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REPRODUCIBILITY OF EXPERIMENTS IN GLOW DISCHARGE AND PROCESSES ACCOMPANYING DEUTERIUM IONS BOMBARDMENT

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ABSTRACT

The problems of reproducibility of experiments in glow discharge (GD) and electrolysis are considered. The difficulty in estimation of nuclear and non-nuclear processes contribution in isotopic and elemental composition change in material irradiated by ions is noted.

The post-experimental charged particles flow from samples in Deuterium GD was measured. The current ranging $\sim 10^{-6} \text{A}\cdot\text{cm}^{-2}$ - $\sim 10^{-13} \text{A}\cdot\text{cm}^{-2}$ was registered in the first 1 - 3 post-experimental minutes for different materials (Pd, Pd alloys, Ag and Mo) and experimental parameters. The emissions duration lasted 30-100 minutes and depended upon experimental parameters.

Analysis of tracks on X-ray films placed inside and outside of a metal GD chamber has shown existence of tracks varying from several to tens of millimeters. The tracks were of various shapes: round and curvilinear, and also rotating including double spirals.

PROBLEMS of REPRODUCIBILITY

The problem of why researchers get different effects in similar experimental conditions in electrolysis and in gas GD experiments attracts a lot of attention. All "pro" and "against" are not clear yet. However the questions of the surface preparation [1], configuration and surface topography [1], the contribution of preferably lighter elements sputtering from the surface, and also the contribution of chemical sputtering seems no less important.

The following processes influence the surface isotopic and elemental composition change in the course of ion bombardment: 1- preferential sputtering of lighter isotopes; 2 - diffusion separation; 3 - concentrations gradient; 4 - mechanical stresses gradient; 5 - implantation of elements from the device parts and from gaseous environment. However, the experimental data and calculations supplied by numerous researchers show that the change of surface composition in binary alloys for $\sim 0,5$ - 5 keV energies were small and were kept within the limits of measurement errors. The change of isotopic surface composition during ionic bombardment resulting from mass distinction and surface connection energy for pure metals is insignificant. The preferential sputtering - isotope separation with energies < 1 keV is equal or < 10 %, for isotopes with masses of ~ 100 it amounts to $\sim 0,003$ % [2].

The influence of hydrogen on anomalies of processes should be taken into account. Among them are enhanced diffusion, interaction of hydrogen complexes with different types of structural defects, accelerated transport of impurities along dislocations and fastening of impurities (including gas impurities) on the defects [3].

It is known that irradiation of materials by low-energy ions is accompanied by such physical processes at the atomic level which proceed simultaneously: knocking of atoms from the crystal lattice nodes, sputtering and re-precipitation of atoms. And the resulting formation of vacancies and interstitions, their clusters, and also of separate dislocations, dislocation loops, grids and dislocations and pores accumulation is going on. Sputtering on the defects leads to changes in surface topography, formation of cones, salients, "whiskers". It can also bring about changes in local current density, microarcing and microexplosions on the "tips" and other defects.

The microarcs and microexplosions, in turn, can be by a reason of high cramping and pulling stresses in microvolumes of the surface layers as result of the fast instantaneous melting and hardening in these microvolumes. The stresses can result to dislocation formation and to intensification of diffusion processes, to acceleration of the transport of impurities along dislocations at hundreds and thousands times and can give the redistribution of impurities.

POST GD MEASUREMENT of NEGATIVE PARTICLES FLOW from the CATHODE SAMPLES

The effect of "life after death" (as M. Fleischmann had called it in his paper "Heat after Death" [4].) can also be registered for negative charged particles from sample exposed to deuterons after GD switch off. "Life after death" was confirmed in the papers published earlier on glow discharge investigations (ICCF2 - - ICCF7 and the 1-7th Russian Conferences on Cold Fusion and Cold Nuclear Transmutation and also in others publications). The authors noted that X-ray films after contact with samples exposed to Hydrogen and Deuteron in GD became black too [5]. It was assumed that the blackening resulted from β -emissions. This kind of emissions was also registered in 15 minutes after removal of samples from the discharge chamber. The "hot points" on the upper Pd samples were observed for the first 2 hours of the exposure.

Some data on charged particles registration from cathode samples after the GD switch off are presented, the measurements having been taken in $\sim 1 - 2$ minutes.

