https://doi.org/10.46813/2025-159-140

ON THE POSSIBLE METHOD OF ULTRA-PURE HYDROGEN GENERATING

G.P. Glazunov, M.M. Bondarenko, O.L. Konotopskiy
National Science Center "Kharkiv Institute of Physics and Technology",
Institute of Plasma Physics, Kharkiv, Ukraine
E-mail: glazunov@ipp.kharkov.ua

The possible method is described for ultra-pure hydrogen production (purity better, then 99.999 vol.%). Pure hydrogen produced as co-product simultaneously with technical hydrogen generation in the electrolysis process. The use of Pd-cathodes provides their high hydrogen saturation during electrolysis process and then, under heating in a vacuum, to get ultra-pure hydrogen flow. It had been shown that the Pd-cathode with the mass of ≈ 14.5 g can absorb up to one liter of hydrogen during 1 h electrolysis process in the 1 wt.% soda solution in water. The rate of hydrogen saturation under electrolysis process is in about six times higher than for molecular hydrogen driven experiments. Activation energy of degassing process, calculated from the temperature dependence of hydrogen out gassing rate on the temperature, is to be ≈ 40 kJ/mole. Mass-spectrometric measurements confirmed high purity generated hydrogen. Physical-chemical mechanisms analyzed to explain the obtained results.

PACS: 52.40.Hf

INTRODUCTION

Ultra-pure hydrogen widely is applied in chemical and electronic industries, gas chromatography, in the different researches, which carry out in many laboratories, etc. Fuel cells also appear to be a broad area of consumption for ultra-pure hydrogen in the future.

There are some methods to produce ultra-pure hydrogen (purity better, than 99.999 vol.%) from technical hydrogen or hydrocarbons, using various technologies [1–11].

In the membrane method [5–9] the fact is used that through some metals and alloys (membranes from nickel, palladium, Pd-alloys, etc.) at a temperature of 400…700 ºC only hydrogen penetrates, due to its very high diffusion coefficient. The raw materials for the production of pure hydrogen are usually used by gas-shaped or liquid hydrocarbons, for example, methane, alcohol, gasoline, etc., including the possibility to produce ultra-pure hydrogen as co-product during fuel combustion [7–9]. However, the disadvantage of these methods is emission of CO and CO_2 during the technology process. Therefore it is possible to poison the surface of membranes with carbon ($CO+H_2=H_2O+C$), which leads to instability and a decrease in process productivity.

The most suitable method is the electrolysis of water [1–4]. Indeed, by electrolysis we get hydrogen and oxygen ($2H_2O=2H_2+O_2$), and then, by burning hydrogen as a fuel ($2H_2+O_2=2H_2O+572.5$ kJ), we get water again. This method is the most ecology clean but, to generate ultra-pure hydrogen, additional technological process and hydrogen purifying systems are required. This leads to a significant increase the cost of ultra-pure hydrogen. In the work [10] the method was suggested for ultra-pure hydrogen production during electrolysis process with the using of cathode made as diffusion-catalytic membrane from the metal with high hydrogen permeability (Ni, Pd). The experiments showed the principal possibility to generate pure hydrogen by this method, but low productivity observed. The estimations showed, to obtain essential hydrogen flows through Ni membrane, necessary to increase its temperature during electrolysis, at least, up to 350…700 °C. This would strongly increase the cost of produced ultra-pure hydrogen.

In our previous works [11, 12] we observed that during electrolysis process with the using of Pd-cathodes, the last saturated by hydrogen up to high concentration. It has been shown, that at an electrolyte temperature of ≈ 50 °C and the pressure in the electrolyze chamber of 1 atm, the generated gas is hydrogen of 92…94 volume percentages without separating procedure of hydrogen from oxygen. At the same time, hydrogen, desorbed in a vacuum from Pd-cathode after electrolysis process, is ultra-pure (better than 99.999 vol.%). Such cathodes made of the metals/alloys with high capacity on hydrogen (e.g., Pd, Pd-alloys, Ni, Ti, Zr-Al, Zr-Ni, etc.) could be use as storages (accumulators) for ultra-pure hydrogen inflow into different high vacuum systems, including plasma devices.

However, to realize this for practice using, it needed to investigate in details the processes of hydrogen interaction with Pd-cathode surface, and its behavior in the metal volume in the conditions of electrolysis process. The study of lifetime of cathode and its productivity under long time influence with electrolyte, creation of a work model of ultra-pure hydrogen generator-electrolyzer, are also important physical-technical questions.

