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G.P. Glazunov, https://orcid.org/0000-0002-8895-927X
M.M. Bondarenko, https://orcid.org/0000-0001-5783-9788
O.L. Konotopskyi, https://orcid.org/0000-0002-1622-6376

National Science Center "Kharkiv Institute of Physics and Technology" of the NAS of Ukraine,
Institute of Plasma Physics, Kharkiv, Ukraine
E-mail: glazunov@ipp.kharkov.ua

Small sized ultra-pure hydrogen generator-electrolyzer

Presented by Academician of the NAS of Ukraine I. Ye. Garkusha

A laboratory-scale model of a small-sized generator-electrolyzer for ultra-pure hydrogen (purity exceeding 99.999 % by volume) was designed, tested, and investigated. Pure hydrogen is produced as a byproduct simultaneously with the production of industrial-grade hydrogen during the electrolysis process. The use of Pd-cathodes ensured their high hydrogen saturation during electrolysis and, subsequently, when heated in a vacuum, enabled the production of a flow of ultra-pure hydrogen. It had been shown that the Pd-cathode with the mass of ≈15 g absorbs up to two normal liters of hydrogen during 2—3 hours electrolysis process at 1 atmosphere pressure and 40—60 °C temperature in the one wt. % soda solution in water. Such performance is more than twice that of previous similar electrolyzer designs, as well as the data reported in the literature for the regime governed by the molecular effect. These cathodes can be used as hydrogen storage devices to supply high-purity hydrogen to various vacuum systems without the need for high-pressure hydrogen balloon. The effect of repeated hydrogen saturation of Pd-cathodes and multiple thermal cycles, including annealing the cathodes in a burning gas flame — on pure hydrogen production was investigated. The optimal electrode shapes and the distance between the anode and cathode were selected to ensure stable operation of the electrolyzer generator. The reasons of surface morphology and cathode shape changes, as the result of $\alpha \leftrightarrow \beta$ phase transition in Pd following prolonged exposure to electrolytic hydrogen are also discussed.

Keywords: *pure hydrogen, palladium, electrolysis, Pd-cathode, hydrogen saturation, degassing.*

1. Introduction. The field of ultra-pure hydrogen applications is rather extensive: it includes the semiconductor and microelectronics industries in the production of ultra-pure materials, as well as numerous laboratories conducting various experiments, including research on thermonuclear plasma. High-purity hydrogen is required in the chemical industry, chromatography, and the energy sector (as an energy source for fuel cells). High-purity hydrogen is crucial to the performance of fuel cells, since certain species can poison the catalysts even at very low concentrations [1, 2].

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The best-known method for producing ultra-high-purity hydrogen (e.g., with a purity of >99.9999 %) is a single-stage process using diffusion-catalytic membranes based on Pd/Ni and its alloys [3, 4]. The feedstock for producing pure hydrogen typically consists of commercial hydrogen (in Pd-hydrogen filters), gaseous or liquid hydrocarbons — such as methane, alcohol, gasoline, etc. — including the possibility of obtaining ultra-pure hydrogen as a byproduct of fuel combustion [5, 6]. However, the use of these methods is environmentally harmful due to CO and CO_2 emissions during the process.

The most environmentally friendly method is water electrolysis [7—10]. However, producing ultra-pure hydrogen with a purity of ≥ 99.999 % requires additional hydrogen purification processes. This leads to a significant increase in the cost of ultra-pure hydrogen.

In previous works [11, 12], the method described for ultra-pure hydrogen production as co-product simultaneously with technical hydrogen (92—94 vol. %) generation in the electrolysis process in the 1 wt. % soda solution in water. The use of Pd-cathodes provided their high hydrogen saturation during electrolysis process and then, under heating in a vacuum, to get ultra-pure hydrogen flow. Palladium is a unique metal capable of absorbing up to 900 volumes of hydrogen at atmospheric pressure and room temperature [1, 13]. The high hydrogen diffusion coefficient in this metal at moderate temperatures makes it suitable for use as a storage medium for ultra-pure hydrogen. However, palladium has essential drawback: metal can destroy after hydrogen saturation to high concentrations (more than 0.6 H/Pd) as consequence of $\alpha \leftrightarrow \beta$ phase transition [1]. Therefore, for the practical implementation of the aforementioned method, it was necessary to study the service life of palladium cathodes under prolonged exposure to electrolytic current and multi-cycle thermal loads, with the aim of improving performance metrics regarding the volume of ultra-pure hydrogen produced.

