CHARGED PARTICLE EMISSIONS AND SURFACE MORPHOLOGY OF THE Pd/PdO:D_x AND THE TiD_x TARGETS UNDER AN ELECTRON BEAM EXCITATION

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Recent *ab-initio* theoretical study [1] of interaction between electromagnetic radiation and metal deuterides indicate a new mechanism for deuteron acceleration, which along with possible large electron screening in the metal targets [2] could potentially strongly enhance the yield of DD-reaction in metal deuterides at room temperature. In this research we continue our study with regards to the role of electromagnetic excitation of hydrogen subsystem in metal deuterides to enhance the yield of low energy nuclear reactions (LENR). To this aim we have carried out 5 series of experiments on charged particle detection using plastic track detectors CR-39, under in-vacuum electronbeam stimulation of various metal deuterides during spontaneous deuterium desorption (if any) from the deuterated samples. In order to identify the type and energy of emitted particles the detectors were covered by metal (Al and Cu) films of various thickness. Moreover, the sequential etching technique, involving CR-39 etching in the range of 7-28 hr (removed CR-39 depth in the range of 9-37 μm) has also been applied. The obtained track diameters were compared with that obtained in calibration experiments, using CR-39 bombardment with charged particles (protons and a) of certain energy. We showed that electron beam stimulation of the D-desorption from Pd/PdO:D_x and TiD_x targets is caused by statistically significant emissions of DD-reaction product (3 MeV protons and 1 MeV tritons), as well as high energy alpha particles. Simultaneously, the blank experiments that included Pd/PdO:H_x charged particle measurements under e-beam excitation as well as the Pd/PdO:D_x and TiDx runs in the spontaneous D-desorption mode in vacuum (without e-beam bombardment) showed zero charged particle emission yield. Extrapolation of both DD-reaction cross section and the enhancement factor to very low deuteron energy (E_d ~ 3.0 eV) with a reasonable screening potential U_e = 750 eV, allowed to describe satisfactorily the detected DD-reaction yield in Pd/PdO:Dx target under e-beam excitation. This result strongly supports the theoretical prediction [1] with regards to electron excitation of the D-subsystem in the Pd/PdO deuteride. The surface morphology of the Pd/PdO:D_x samples loaded with deuterium electrochemically, prior to and after the electron beam bombardment has also been studied using SEM, AFM and STM techniques. It was found that electron beam interaction with the surface of the Pd/PdO:D_x produce specific channels of 100-500 nm in diameter as well as deep craters with the diameter of several microns, while the surface which has not been subjected to the e-beam bombardment shows smooth PdO structure. The origin of these surface defects and their role in deuteron acceleration (channeling) will be discussed.

[1] V. Silikin, I.Chernov et al, Phys.Rev.B, **76**, 245105 (2007).
[2] H. Yuki, J. Kasagi , A. G. Lipson et al, JETP Lett, **68**(11) , 785, (1998).

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.