The main task of this work was to study in detail physical-chemical mechanisms of hydrogen saturation of Pd-cathodes during electrolysis process, to measure its out gassing rate during Pd-cathodes heating in a vacuum, to determine the activation energy value of degassing process and to estimate hydrogen capacity of the Pd-cathodes in different regimes.

1. EXPERIMENTAL AND RESULTS

The electrolysis setup used in present experiments was similar to that in work [11]. The scheme of the electrolysis module and the photo of Pd-cathode shown in Fig. 1. There is only one difference being that polarity of electrodes was changed. Now, on contrary with the scheme of experiment in [11], cathode was Pd tube 1, with a diameter of 3 mm, a thickness of 1 mm and length of 200 mm, mechanically fixed from the one end to flange of electrolysis chamber through a flange fitting 5 with good electrical contact. Another end of the tube hermetically sealed by brazed with silver welding alloy. Such cathode construction allows to easily demounting it for examinations. The anode 2 was Pd tube with a diameter of 10 mm, a thickness of 0.2 mm and a length of 200 mm, mechanically fixed to cathode through fluoroplastic insulators 3. The anode-cathode distance was ≈ 3.3 mm. To exclude an influence of the walls of electrolysis chamber 6 made of stainless steel 12Cr18Ni10Ti, electrodes placed in the plastic container 7 with volume of 350 ml filled up the 1 wt.% solution of soda in water electrolyte. The square of cathode, facing to electrolyte, was $F_c \approx 14$ cm^2, and the mass is ≈ 14.5 g. The square of internal surface of anode, facing to electrolyte $F_a \approx 45$ cm^2, and the mass is ≈ 13.5 g. Thus, the masses of the anode and cathode are approximately equal, and the surface area facing the electrolyte for the anode is approximately three times greater than that of the cathode. It was necessary to establish to what extent the ratio of the anode and cathode areas and the change in polarity affect the current-voltage characteristics and the amount of hydrogen absorbed in the cathode (productivity).

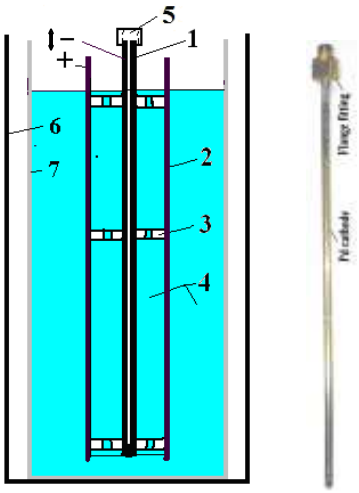

Fig. 1. Scheme of the electrolysis module: 1 – cathode; 2 – anode; 3 – fluoroplastic insulators; 4 – electrolyte; 5 – flange fitting; 6 – wall of chamber; 7 – plastic container; to the right-photo of Pd-cathode

An electric potential supplied to both anode and cathode from power supply, which was a rectifier on two V200 diodes, connected through current transformer to the autotransformer. Hydrogen produced by electrolysis similar as in works [11, 12]. The power consumption for hydrogen generation was up to 70...80 W.

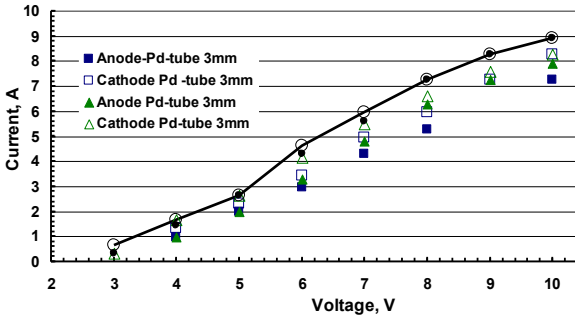

Fig. 2. The current-voltage characteristics of the electrolysis process: white square and triangles the cathode was Pd tube of 3 mm diameter (right polarity); dark square and triangle points – the cathode was Pd tube of 10 mm diameter (inverse polarity). The curve (circle points) is for the experiment, when plastic container was absent

The current-voltage characteristics measured during electrolysis process presented in Fig. 2. Two series of experiments in the different time carried out. In one case cathode was Pd tube with a diameter of 3 mm, a thickness of 1 mm and length of 200 mm (white points in Fig. 2, so called right polarity regime). In another, cathode was Pd tube with a diameter of 10 mm, a thickness of 0.2 mm and a length of 200 mm (dark points in Fig. 2, inverse polarity regime). It seen, the current-voltage characteristics are similar to each other, despite the fact that cathode surface facing to electrolyte changed in three times. The experiments without of the plastic container carried out, too. There not observed strong influence of chamber walls or plastic container on the electrolysis characteristics.