The main objective of this work was to develop a working model of a high-throughput ultrapure hydrogen electrolyzer, as well as to study the effect of repeated hydrogen saturation and multi-cycle thermal loading on the behavior of Pd-cathodes during hydrogen absorption and degassing. In addition, it was very important to evaluate the effect of changes in the shape and surface morphology of the electrodes following prolonged exposure to electrolytic hydrogen and thermal cycles on the performance of the Pd-cathode as a hydrogen storage medium.

2. Experimental setup, methods and results.

2.1. Experimental setup. A schematic view of the experimental installation (E-5 small-sized model of electrolyzer) shown in Fig. 1. It consists of a glass chamber *1* with a volume of approximately one liter, filled with electrolyte *2* (H_2O + 1 wt. % $NaHCO_3$ or H_2O + 1 wt. % Na_2CO_3). Inside chamber *1* is a tubular anode electrode *3* made of palladium (Pd) with a diameter of 3 × 1 mm and a length of 200 mm; outside the anode electrode is a tubular cathode electrode *4* made of palladium (Pd) with a diameter of 6 × 0.25 mm and a length of 200 mm. The anode and cathode are secured to the chamber lid *6* via an insulator *7* and flanged fittings *8* and are electrically connected to a power source. The other end of the palladium anode is hermetically sealed by soldering with a silver welding alloy.

The distance between anode-cathode surfaces is ≈1.2 mm and provides by spiral insulator *5*, in difference from previous model of electrolyzer E-4 [12], when the anode-cathode distance was ≈3.3 mm, which provided special fluoroplastic insulators. With the using of Pd-cathodes Ø 6 × 0.5 mm and isolator made of glass fabric net this distance decreased up to 1 mm. It was assumed that reducing the interelectrode distance would increase saturation efficiency by raising the hydrogen concentration above the surfaces of the Pd-cathodes.

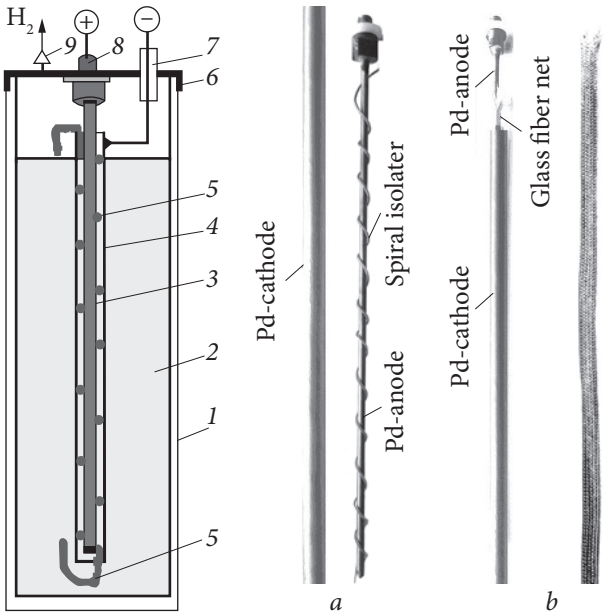

Fig. 1. Scheme of the E-5 model of the electrolyzer: *1* — chamber; *2* — electrolyte; *3* — Pd-anode; *4* — Pd-cathode; *5* — helical-like (spiral) insulator; *6* — chamber cap; *7* — seal-insulator; *8* — flange fitting; *9* — safety valve. To the right, the photos of different constructions of electrolysis module shown: *a* — with spiral, *b* — with glass fiber net (glass fabric) insulator

Hydrogen was generated through electrolysis in chamber *1*, similar to the method described in [11, 12]. Safety valve *9* ensured the removal of excess hydrogen from the chamber. The power consumption for hydrogen production was 20—30 W (the current varied between 4 and 4.5 A, and the voltage between 6 and 6.5 V). The current-voltage characteristics of the electrolysis process measured in various regimes with the different electrolytes: (H_2O + 1 wt. % $NaHCO_3$ and H_2O + 1 wt. % Na_2CO_3). It seen in Fig. 2 the electrolytic current at the same voltage for soda electrolyte is in two times lower than for soda ash electrolyte. The possible reason of such behavior could be increase of Na^+ ions in the soda ash-water electrolyte. In present experiments, low power regime with the using 1 wt. % soda solution in water electrolyte had chosen (solid curve in Fig. 2). On the other hand, the lower temperature of the Pd-cathode creates more favorable conditions for saturation, reducing the backflow of hydrogen from the sample.