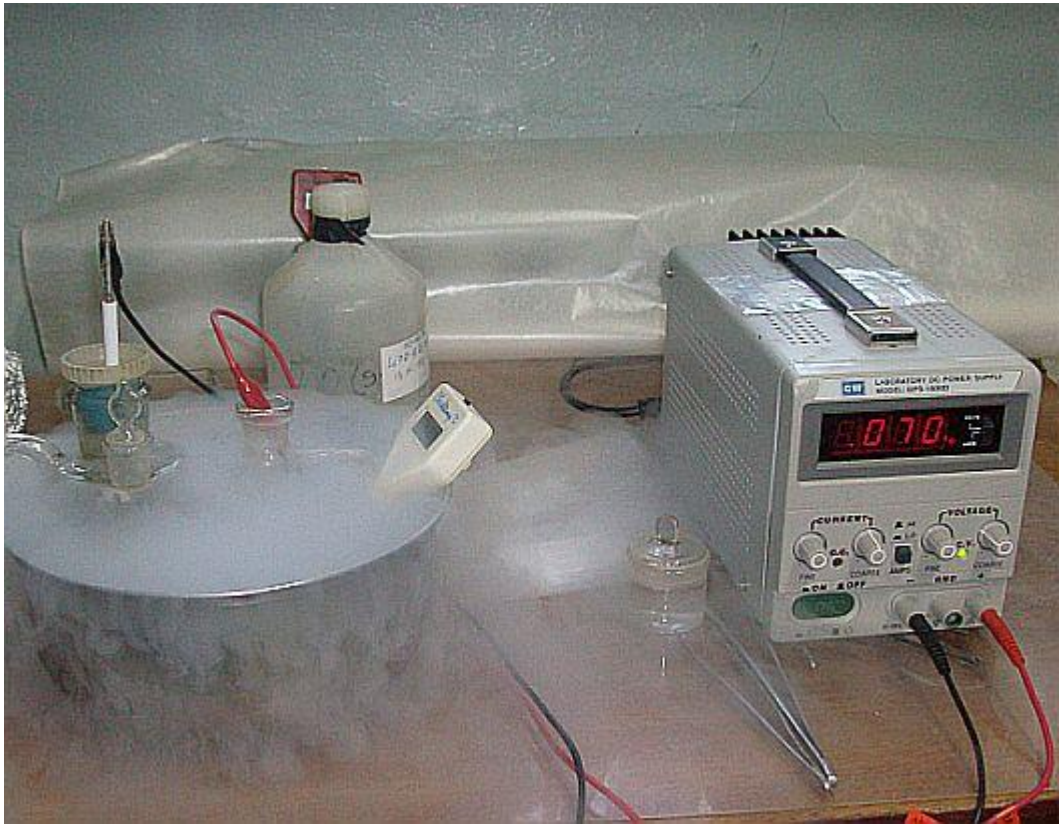
Objectives

- 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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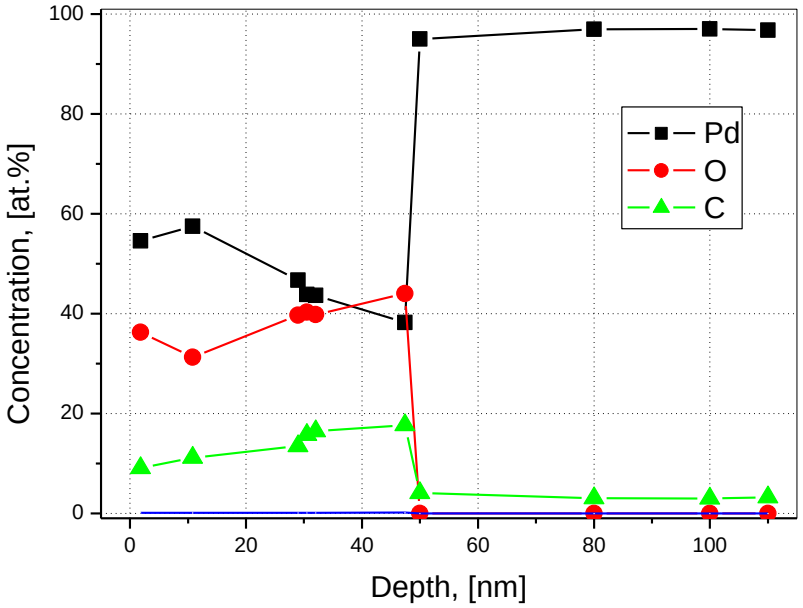
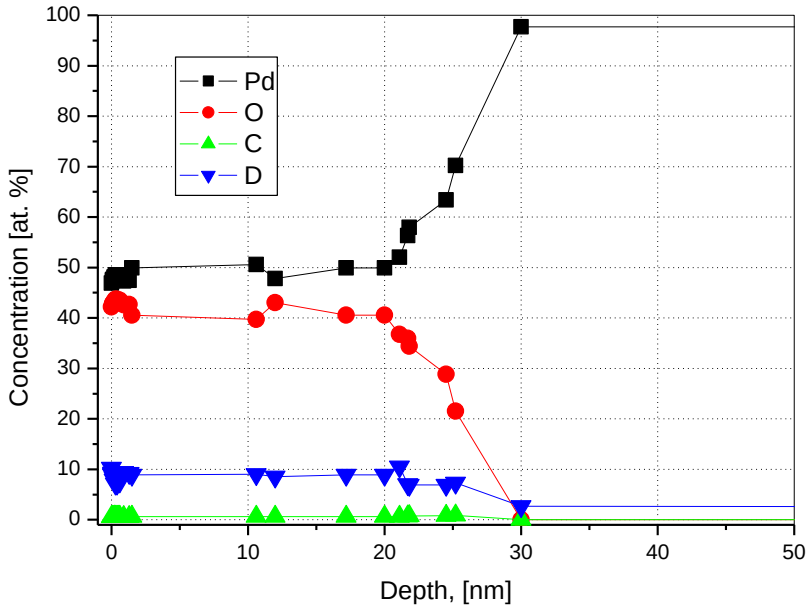
Pd/PdO:Dx Sample preparation

- 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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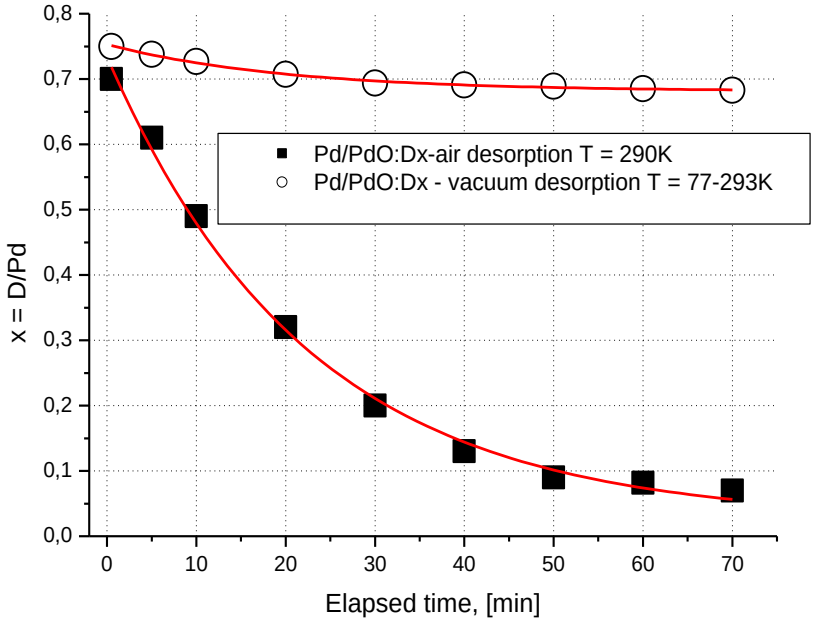
D-desorption from the Pd/PdODx sample in vacuum without e-beam excitation and in air. Under e-beam the desorption rate is $J_D \sim 3.3 \times 10^{15}$ D/s-cm^2. This number of desorbed deuterons is consistent with the rate of D-desorption in air at ambient conditions



