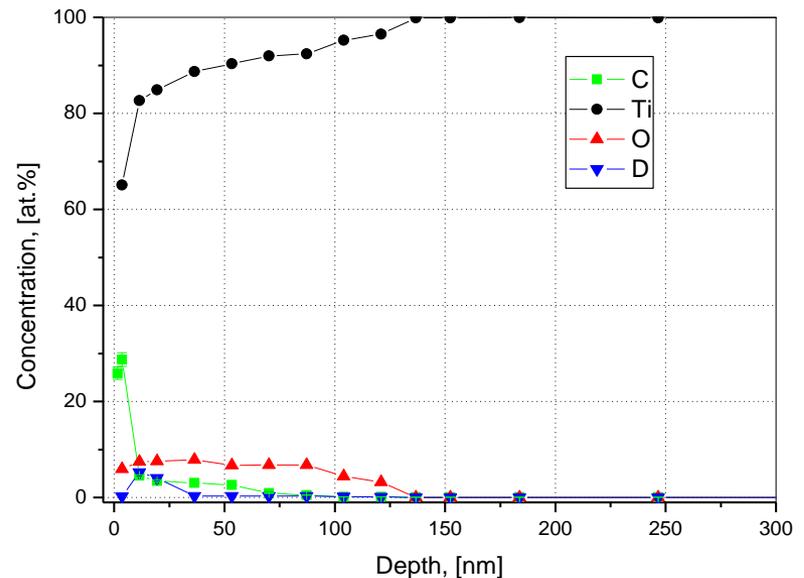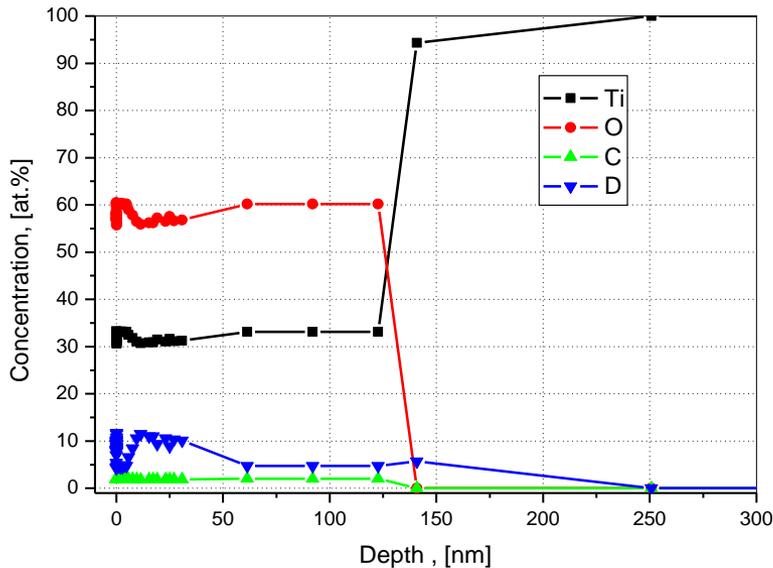
SEM (left) and STM images (right) of the PdOx surface of reference original Pd/PdO sample

2009.07.09   07:03   L   x6,0k   10 um

1.722 mkm (Z)

0.0 (Z)
16.76 mkm (X)

0.0 (Y)

16.68 mkm (Y)