Two series of experiments were conducted at different times. In the first series, Pd 99.98 % (by mass) tubes with a diameter of 6 mm, a thickness of 0.25 mm, and a length of 200 mm were used as cathodes (see Fig. 1, *a*). Three cathode samples were used: Pd-cathodes No. 1, No. 2, and No. 3 (Table). The anode was a Pd tube with a diameter of 3 mm, a thickness of 1 mm, and a

The used Pd-cathodes dimensions and weights

Cathode number	Diameter, mm	Thickness, mm	Length, mm	Weight, g
1	6	0.25	200	15.9
2	6	0.25	200	14.54
3	6	0.25	200	13.93
4	6	0.5	144	16.74
5	6	0.5	143	16.93
6	3	1	200	13.48
7	3	1	200	13.29

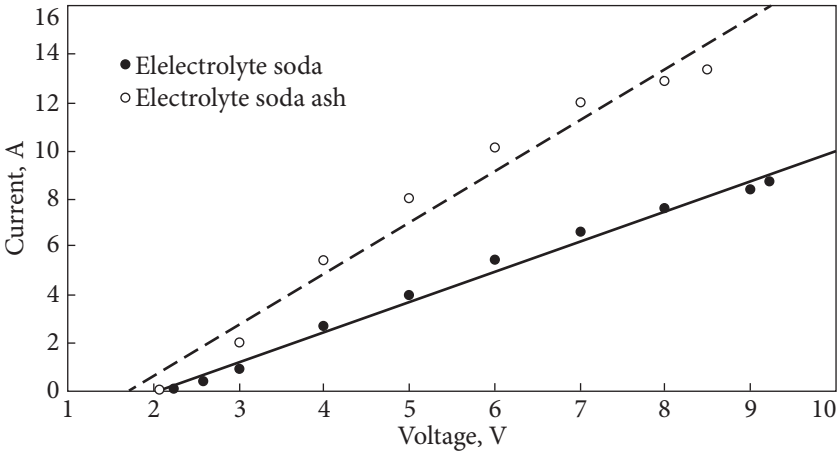

Fig. 2. The current-voltage characteristics of the electrolysis process in the E-5 electrolyzer with different electrolyte: solid curve — electrolyte (H_2O + 1 wt. % $NaHCO_3$, soda); line dotted — electrolyte (H_2O + 1 wt. % Na_2CO_3, soda ash)

length of 200 mm. The spiral isolator had placed between electrodes that specified anode-cathode distance ≈1.2 mm.

In the second series of the experiments (see Fig. 1, *b*) the glass fiber net used instead of spiral insulator between electrodes. In this case, the tube Pd-cathodes No. 4 and No. 5 used and inter-electrode distance was ≈1 mm. In this isolator configuration the experiments with castled polarity of electrodes carried out, too, i.e., Pd-anodes Ø 3 × 1 × 200 mm served as cathodes No. 6 and No. 7 (see Table). This tubular form and the availability of a flanged connection make it easy to use these cathodes as storage-and-filtering devices for supplying high-purity hydrogen to vacuum systems.

2.2. Methodic and results. Firstly, the Pd-cathodes were saturated with hydrogen in an electrolyzer for 0.1—7 h. The processes of hydrogen generation and its interaction with the palladium cathode have been considered in work [11]. Whereupon the hydrogen saturation, the electrolysis process stopped and Pd-cathode removed from the electrolysis chamber, dried and cleaned with such procedures: wiping with special fabric wetted in clean branded gasoline, drying, wiping with special fabric wetted in 96 % ethanol, drying. The sample was then weighed on a VLR-200 balance to determine the amount of absorbed hydrogen Q (capacity) using the gravimetric method. The value Q divided the mass m of Pd-cathode means ultra-pure hydrogen specific productivity of electrolyzer, $q = Q/m$ (Ncm^3/g Pd).