After saturation of the cathode with hydrogen for 0.5…1 h, the electrolysis process stopped and Pd-cathode removed from the electrolysis chamber, dried and, before placing in the vacuum chamber of the stand, it was cleaned with such procedures: wiping with special fabric wetted in clean branded gasoline, drying, wiping with special fabric wetted in 96% ethanol, drying. The cathode photos before, and after hydrogen saturation, presented in Figs. 3a, 3b.

Then, the sample weighted with the VLR-200 balance scales, and placed into the GAS device high vacuum chamber. The electrolysis chamber connected to the inlet system of the high-vacuum installation GAS [12] that allows, using mass spectrometer, to determine the composition of the mixture of gases formed during the electrolysis and degassing. After pumping to a pressure of ~ $5\cdot10^{-7}$…$5\cdot10^{-6}$ Torr, measurements carried out of mass-specter, and the dependences of the hydrogen out gassing rate on the temperature in the range of 200...650 °C using the methods similar as in the work [11]. Processing of the spectral instrumental data allows saying that at the electrolyte temperature of ≈ 50…60 °C and the pressure in the electrolyze chamber of 1 atm, the generated gas is hydrogen of 92 vol.%. Nevertheless, the gas, desorbed from Pd-cathode, is ultra-pure (better than 99.999 vol.%) hydrogen.

The samples (cathodes) heated by the direct current up to temperatures of 200…650 ºC and, simultaneously, the pressure time dependence and mass-specter of desorbed gases measured. Apparatus curves processing gives the data for estimation of hydrogen amount desorbed from a sample due to the equation $Q \cdot t = (P\text{-}P_0) \cdot S \cdot t$, where Q is out gassing rate; P – maximum pressure; P_0 – initial pressure; S – pumping speed; t – time. It taken into account, that the sensitivity of the mass spectrometer for hydrogen is approximately two times lower than for air. For the pressure measurement with a gauge PMI-10, this coefficient is four.

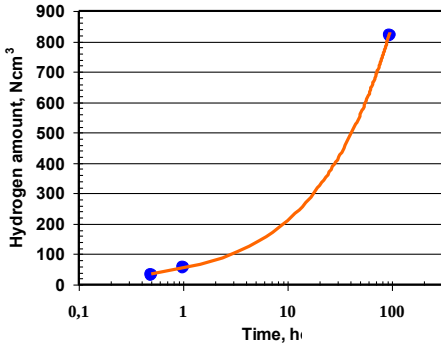

Fig. 3a. Amount of hydrogen, absorbed in Pd-cathode, v/s the time (hydrogen molecular gas driven regime)

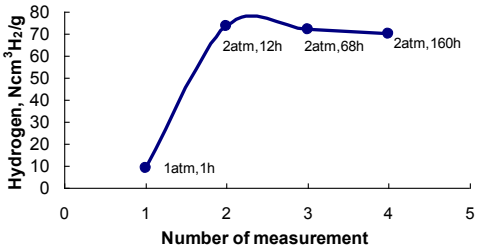

Fig. 3b. Hydrogen amount absorbed in Pd foils Ø 25x0.3 mm v/s time of saturation

The measurements showed the hydrogen amount, desorbed from the cathode (weight of ≈ 14.5 g), as 840 Ncm^3 (≈ 58 Ncm^3/g). The amount of hydrogen, measured by weighting of cathodes before and after saturation in molecular hydrogen atmosphere at the pressure of 2 atm during 12…94 h, was to be ≈ 850 Ncm^3H_2 (≈ 58.5 Ncm^3/g, see Fig. 3a). In Fig. 3b presented the data on hydrogen saturation of Pd foils with the diameter of 25x0.3 mm in different regimes. At the low time and pressure (1 h, 1 atm) amount of absorbed hydrogen was only ≈ 10 Ncm^3/g. When pressure was 2 atm and time changed in the range of 12…160 h, amount of absorbed hydrogen was ~ 70 Ncm^3/g. Moreover, with the increasing time of saturation in the range 12…160 h an amount of absorbed hydrogen (productivity) decreased. From these data, we can say that hydrogen saturation rate in electrolysis process essentially higher (about six times) than that in molecular hydrogen driven regime. If to neglect hydrogen amount absorbed from molecular phase, and to take into account Pd-cathode surface facing to electrolyte only, we will get ≈ 74 Ncm^3/g. This is near to data for Pd foils saturated during about 12 h (see Fig. 3b).