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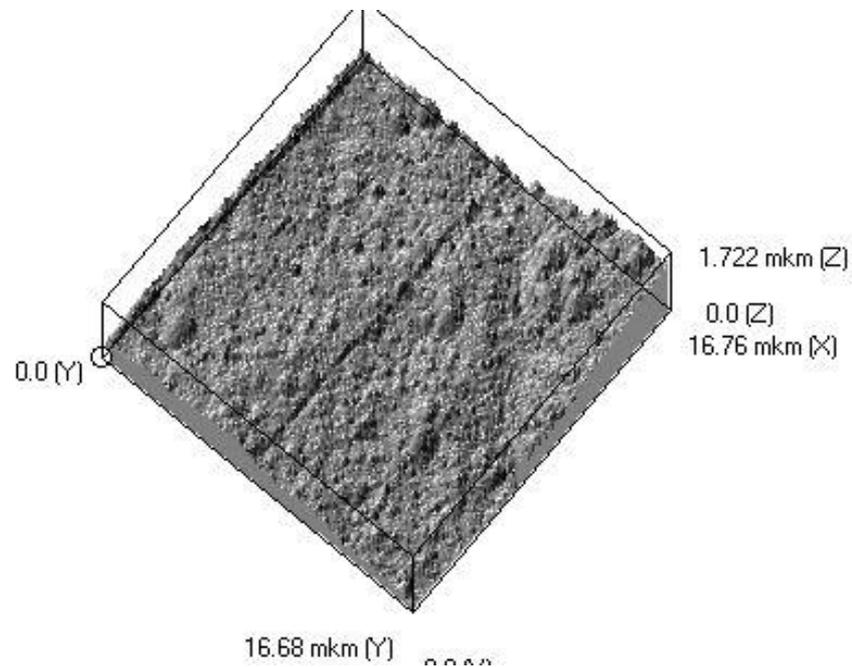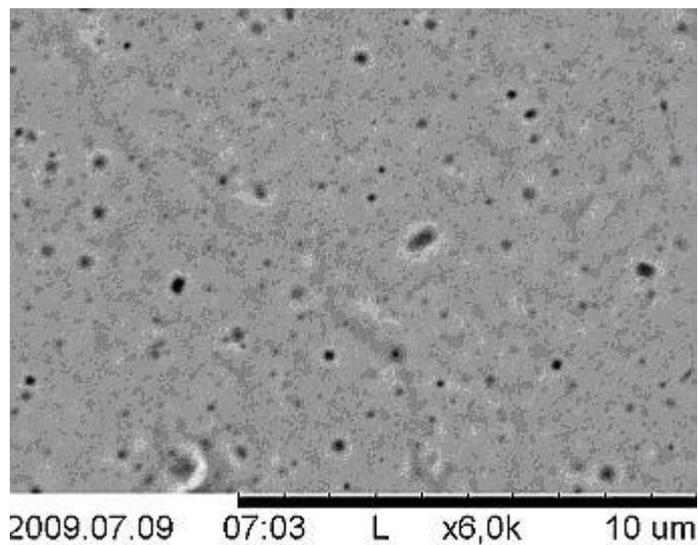
Pore diameter distribution in virgin (unloaded) Pd/PdO sample

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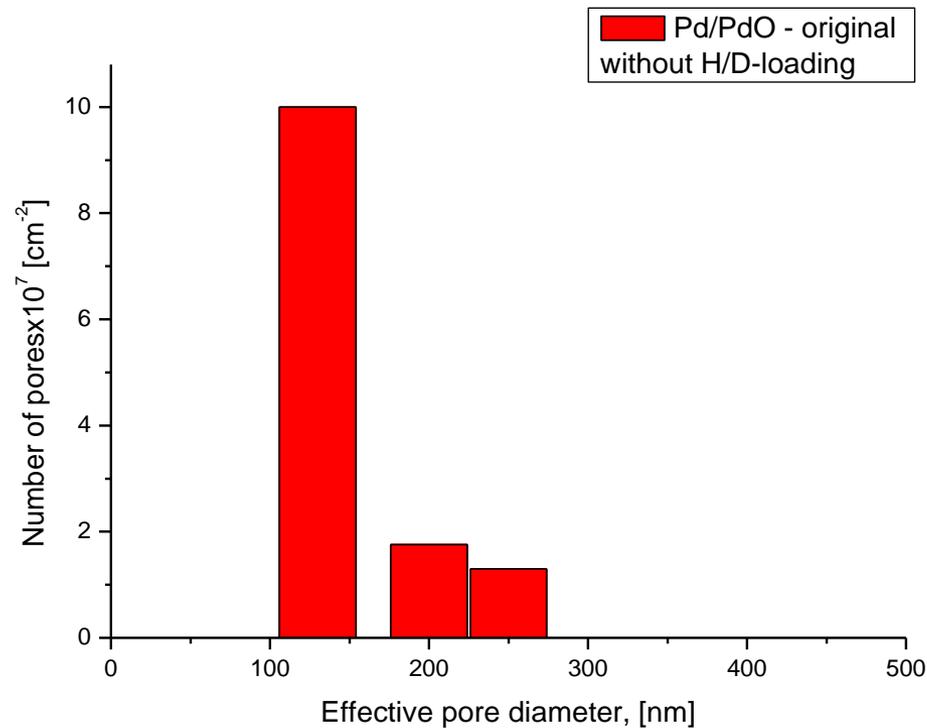
Reference Pd/PdO:Hx after electrolysis (left); after electrolysis +e-beam (right)