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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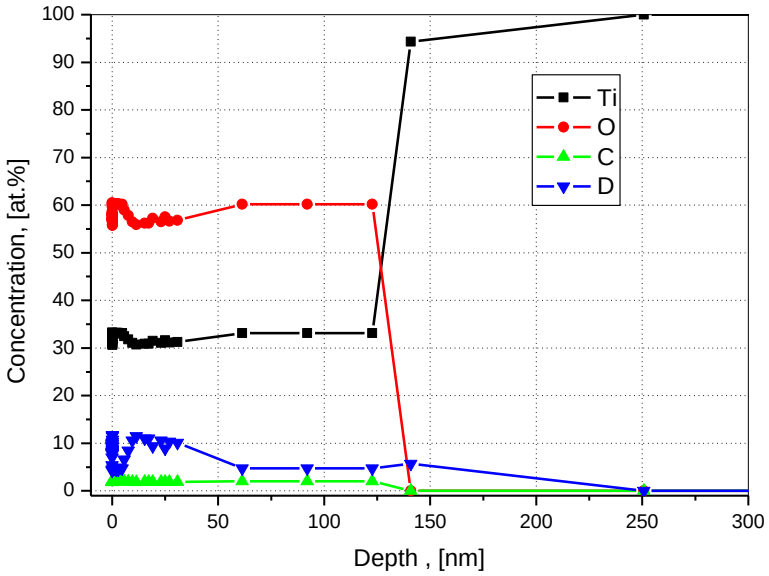
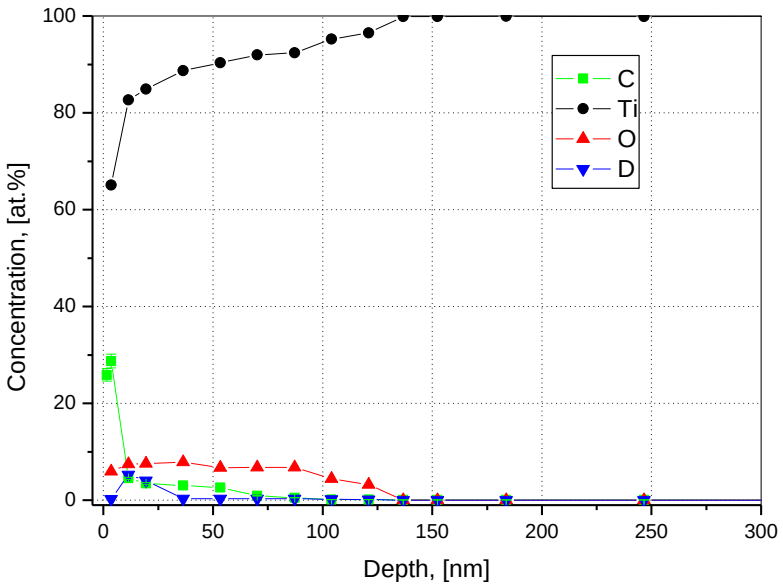
TiD_x sample preparation