The measurement of charged particles flow was carried out with the help of U5-6 electrometric amplifier. The electrometric amplifier is designed for measurement of positive or negative charged particles flow (for stationary and slowly changing currents) within the range of $1 \cdot 10^{-13} - 1 \cdot 10^{-6}$ A. U5-6 amplifier operates on the principle of small currents measurement in accordance with the number of voltage drops on the fixed resistance ($1 \cdot 10^{12}$; $1 \cdot 10^{10}$; $5,1 \cdot 10^6$ Ohm). There are three overlapping ranges for current measurement: I - ranging $\sim 1 \cdot 10^{-13} - 1 \cdot 10^{-10}$ A; II - ranging $\sim 1 \cdot 10^{-12} - \sim 1 \cdot 10^{-8}$ A; III - ranging $\sim 1 \cdot 10^{-9} - \sim 2 \cdot 10^{-5}$ A. The measurement error of the first range amounted to ~ 10 %, the second and the third ± 5 %, correspondingly. The peak value for low-frequency fluctuations voltage corresponds to noise for each range: I $\sim 3 \cdot 10^{-15} - 5 \cdot 10^{-16}$ A, II $\sim 1 \cdot 10^{-13} - 2 \cdot 10^{-14}$ A, III $< 1 \cdot 10^{-11}$ A.

The samples were of 20 mm in diameter, ~ 100 μm in thickness and the exposure area of 1 cm^2 . The operating gas used was Deuterium. The chamber pressure was down to 10^{-4} Torr and up to 5-15 Torr when filled with gas. The samples were exposed to irradiation at 30-300 mA current, 50-1100 Volts voltage, the time duration ranging 1,5-4 hours. The charged particles measurement system calibration was carried out on a tritium target placed instead of a sample in the vacuum discharge chamber. The electrometer showed a current $\sim 5 \cdot 10^{-11}$ A from a tritium target from $\sim 1 \text{ cm}^2$ area with radioactivity $\sim 4,1 \cdot 10^9 \text{ cm}^{-2} \text{ c}^{-1}$ in vacuum $\sim 1 \cdot 10^{-4}$ Torr. It means that β -emission after ten minutes post-irradiation was usually $< 10^7 \text{ cm}^{-2} \text{ c}^{-1}$.

The post-irradiation sample current measurement diagram is given in Fig. 1

The duration of the emission varied by two or three times and the registration duration amounted to $\sim 30 - 100$ minutes. The current within first minutes was usually the biggest. However some samples showed weak current within the limit of electrometer sensitivity threshold. It depended upon the following parameters: current density at the first stage (during loading), quantity of loaded Deuterium at this stage, cathode temperature and less dependence from Voltage. The emission current for various Pd samples ranged $\sim 1 \cdot 10^{-6} - \sim 2 \cdot 10^{-13}$ A and was dependent upon the parameters of the process. The tendency to increase current for Pd during gas evacuation from 10^{-1} to 10^{-3} Torr was observed. Current was changed from $9,7 \cdot 10^{-7}$ to $1,2 \cdot 10^{-6}$ in the course of 80 minutes. Current from Ag sample increased by -20 % (to $\sim 1,2 \cdot 10^{-6}$ A) in the course of 15 minutes of gas pumping-out of deuterium from $1 \cdot 10^{-1}$ Torr to 10^{-4} Torr and was decreased to $\sim 10^{-14}$ A. One hour post-irradiation (β -emission was always $< 10^6 \text{ cm}^{-2} \text{ c}^{-1}$). So, 10^{-13} A correlates with $10^7 \text{ cm}^{-2} \text{ c}^{-1}$ β -emission. It conformed well with Fig. 4 [5].