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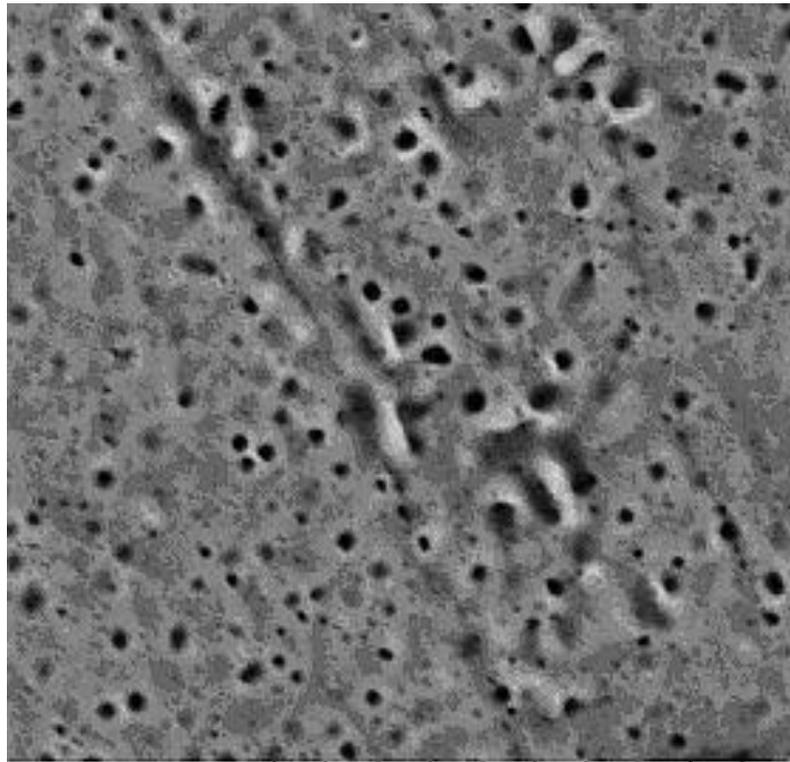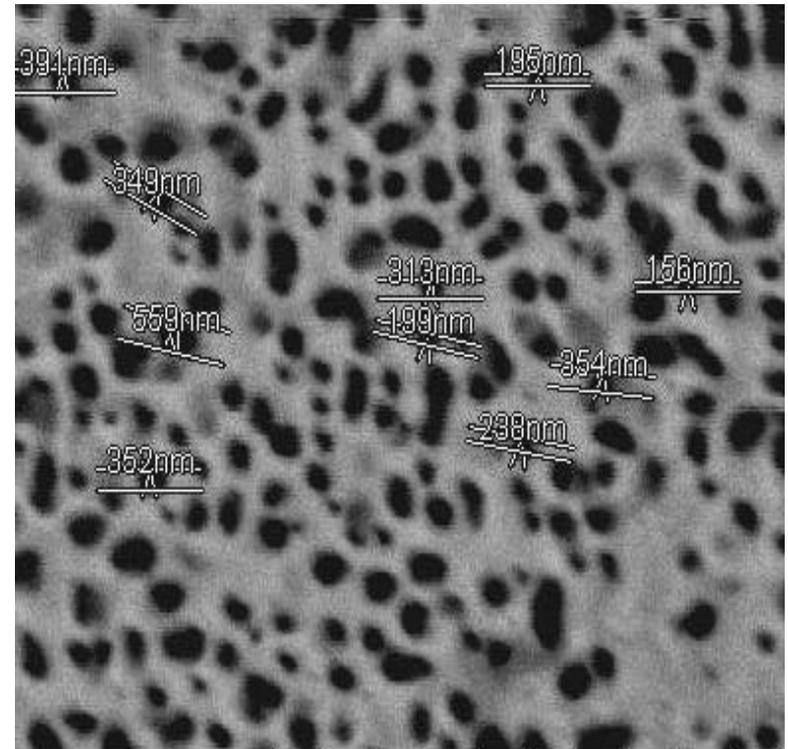
SEM Pd/PdO:D_x-left and Pd/PdO:H_x-right images after electrolysis + e-beam. Notice