After measurement of absorbed hydrogen quantity, the sample heated in the gas oven (300—400 °C) or in the flame of gas combustion (≈700 °C), cleaned, weighted and used for next cycle of saturation and degassing. The scheme of Pd-cathodes heating (annealing) in the flame of gas combustion is clear in Fig. 3. We used the universal stove model No. 06630 (USA) (Fig. 3). The possibility existed to move a sample (cathode) in horizontal plane inputting different parts of a sample into high temperature zone of the flame. Earlier, in work [6] such a stove was used to heat diffusion membranes made of Pd/Ni tubes from a pure hydrogen generator to temperatures of 700—800 °C. It should be noted that during heating (degassing) of highly saturated Pd-cathodes in a flame, hydrogen evolution from the sample leads to the formation of a flame tongue at the end of the tube. On the outer surface of the tube, the escaping gas (hydrogen) caused an increase in the intensity of the gas flame and, consequently, a rise in the sample temperature.

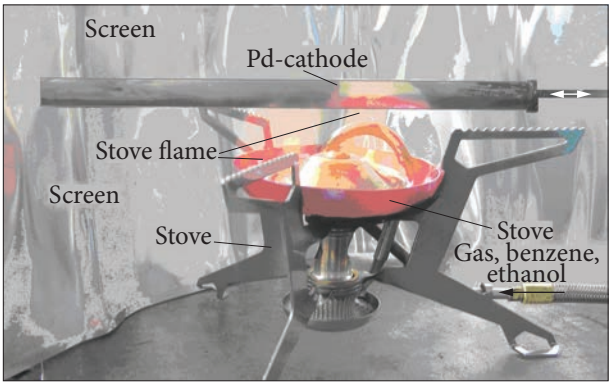

Fig. 3. Stove for cathodes degassing

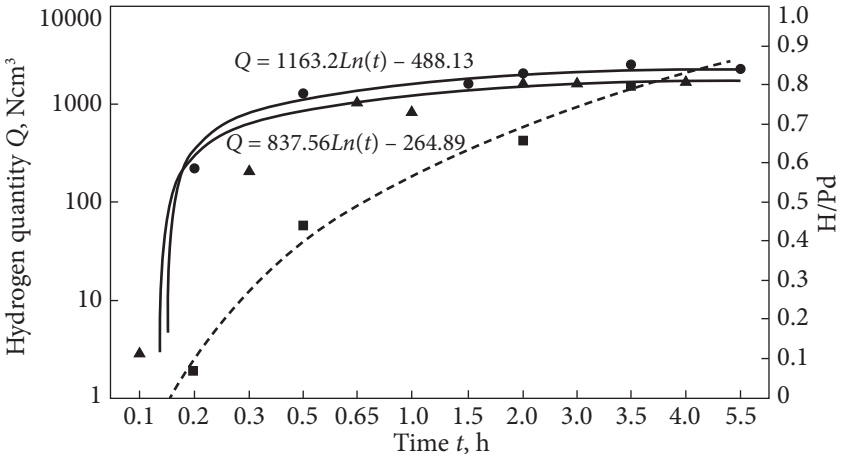

Fig. 4. Hydrogen quantity Q in Pd-cathode No. 1 (triangles), and No. 2 (circles), in dependence on saturation time in the E-5 electrolyzer. Square points means hydrogen concentration H/Pd in Pd-cathode No. 2