In PdPtW alloy the current was increased by ~ 2 times for 20 minutes after GD switch off (up to $\sim 4 \cdot 10^{-5}$ A). The unstable short-term increase of emission current on PdPtW for the first 1 - 2,5 minutes after the GD switch off was noted. The current after GD switch off decreased by $\sim 5 \cdot 10^{-6}$ A $\sim 4 \cdot 10^{-7}$ A, and then increased up to $\sim 1 \cdot 10^{-6}$ A. The emission current was lower by $\sim 10^{-5} - 10^{-7}$ times when the cathode temperature was higher by $10 - 20^\circ$ in the course of the experiment (for example, $\sim 10^{-7}$ A and $\sim 10^{-13}$ A). Some data on current from samples 2 minutes after GD switch off are given in Fig. 2. The post-experimental current did not diminish with drop of temperature by $100 - 150$ $^\circ\text{C}$. It was not the current of a thermo-ionic emission because for Pd and Mo at temperature 500°C it amounts to $\sim 10^{-21}$ and $\sim 10^{-23}$ A cm^{-2} , accordingly. With temperature drop the current of a thermo-ionic emission impinges. The correlation of quantity and duration of current after the GD switch off versus a plasticity and brittleness of a material sample is not observed, therefore it was assumed, that the current does not result from the stress electronic emission.

STUDY of the TRACKS ON X-RAY FILMS.

The method for X-ray films blackening estimation for registered radiation was also applied. The X-ray films with a series of the screens (metallic and organic) inside and outside of the GD chamber were used. We observed the blackening of X-ray films, the diffraction spots and the tracks of unusual shape. The tracks on X-ray films placed inside the GD chamber were spaced at 350 mm from the samples and were similar to the tracks in (Wilson) expansion chamber and in the bubble chamber. The aggregate of paths with regard to the black specks, some rectilinear, some curvilinear, some as though from tracks of the particles refracted by the strong electric field. Sometimes these tracks reminded tracks from "impinging stars" or comets. The clearly visualized spiral tracks were

most unusual. These tracks had sizes ranging from one to tens of millimeters. Such tracks were observed on x-ray films inside the chamber at a distance of 300 - 350 mm from the cathode and outside the stainless steel chamber with ~ 5 mm thick walls (Fig. 3).

Similar tracks were observed by Takaaki Matsumoto. He termed them "artificial ball lightning", which can be initiated by cold fusion processes [8]. We also observed the spiral tracks with dimensions up to several tens of millimeters. The coils of the spiral were of varying brightness. These tracks are shown on Fig. 3. A lot of small tracks look as separate zigzags, irregular tracks. It is impossible to explain the spiral and zigzag tracks as resulting from the ion bombardment or as the flaws of the x-ray films. Similar effects were absent from the x-ray films - witnesses placed at a distance of several meters from the GD chamber. Maximum tracks on x-ray films (for films exposed inside and outside the GD chamber) for maximum isotopic and chemical changes were observed.

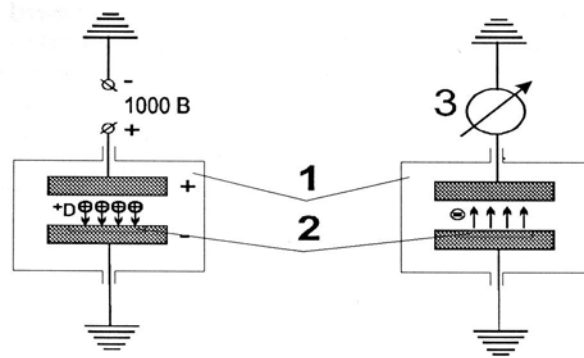


Fig. 1 The post-irradiation sample current measurement diagram after Glow Discharge switch off Left-irradiation diagram; right-post-irradiation measurement (1-vacuum chamber, 2-sample, 3-amplifier-electrometer)

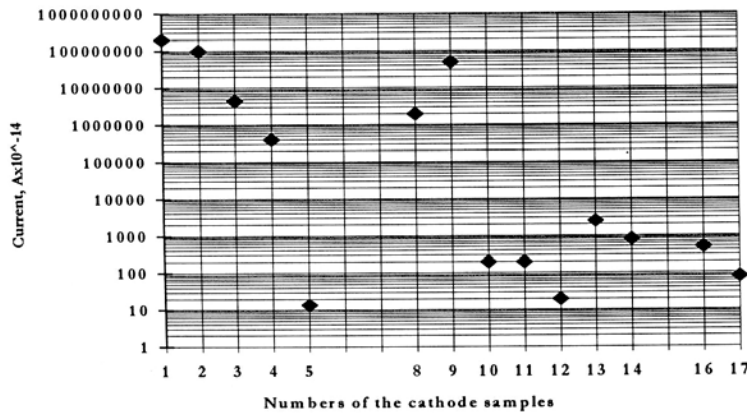


Fig. 2. Current from samples "in situ" after two minutes Glow Discharge switch off, $A \cdot 10^{-14}$ (1-17-number of the samples: #1-5 Pd; #8 - PdRu; #9 - Ag; #10-11 PdPtW; #12 -PdNi; #13-14 - Cu-Pd; #16-17 Mo)