The value of activation energy E of out gassing process was calculated using temperature dependence of hydrogen out gassing rate (Fig. 5) similar as in work [11] and determined to be 40 kJ/mole.

2. DISCUSSION

2.1. HYDROGEN SATURATION OF Pd-CATHODE

The analysis of the data shown in Fig. 2, allows saying, that the maximum power inputted in the electrolysis process was ≈ 80 W. This power leads to electrolyte heating during experiment (≈ 1 h) up to the temperature of 50…60 °C. According the first law of Faraday: $m=k \cdot I \cdot t$ [13], where m – mass of generated gas; k – electrochemical equivalent; I – current; t – time, the mass of generated gases is proportional to current value.

Let us estimate the real hydrogen ions flow to cathode. In the experiments we used low concentration of electrolytic matter, ≈ 1 wt.%, therefore, for simplicity, analyze the simple case of water decomposition: $H_2O \leftrightarrow 4H^+ + 2O^- \leftrightarrow 2H_2 + O_2$. The number of hydrogen ions is twice as large as the number of negative oxygen ions. Therefore, we can assume that the ion current of protons to the cathode is ≈ 0.6I, where I is the measured total current. The energy of ions is low, so we can neglect an erosion effect. The real situation during electrolysis is more complete. Besides of protons the electrolyte facing surface of Pd-cathodes can interact with negative ions H^-, atoms H, molecular hydrogen H_2, $H_2^{-,+}$. The ion current consists from not only positive protons and negative oxygen ions, as the result of water decomposition, but includes ions of electrolytic matter ($NaHCO_3$) components.

At current of 8 A, we get a fluency $F = 6.25 \cdot 10^{18} \cdot 0.6 I \cdot t = 1.1 \cdot 10^{23}$ protons or $5.5 \cdot 10^{22}$ molecular H_2, where $I = 8$ A and $t = 1$ h = 3600 s. Therefore the maximum amount of electrolytic produced hydrogen $N = 5.5 \cdot 10^{22}$/L ≈ 2000 Ncm^3 = 2l, where $L = 2.7 \cdot 10^{19}$ – number of molecules in the gas volume of the 1 cm^3 (Loshmidt's number). Really [14] one volume of Pd can absorb up to 800…900 volumes of H_2 (i.e., 100 g of metal can be the storage for ≈ 7 l of ultra-pure hydrogen at atmosphere pressure).

In our case one Pd-cathode with mass 14.5 g (volume = 1.2 cm^3) can absorb during 1h electrolysis process in, above mentioned, regime 840 Ncm^3 ≈ 0.8 liter of H_2 (58 Ncm^3/g). In many cases, it is enough to carry out the experiments with the using of ultra-pure hydrogen. Note that the use of a hydrogen-saturated cathode is, in some sense, the method similar to the operation of Pd filters or the hydrogen input from getter [9]. Estimation shows, e.g., for the work of DSM-1 plasma device [15, 16] in the regime with pumping speed of 500 l/s and the pressure of 10^{-3} Torr in measurement vacuum chamber it is enough the hydrogen flow $Q = 0.3…0.5$ Torr l/s ≈ 0.5 cm^3/s from one Pd-cathode. If necessary, the number of Pd cathodes or their mass can be increased. Note, that only some part of hydrogen, produced during electrolysis, can be used for cathode saturation (≈ 14% in [11]). The main amount of produced hydrogen one can use in another technological process, e.g., for fuel or explosive

production. It will provide low prime cost of produced ultra-pure hydrogen. An additional advantage of this method is an absence of a high-pressure hydrogen balloon, which, in some cases, cannot be use for safety reasons.