larger diameter pore generation in Pd/PdO:D_x
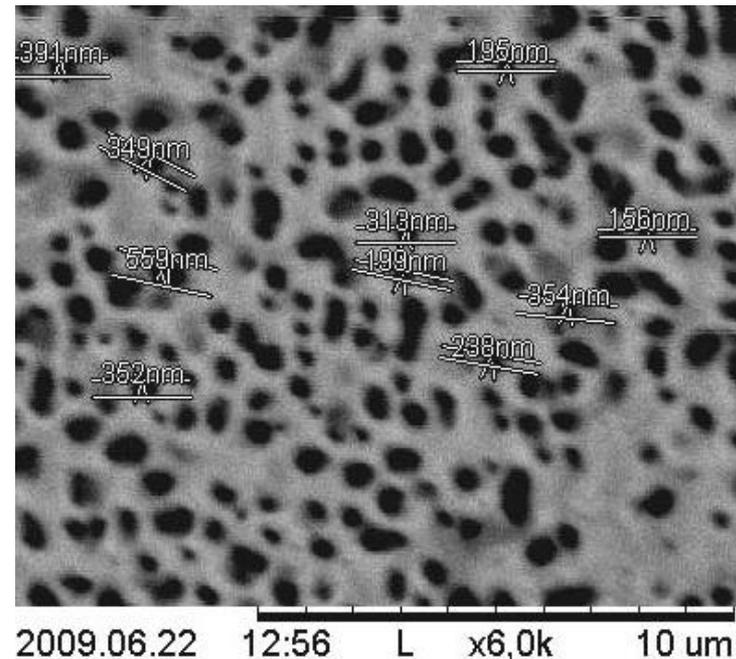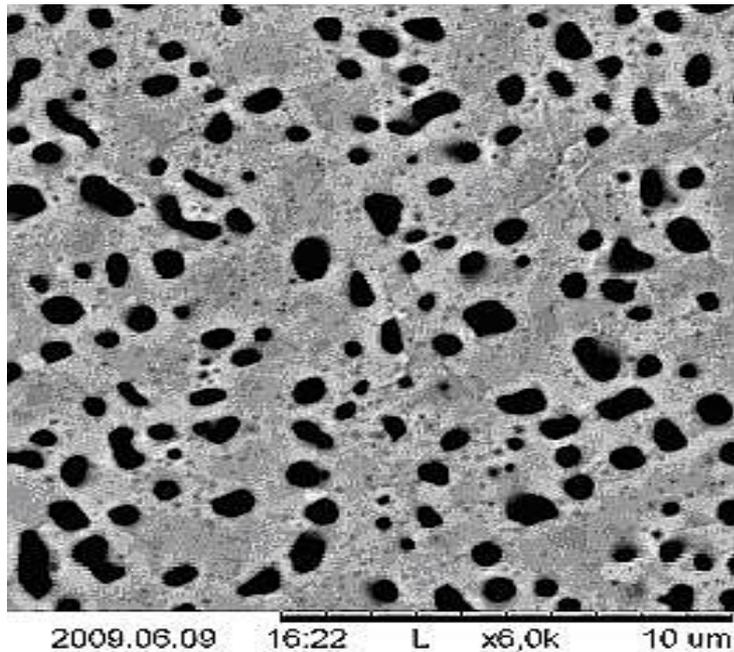
Pore diameter distributions in Pd/PdO:Dx and Pd/PdO:Hx before and after electrolysis