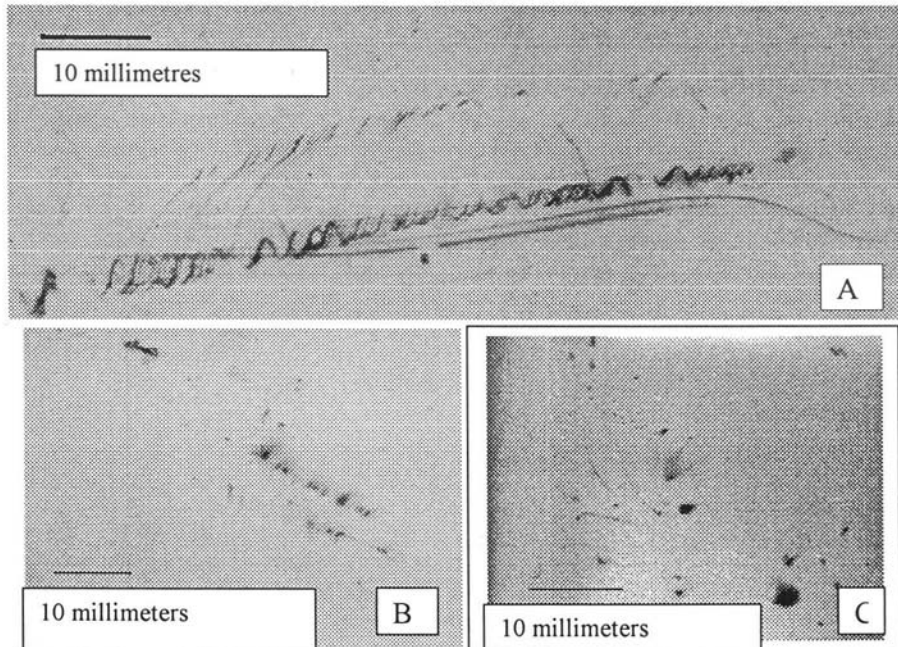


Fig. 3. The unusual traces on x-ray film outside (A, B) and inside the vacuum chamber after deuteron irradiation in Glow Discharge (C)

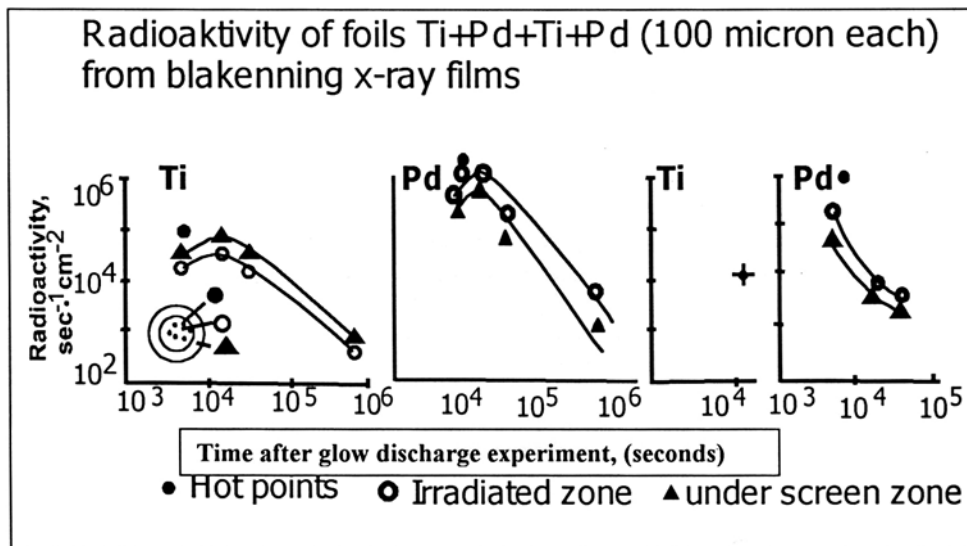


Fig. 4.