- 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 $J_D \sim 1.0 \times 10^{14}$ 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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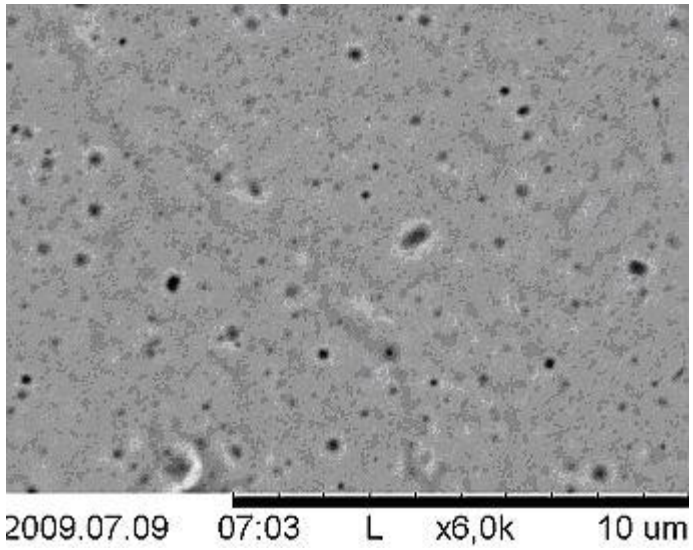
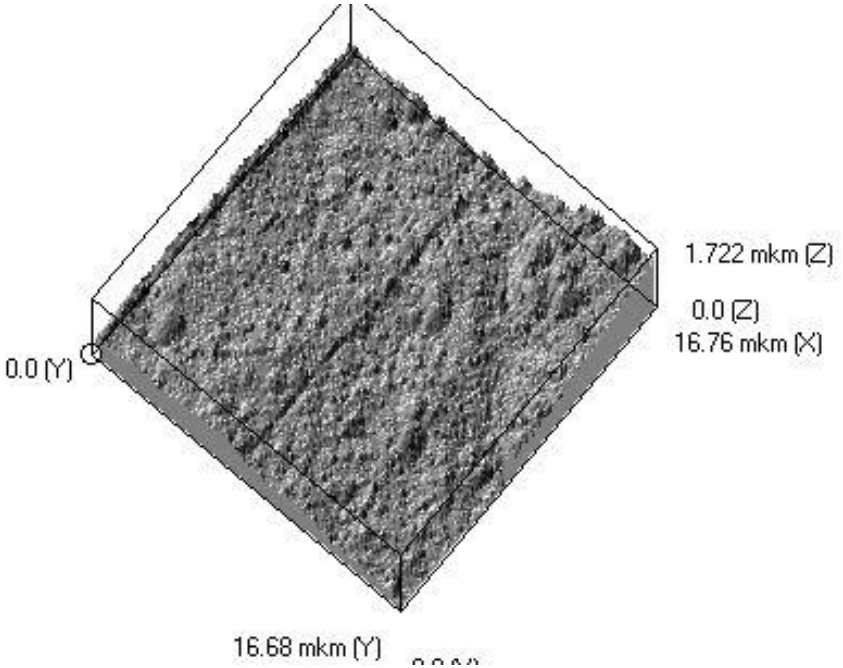
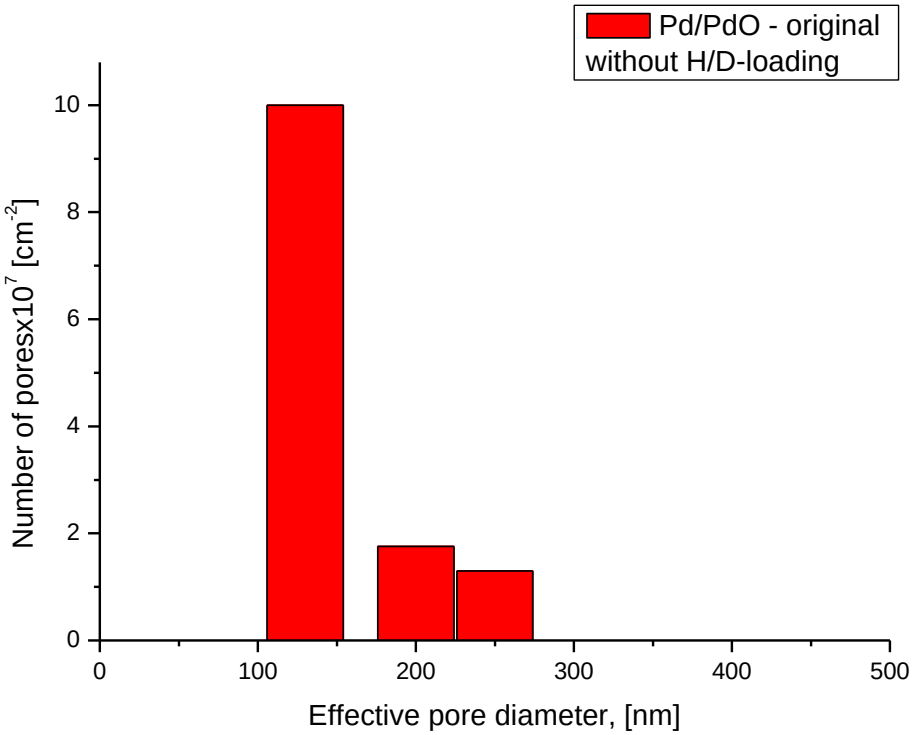
SEM (left) and STM images (right) of the PdOx surface of reference original Pd/PdO sample