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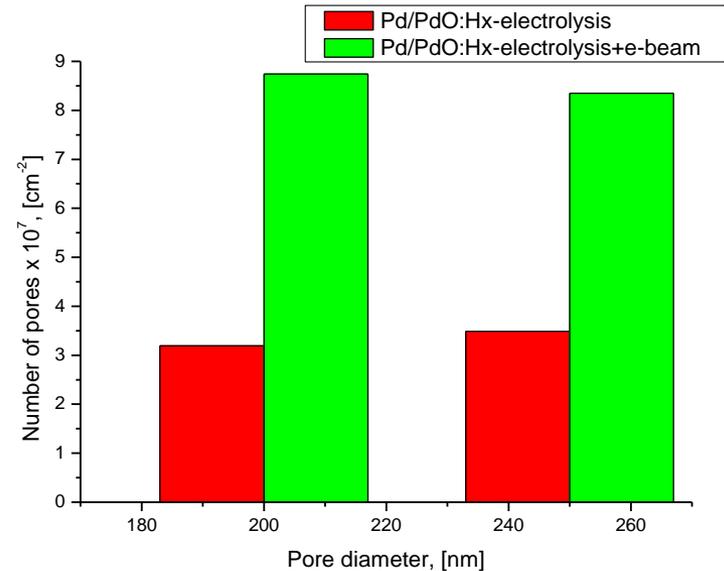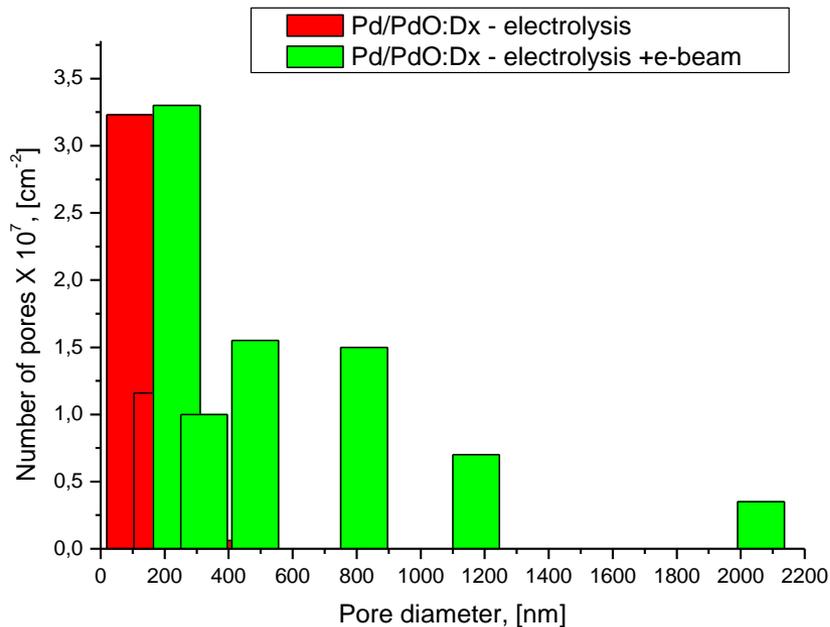
Example of the ~ 12 μm ⌀ "crater" at the Pd/PdO:Dx surface after e-beam bombardment
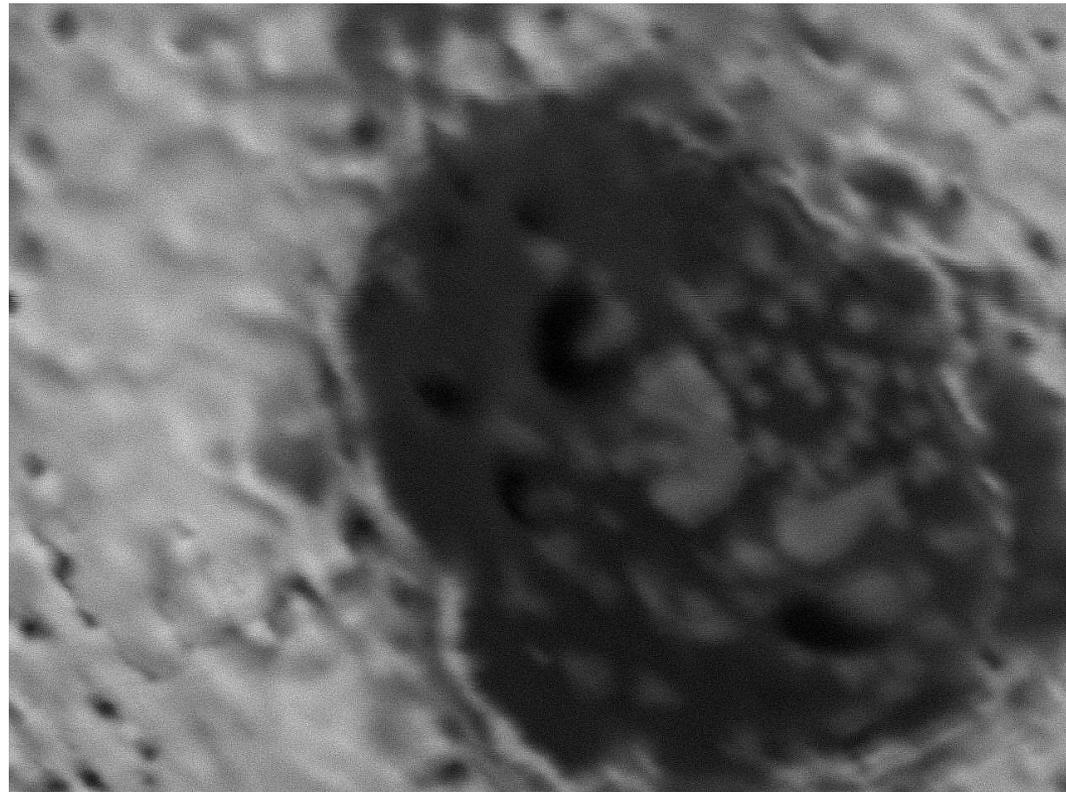
TM-1000_0550   2009.06.10   15:00   L   x6,0k   10 um