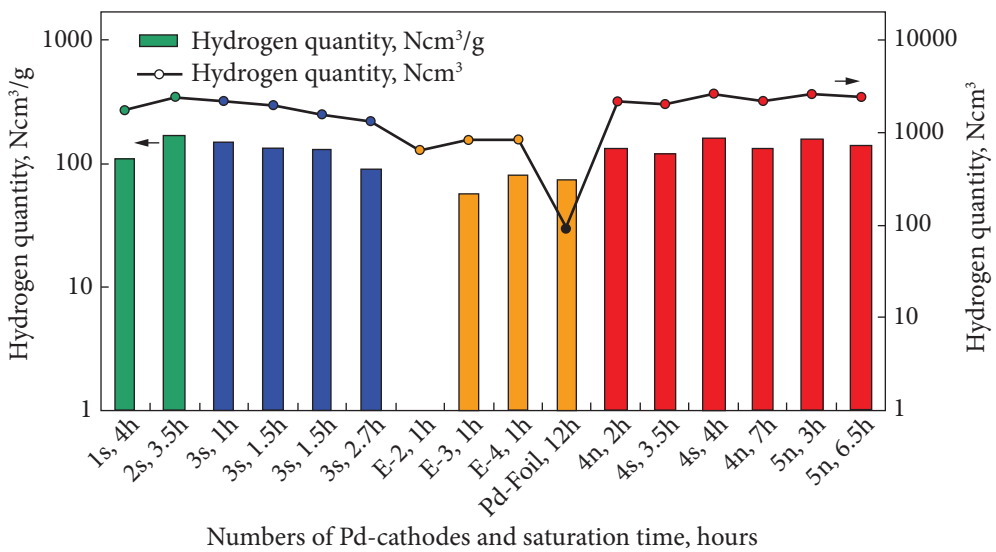

Fig.5. Hydrogen volumetric quantity in Pd-cathodes in dependence on saturation time in the E-5 electrolyzer. Index (s) means using spiral isolator, index (n) means using glass fiber net isolator

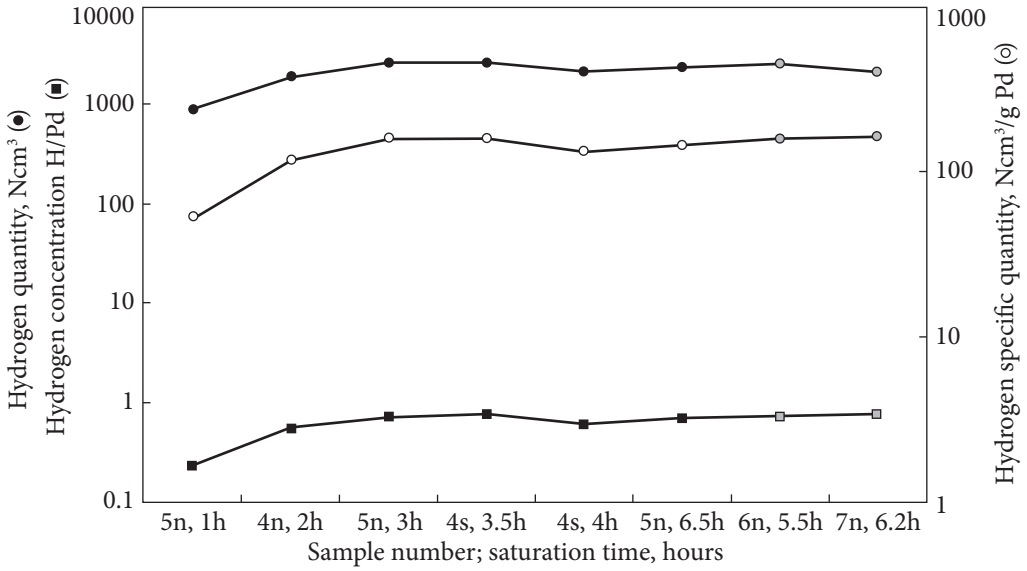

Fig. 6. Hydrogen volumetric quantity of hydrogen absorbed in Pd-cathodes No. 4 and No. 5 after long time saturation. Last two points are for samples No. 6 and No. 7 — when Pd-anodes Ø 3 × 1 × 200 mm served as cathodes

In the Fig. 4, hydrogen volumetric quantity Q in Pd-cathodes No. 1 and No. 2 and hydrogen concentration H/Pd in the cathode No. 2 are shown in dependence on saturation time t in the E-5 electrolyzer ($P \approx 1$ atm, temperature of 20—50 °C, ion current ≈4 A, voltage ≈6.5 V).