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



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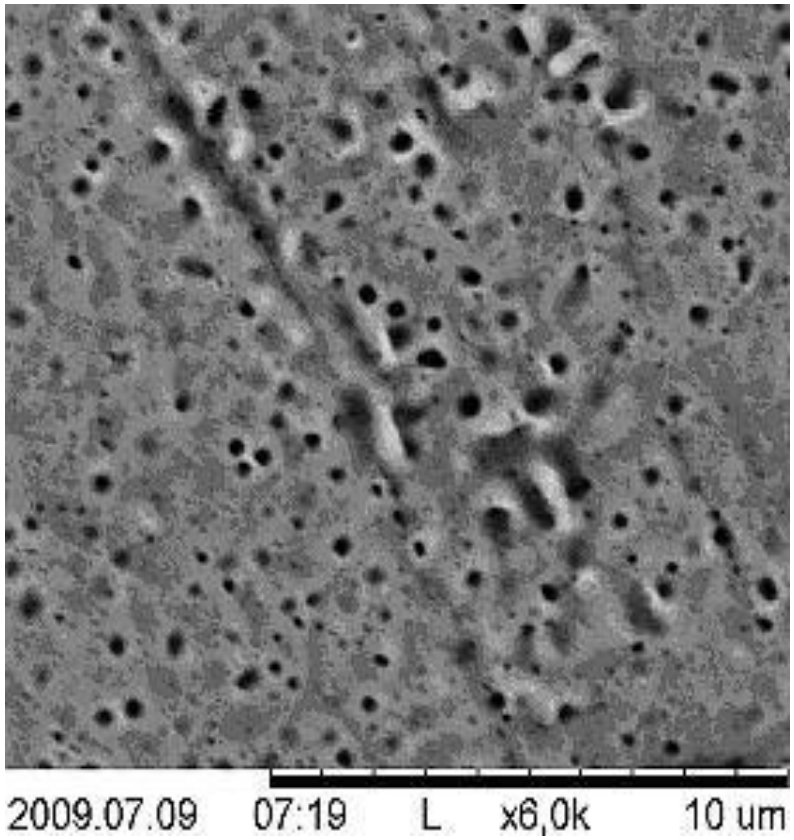
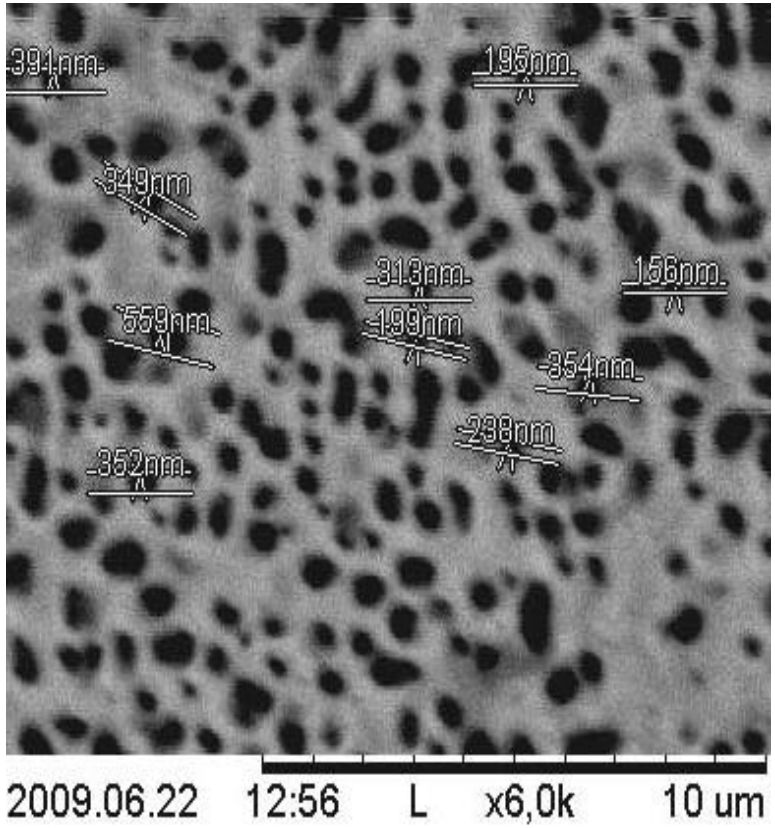
Reference Pd/PdO:Hx after electrolysis (left); after electrolysis +e-beam (right)