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TiD$_x$ surface with fragments of TiO$_2$ + TiC$_x$ coating (left), after e-beam (right)
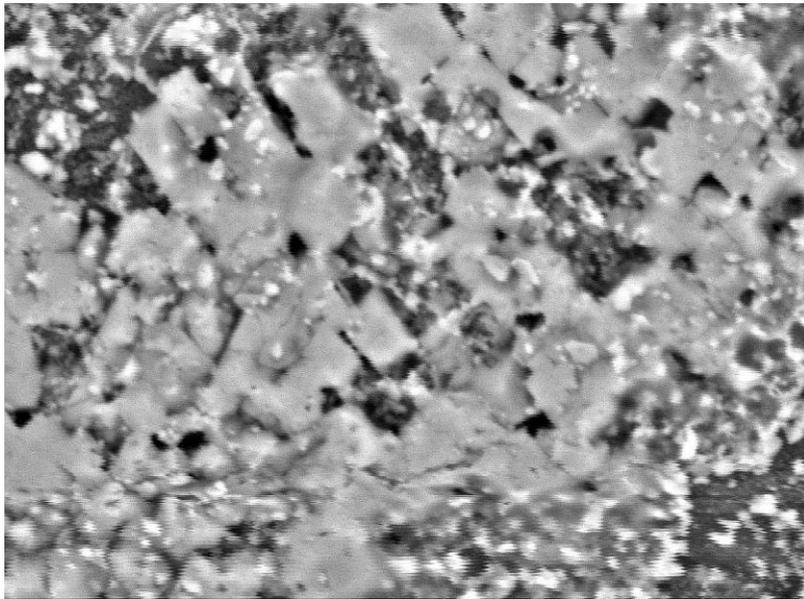
TM-1000_0480   2009.06.10   12:06   L   x6,0k   10 um
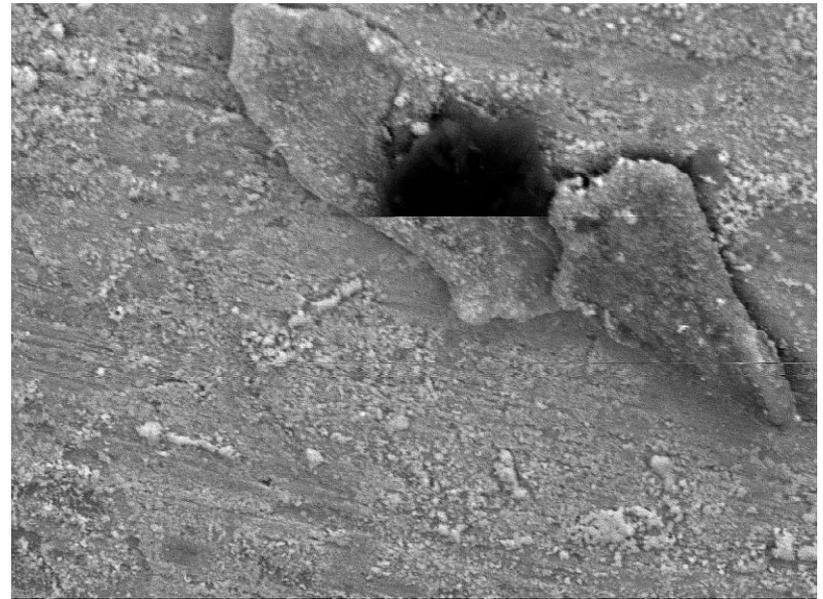
TM-1000_0541   2009.06.10   14:30   L   x2,0k   30 um