Fig. 5 presents the results of a study on the effect of repeated hydrogen saturation and multi-cycle thermal treatment on the hydrogen absorption characteristics of Pd-cathodes. The samples No. 1 and No. 2 (green points) exposed to electrolytic hydrogen long time. Spiral isolators used for both samples. Cathode No. 3 (blue points) four times saturated up to high hydrogen concentration. After saturation, it degassed during one hour heating in the gas oven at the temperature of T = 300—400 °C. In addition, Fig. 5 (orange points) shows data taken from [11, 12], obtained using E-3 and E-4 electrolyzers, as well as for the molecular drive mode, in which a palladium foil was saturated with molecular hydrogen for 12 h.

In Fig. 6 (see also red points in Fig. 5), hydrogen quantity absorbed in Pd-cathodes No. 4 and No. 5, and hydrogen concentration H/Pd in dependence on saturation time and thermal cycling presented. Both spiral and (primarily) fiberglass mesh insulators were used. After each saturation procedure, the samples were degassed in a gas burner flame at a temperature of 600—700 °C for approximately 10 min. The total time of cathodes exposure to electrolysis current amounts to 16.5 h and 9.5 h for samples No. 4 and No. 5, respectively. Last two points in Fig. 6 are for samples No. 6 and No. 7 — when polarity of electrodes was castled, i.e., Pd-anodes Ø 3 × 1 × 200 mm served as cathodes.

2.3. Changes in the morphology and surface shape of Pd-cathodes, illustrated in Figs. 7, 8, 9, and 10.

3. Discussion. In Fig. 4 seen, that the main hydrogen quantity sorbs in the first 2 h of saturation and its value corresponds to logarithmic dependences. In this time, hydrogen concentration in Pd achieves H/Pd ≈ 0.65, i.e., palladium transits into β-phase [1, 13]. At further increase of

Fig. 7. Photos (×4) of screw-like surface of Pd-cathode Ø 6 × 0.25 mm after hydrogen saturation up to 0.8 H/Pd in the E-5 electrolyze module with spiral isolator

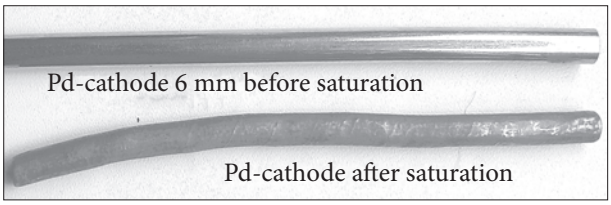

Fig. 8. Photos Pd-cathode No. 7, Ø 6 × 0.5 mm, before saturation and, after long time saturation and annealing

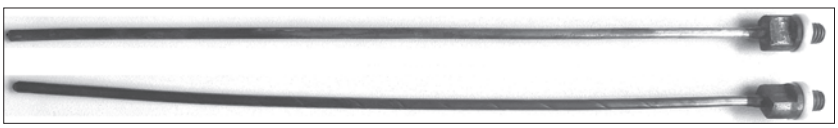

Fig.9. Photos Pd-anode No. 7, Ø 3 × 0.5 mm after long timework as anode, and (below) as cathode after long time hydrogen saturation

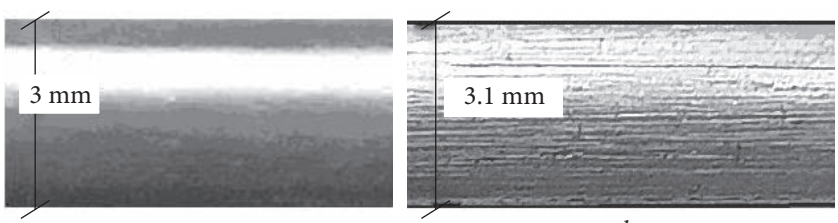

Fig. 10. Surface morphology of Pd-cathode Ø 3 mm before (*a*) and after (*b*) hydrogen saturation to ≈0.5 H/Pd in molecular driven regime

saturation time, hydrogen concentration amounts to H/Pd ≈ 0.8 but such regime turns out to be economically unprofitable due to a strong increase in electricity consumption per unit of production. Moreover, a slight increase in temperature during the prolonged electrolysis process may cause hydrogen to flow back from the metal.