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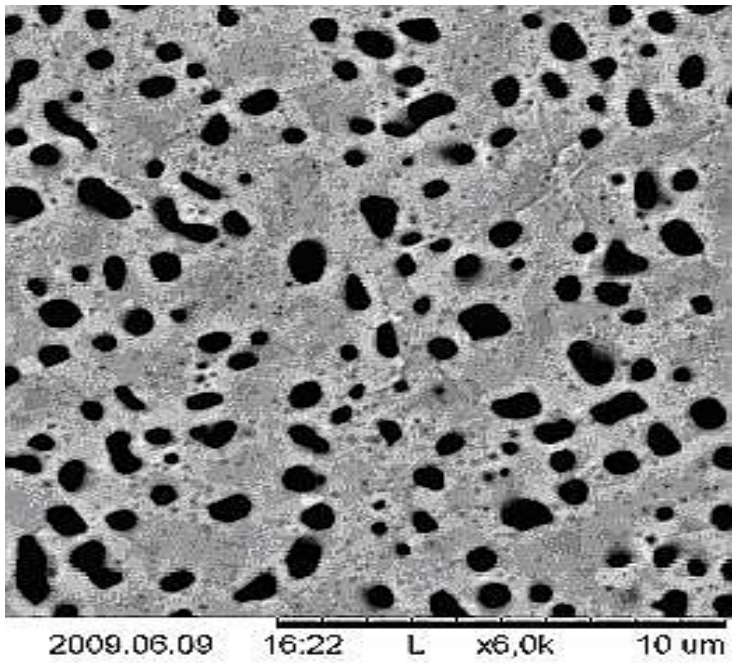
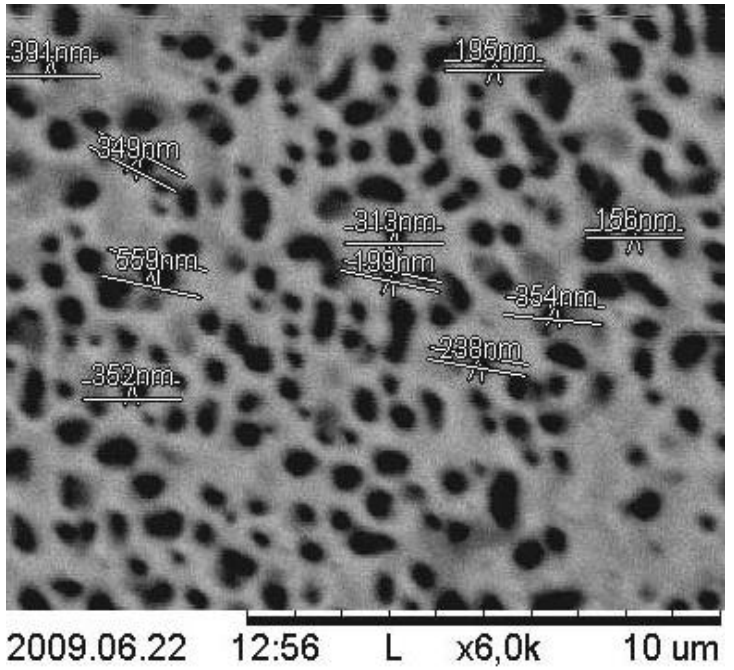
SEM Pd/PdO:D_x-left and Pd/PdO:H_x-right images after electrolysis + e-beam. Notice larger diameter pore generation in Pd/PdO:D_x





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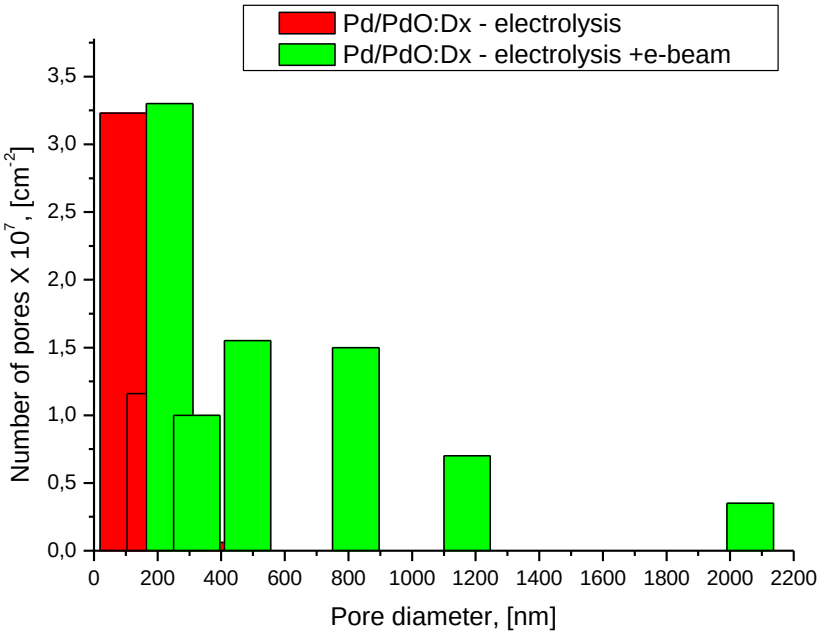
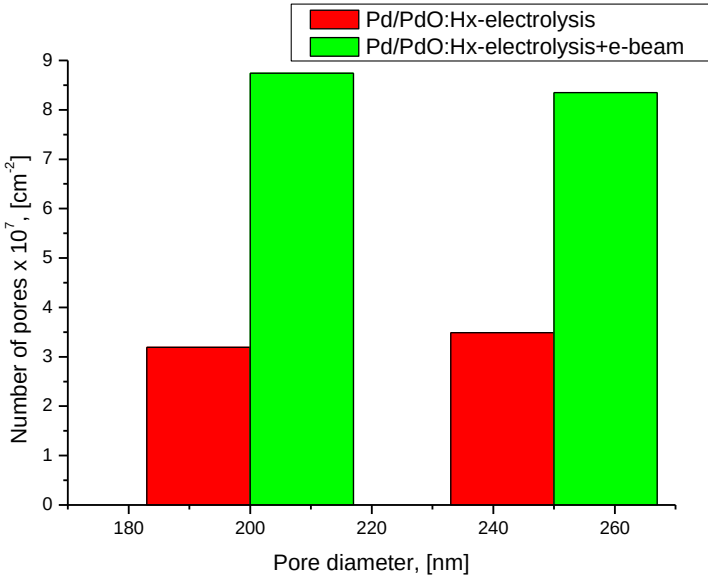
Pore diameter distributions in Pd/PdO:Dx and Pd/PdO:Hx before and after electrolysis