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Large crater at TiD$_x$ surface after X-ray irradiation
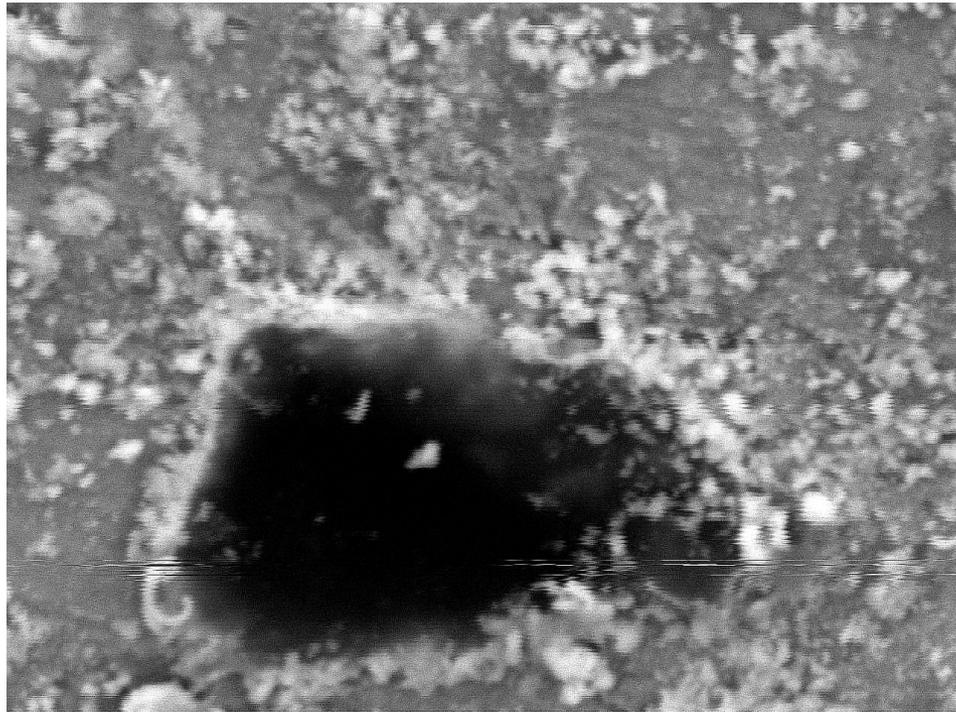
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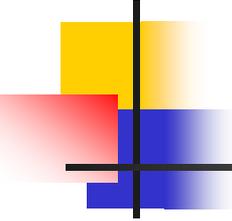
Results for Pd/PdO:D$_x$ target