Since electrolysis in the present study takes place at relatively low temperatures (40—60 °C), according to the literature [1, 13], palladium in the molecular drive mode at room temperature can become saturated with up to 900 volumes of hydrogen. The weight of Pd in the cathode No. 2 is 14.54 g. It corresponds to 1.21 cm^3 Pd, i.e. about ≈1000 Ncm^3 pure hydrogen could be absorbed. The experiment (see Figs. 4—6) had shown that in some cases of electrolytic saturation this quantity of ultra-pure hydrogen can be twice as much (≈2000 Ncm^3 H_2, that corresponds to ≈1000 at. hydrogen pressure if to transform absorbed hydrogen into molecular gas phase in 1 cm^3 volume). On average, the data obtained in previous studies [11, 12] on the hydrogen saturation of Pd-cathodes in other E-3 and E-4 electrolyzer designs, as well as Pd foil under molecular drive conditions (orange dots in Fig. 5), are significantly lower (≈70 Ncm^3/g) compared to the data from this study

(over 100 Ncm^3/g). It should be noted that the stability of the saturation characteristics for samples No. 4—7, annealed in a combustion gas flame (700—800 °C, see Fig. 6), is higher than that of samples No. 1—3, heated in a gas furnace at temperatures of 300—400 °C (see Fig. 5).

Strong macro-changes of Pd-cathodes shape and surface are observed which caused by hydrogen saturation to high concentration (see Fig. 7, 8). As screw-like surface observed for Pd-cathode Ø 6 × 0.25 mm after hydrogen saturation up to 0.6—0.8 H/Pd in the E-5 electrolyze module with spiral isolator. Most probably, the places, where isolator contacts with the internal surface of Pd-cathode, isolated from an electrolyte and therefore weakly saturates with hydrogen. On the accessible part of sample surface, hydrogen absorbs to high concentration and $\alpha \leftrightarrow \beta$ transition occurs. At temperatures of 40—60 °C, the hydrogen diffusion coefficient is low, and a strong concentration gradient—and, consequently, a stress gradient — arises in the metal lattice, leading to macroscopic changes in the surface (see Fig. 7) and shape (see Fig. 8) of the Pd-cathodes. Note that the screw-shaped surface damage to the Pd-cathodes was not observed in experiments using a glass fiber mesh insulator instead of a spiral-shaped one. Strong change of cathode shape, it becomes curve (see Fig. 8), explains similar to that in work [11] by the phase transition $\alpha \leftrightarrow \beta$ in palladium, when the lattice parameter changes significantly and, as the result, the arising gradient of elastic stresses leads to a form strong change. High-temperature annealing did not restore the sample's shape or mechanical alignment, which are necessary for reusing the sample. For Pd cathodes measuring 3 × 1 mm, this effect is less pronounced (see Fig. 9).

In addition, there are the damages in the form of tracks on the cathode surface visually observed. Texture of surface layer after producing, and above-mentioned $\alpha \leftrightarrow \beta$ transition in Pd could be the reasons. Similar damages (see Fig. 10) observed on Pd-cathode surface after hydrogen saturation to ≈0.5 H/Pd in molecular driven regime in the work [11]. Note such tracks did not observe in the places of isolator contact with metal surface (see Fig. 7).

Despite the significant changes occurring on the surface of the Pd-cathodes and in their structure under the influence of an electrolytic current over an extended period of time, the experimental data presented above indicate that the electrolysis module of this miniature generator-electrolyzer operates stably. The best results were obtained when the distance between the anode and cathode was ≈1 mm and a fiberglass mesh insulator was used between the "thick" electrodes (0.5 and 1 mm thick). Such cathodes could use as storage-accumulators, similar as Pd-filters, for extra pure hydrogen inflow to high vacuum systems. However, it was subsequently possible to resolve the issues of uniform saturation across the entire surface of the cathodes and hydrogen retention in palladium during long-term storage, which made it possible to increase the durability of Pd-cathodes as storage media for ultra-pure hydrogen.

Conclusions. The new compact model of an ultrahigh-purity hydrogen generator-electrolyzer (purity greater than 99.999 % by volume) has been developed, tested, and studied. The current-voltage characteristics of the electrolysis process were measured under various operating conditions using different electrolytes: (H_2O + 1 wt. % $NaHCO_3$ and H_2O + 1 wt. % Na_2CO_3). It has been shown that, at the same voltage, the electrolytic current for the soda-based electrolyte is half that of the calcined soda-based electrolyte.