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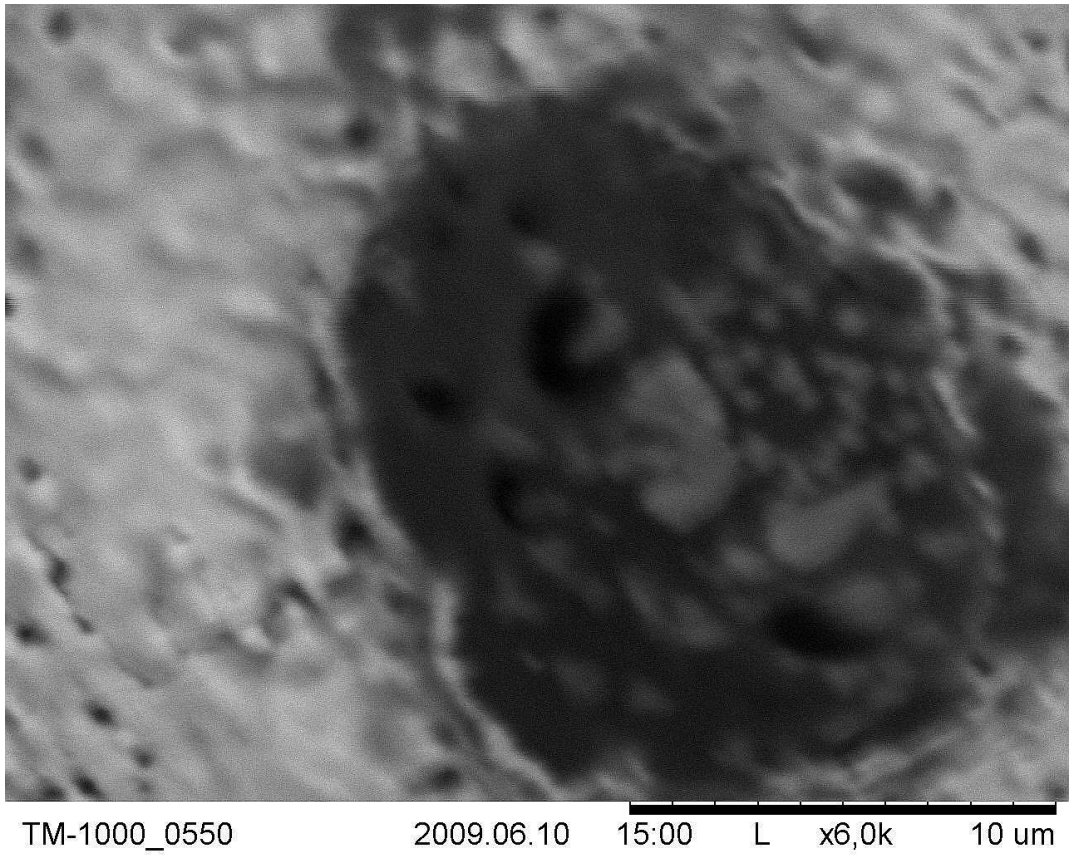
Example of the ~ 12 μm ∅ "crater" at the Pd/PdO:Dx surface after e-beam bombardment