- After 50 min of e-beam bombardment (J=0.6 µA/cm2 U = 30 kV) some moderate reduction of PdO and carbon layers is observed (from 40 to 25 nm). The residual D is located within the PdO layer.

- The mean D-desorption rate under e-beam in vacuum is compatible with that of D-desorption in air atmosphere (~ 2-3x 10^{15} D/s-cm^2).

- E-beam bombardment is accompanied by formation of numerical pores (from Pd through the PdO) with diameters in the range of 100 – 2000 nm. The larger $\varnothing$ pores ($\varnothing$ > 350 nm) have not been found in the reference Pd/PdO:Hx samples after e-beam.

- Large craters with the $\varnothing$ ~ 10-12 µm are also presented at the Pd/PdO:Dx surface after e-beam treatment.
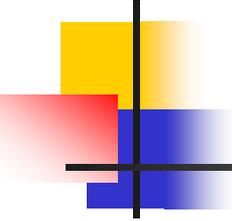
Results for TiD$_x$ target

- RBS data taken before e-beam treatment showed presence of TiO$_2$ oxide layer at the surface down to 150 nm. After e-beam the stoichiometry of the Ti oxide changes toward reduction of oxygen (TiO$_{0.1}$). The O-depth does not change. The X-ray irradiation show almost complete oxide reduction

- Estimate of D-desorption rate using RBS data before and after e-beam gives v(D) = 1.5x10^{14} D/s-cm^2, which is 20 times less than v(D) in Pd/PdO:Dx.

- The pore size is very small. Some large diameter craters ($\varnothing$ > 10 μm) are appeared at the TiDx surface after both e-beam and X-ray irradiation.

Conclusions: Surface Morphology and Consequences for LENR mechanism in Pd/PdO:D$_x$ target

- Presence of 25 nm thick PdO layer on top of the Pd foil does not allow to provide significant (Ed > 0.1 eV) acceleration of deuterons diffusing/desorbing from the bulk (metal Pd side). Reasons: the PdO is a semimetal with high enough conductivity (~ ρ 0.1 Ωxcm)- no surface polarization. The Debay screening radius is ~ 10 nm – not enough to get energy >> kT.

- Presence of long pores (at least 50 nm in length) make it possible to increase initial deuteron energy (provided by plasmons generated by e-beam) due to channeling. So, the screening potential Ue could be less than 750 eV (Ue ~ 600 eV requires E$_d$ ~ 20 eV).

- Formation of large craters at the surface indicates to high energy density (energy concentration) at some specific sites of the Pd/PdO surface through which the energy release is occurred (D+D -> D2+Q). These sites can show enhanced nuclear emission.
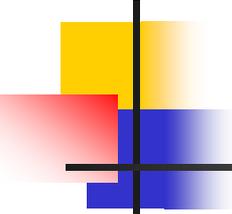
Conclusions: Consequences for nuclear emission mechanism/LENR in TiDx target

- To provide DD-reaction rate of the order of ~ 0.01 p/s-cm2 in the TiDx target under e-beam the kinetic energy of deuteron flux (in laboratory system) has to be rather higher (E_d ~ 500 eV), cause $U_e \leq 100$ eV for Ti.

- In the case of Ti/TiO2 interface with thick enough oxide layer, high kinetic energy of movable D+ can be gained by their acceleration in a strong electric field created by electrostatic charging of the TiO_2 surface with e-beam.

- $(E_d)_{eff} = \varepsilon_0 + e\ E(TiO_2)x\ h(TiO_2)$

- At ε_0 ~ 3 eV (the initial kinetic energy of D+ in Ti caused by plasmon generation) , $E(TiO_2)$ ~ 3.5×10^7 V/cm (electrical strength of TiO_2) and $h(TiO_2) = 1.5 \times 10^{-5}$ cm, the $(E_d)_{max}$ ~ 500 eV!!! suggesting D+ acceleration by a strong electric field during deuteron drift and diffusion through the Ti oxide.