DISCUSSION

It is difficult to share the contribution of nuclear and atomic processes in balance of measurements for mass (chemical) composition change, heat measurement and energy which is carried away with radiation. However even the most accurate measurements can not describe all apparent processes going on simultaneously on GD cathode fully enough.

Here are possible reasons for non-identical reproducibility of experiments in GD resulting from stochasticity: 1. formation of various structural defects for every experiment: vacancies, interstitions, its clusters, voids and pores, inter-weavings, separate dislocations, segregation and nets of dislocation, hydrogen - vacancy complexes; 2. preferential sputtering on structural defects of a crystal lattice; 3. preferential accumulation of deuterium on defects of crystal lattice during ion implantation; 4. separation of elements with various mass number; 5. shaping of craters in places of micro-arcs and micro-explosion formation; 6. change of surface topography, formation of whiskers and cones. The processes (1-6) are going on at the atomic level.

The following facts result from nuclear reaction: 1) the local blackening of X-ray films in contact with samples irradiated by deuterium ions in GD is consequence of "the hot points" — the local nuclear reactions (Fig. 4), and the radioactive isotope formation [5, 9]; 2) the tracks of various shape on X-ray films inside and outside the GD chamber can proceed from effect from different particles from nuclear reactions; 3) formation of local micro-segregations of impurities and "new" elements (chemical elements); 4) detection of dot aggregates of impurity elements on mass-spectra and distinction of mass-spectra in initial samples before and after experiments can also result from nuclear reactions.

A non-uniform blackening, "hot points" on the x-ray films from ion irradiated samples is the certificate of stochasticity of nuclear reactions and impossibility to receive identical effects on change of elemental and isotopic composition and heat effects. It was suggested that these local processes were fusion and fission of low energy nuclear reactions [5 - 7, 9, 10]. For experiments in electrolysis all the above processes are relevant but at more local volumes of gas bubbles on the cathode. Such processes as micro-explosions, micro-arcs occur both in electrolysis and in ion bombardment. This leads to the instability of the process.

These processes in the surface layers can result from: the pressure in micro-pores, generated during irradiation of metals by low-energy ions (of Hydrogen and Deuterium) with high-density of ion current can achieve tens and hundreds kg/cm; the delay of micro-pores and output of protons, the change of surface topography irradiated by ions, shaping of cones and other outgrowths on the cathode. It can also lead to change of electron subsystem in a crystal lattice. The local changes in electric and magnetic fields can influence nuclear processes more delicately, can serve as a trigger mechanism for these phenomena and stimulate the phenomena on the nuclear level.

Let us assume that occurrence of "new" elements and change of isotopic composition resulted only from ionic bombardment (for example, preferential sputtering of light elements). In this case maximal effects "new" elements occurrence and isotopic composition change after Hydrogen, Deuteron and Argon irradiation of samples in identical condition should be in the samples exposed to heavy Argon ions. However the maximum isotopic effects after Deuteron irradiation of Pd are observed. It was published earlier (the ICCF6). These changes were presented quantitatively in a row: Deuterium / Hydrogen / Argon with ratio $\sim (6-10) / (2-4) / 1$ by x-ray microprobe analyses [6].

CONCLUSIONS

1. The processes in GD experiments are stochastic and accompanied by formation of various defects, "hot points" with localized isotopic and elemental structure changes and are not equally reproducible. Only special structure and design could contribute to solving of the problem.
2. The charged particles emission from the samples exposed to Deuterium after GD current switch off show: the maximum value for Pd and Pd with small addition of refractory metals. The increase of current emission and its duration with decrease of residual gas pressure at discharge chamber can determine the charged particle current as P emission.
3. Tracks on X-ray films accompanying deuterium bombardment depended upon the exposed materials and parameters of process. There are more tracks for experiments with increasing new elements on the cathode surface.

The stochasticity of nuclear reactions, the impossibility to receive identical results on change of elemental and isotopic composition and heat effects testify to the necessity of the process further study. The impossibility to explain some parties of the phenomenon, to connect all verges of process and to count the complete energy balance probably will show necessity of the more profound research of this phenomenon.

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