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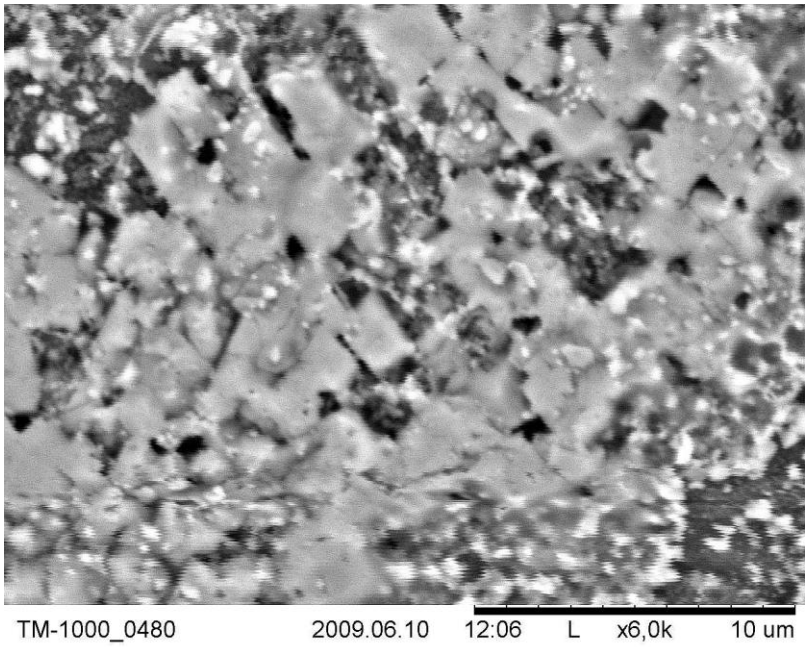
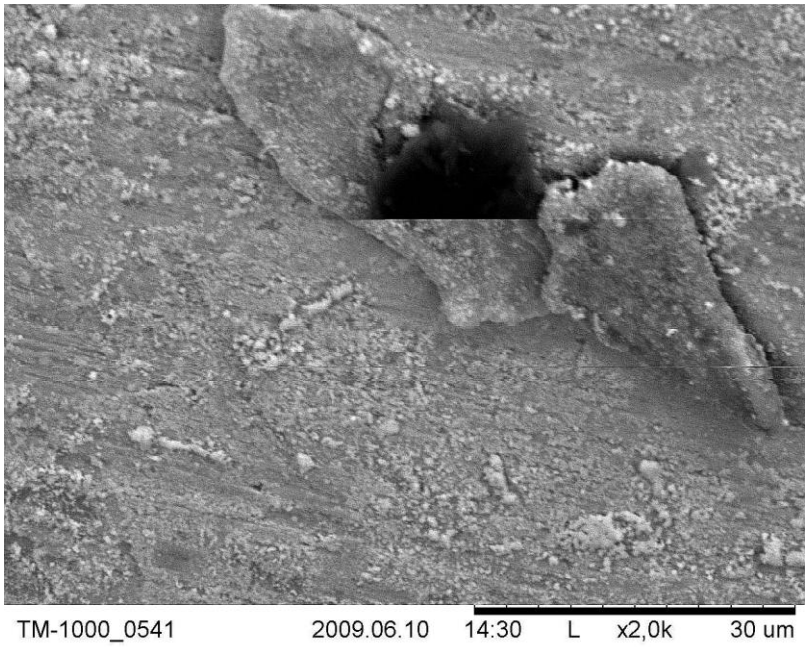
TiD_x surface with fragments of TiO_2 + TiC_x coating (left), after e-beam (right)





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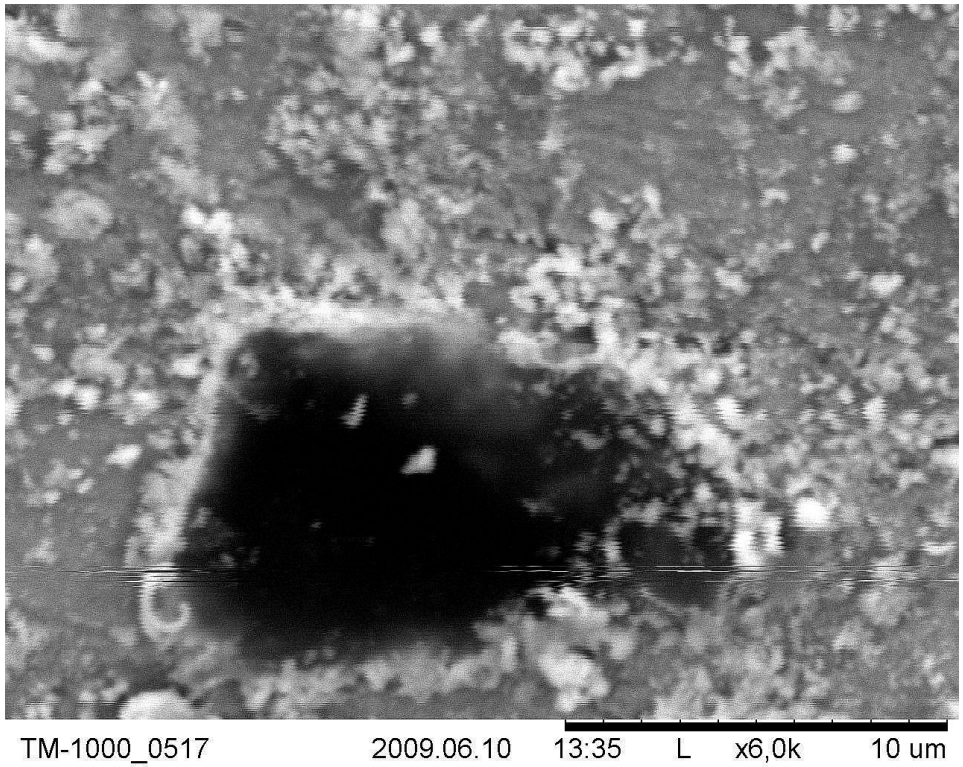
Large crater at TiD_x surface after X-ray irradiation



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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 ∅ pores (∅ > 350 nm) have not been found in the reference Pd/PdO:Hx samples after e-beam.
- Large craters with the ∅ ~ 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) = 1.5×10^{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 (∅ > 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) , $E(TiO_2) \sim 3.5x10^7$ V/cm (electrical strength of TiO_2) and $h(TiO_2) = 1.5x10^{-5}$ cm, the $(E_d)_{max}$ ~ 500 eV!!! suggesting D+ acceleration by a strong electric field during deuteron drift and diffusion through the Ti oxide.