The amount of hydrogen, absorbed by Pd-cathodes (productivity), measured. It had been shown that the Pd-cathode with the mass of ≈15 g absorbs up to two normal liters of hydrogen during 2—3 h electrolysis process at 1 atmosphere pressure and 40—60 °C temperature in the one wt. % soda solution in water. Such performance is more than twice that of previous similar elec-

trolyzer designs, as well as the data reported in the literature for a regime driven by the molecular effect. One can use such cathodes as a hydrogen generator and storage device to supply high-purity hydrogen to various high-vacuum systems, including plasma devices, without the need for a high-pressure hydrogen cylinder.

The investigations of the influence of many times repeated hydrogen saturation of Pd-cathodes and thermal multi-cycling exposure, including cathodes baking in the flame of gas combustion, on pure hydrogen productivity carried out. The proposed miniature model of an ultra-pure hydrogen generator-electrolyzer was demonstrated to be functional. The optimal distance between the anode and cathode (≈1 mm) and the use of a glass fiber mesh insulator between the "thick" electrodes ensured stable operation of the electrolysis module in the presented miniature generator-electrolyzer.

The impact of long time interaction with electrolytic hydrogen and thermal multi-cycling exposure on surface morphology and Pd-cathode shape changes as the result of $\alpha \leftrightarrow \beta$ phase transition in Pd examined. Significant macroscopic changes in the shape (curvature) and surface (helical and track-like damage) of palladium cathodes are observed, caused by hydrogen saturation to high concentrations and a strong concentration gradient, which, in turn, leads to the formation of a stress gradient in the metal. In the future, it will be necessary to ensure uniform saturation across the entire surface of the cathodes and to address the issue of hydrogen retention in palladium during long-term storage in order to improve the durability of Pd-cathodes as ultra-pure hydrogen storage devices.

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Г.П. Глазунов, https://orcid.org/0000-0002-8895-927X
М.М. Бондаренко, https://orcid.org/0000-0001-5783-9788
О.Л. Конотопський, https://orcid.org/0000-0002-1622-6376

Національний науковий центр "Харківський фізико-технічний інститут" НАН України,
Інститут фізики плазми, Харків, Україна
E-mail: glazunov@ipp.kharkov.ua

МАЛОГАБАРИТНИЙ ЕЛЕКТРОЛІЗЕР-ГЕНЕРАТОР НАДЧИСТОГО ВОДНЮ

Розроблено, випробувано та досліджено лабораторну малогабаритну модель генератора-електролізера надчистого водню (чистота понад 99,999 об. %). Чистий водень утворюється як побічний продукт одночасно з виробництвом технічного водню в процесі електролізу. Використання Pd-катодів забезпечує їх високе насичення воднем під час електролізу, а потім, під час нагрівання у вакуумі, — отримання потоку надчистого водню. Показано, що Pd-катод масою ≈15 г поглинає до двох нормальних літрів водню протягом 2—3 год процесу електролізу за тиску 1 атм та температури 40—60 °C в 1 мас. %-му розчині соди у воді. Така продуктивність більш ніж удвічі вища, ніж у попередніх конструкцій аналогічних електролізерів, а також за молекулярно-керованого режиму, про який повідомляється в літературі. Такі катоди можна використовувати як генератори-накопичувачі водню для подачі чистого водню в різні вакуумні системи без водневого балона високого тиску. Було досліджено вплив багаторазового насичення воднем Pd-катодів та багатоциклового термічного впливу, зокрема нагріву катодів у полум'ї горіння газу, на продуктивність чистого водню. Для забезпечення стабільної роботи генератора-електролізера було визначено оптимальні форму електродів та відстань між анодом і катодом. Також розглянуто причини змін морфології поверхні та форми катодів внаслідок фазового переходу $\alpha \leftrightarrow \beta$ у Pd після тривалої взаємодії з електролітичним воднем.

Ключові слова: *чистий водень, паладій, електроліз, Pd-катод, насичення воднем, дегазація.*