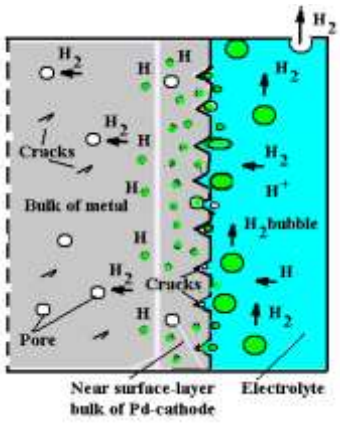

Fig. 4. Hydrogen bubbles forming on the Pd-cathode surface

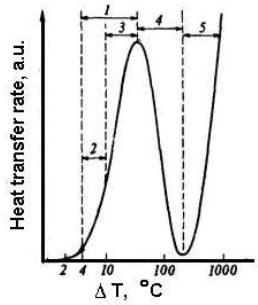

Fig. 5. The curve of water boiling: 1 – nucleate boiling; 2 – isolated bubbles; 3 – columns and "pieces" of steam; 4 – transitional boiling regime; 5 – film boiling. (The abscissa axis shows how much the surface temperature is above the boiling point of water)

Part of hydrogen atoms (protons), already dissolved in the metal, diffuses to a surface, recombine into the molecules and together with electrolytic hydrogen accumulates near sorption centers (mainly defects of metal – cracks, micro cracks, vacancies, dislocations, grain boundaries, etc.). When hydrogen amount increases, hydrogen gas bubbles forms near the cathode surface Fig. 4. The bubbles grow, break away from the cathode surface and float upward, releasing hydrogen on the electrolyte surface. With an increase the intensity of hydrogen generation (increase the potential between the electrodes, and the electrolyte temperature), the bubbles can form a continuous coating of the cathode. Since the heat transfer from the metal surface directly to the liquid is much higher than through the vapor layer, the heat transfer from the cathode to the electrolyte is sharply reduced (so-called Leidenfrost effect). In the Fig. 5, the heat transfer rate dependence on the difference between the metal surface temperature and water temperature, it accords to point of the minimum of heat transfer. The electrolysis mode, which obeys Faraday's laws (Faradaic regime), can transit to the plasma discharge regime [17–19] and strongly to increase the cathode temperature, causing electrolyte boiling. As for the production of hydrogen in this mode, it shown in [19], it turns out to be economically unprofitable due to a strong increase in electricity consumption per unit of production.

Since the electrolysis in the present work was carried out at rather low temperatures (50…60 °C), a transition of palladium from α-phase–to–β-phase is possible, when the lattice parameter changes noticeably, from 3.89 to 4.025 Å [20].

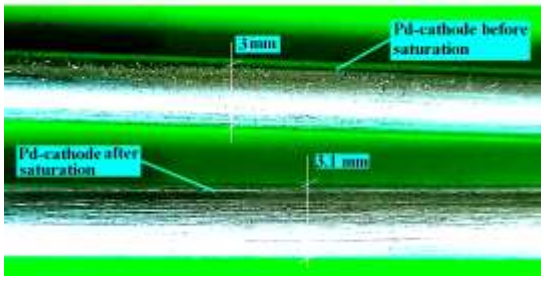

Fig. 6. Photo of Pd-cathode before and after hydrogen saturation

In our case, after saturation up to H/Pd ≈ 0.5 in the hydrogen molecular driven regime, the cathode diameter increased from 3 to ≈ 3.1 mm (Fig. 6), after saturation up to H/Pd ≈ 0.5. It means relative change of volume $\Delta V/V \approx 0.03$ that is in about three times lower than in the literature data [20]. There are the damages in the shape of longitudinal tracks on the cathode surface visually observed (see Fig. 6). Perhaps, texture of surface layer after producing, and above-mentioned α↔β transition in Pd could be the reasons. Nevertheless, additional studies, with multiple hydrogen saturation of the cathodes, and thermal cycling, are required to draw conclusions.

Despite the significant number of works devoted to the study of the hydrogen-palladium system [20], there is insufficient clarity on the state of hydrogen in the lattice of this metal. One of the possible states, namely, lattice gas plasma, discussed in [21–24]. We first supposed that the palladium lattice should play the role of an original ionizer and trap and then we considered the hydrogen plasma characteristics without taking into account the plasma-lattice interaction. Figs. 7, 8 show the phase diagrams of hydrogen in the region of real hydrogen plasma in the n-T plane, taken from [21–24].

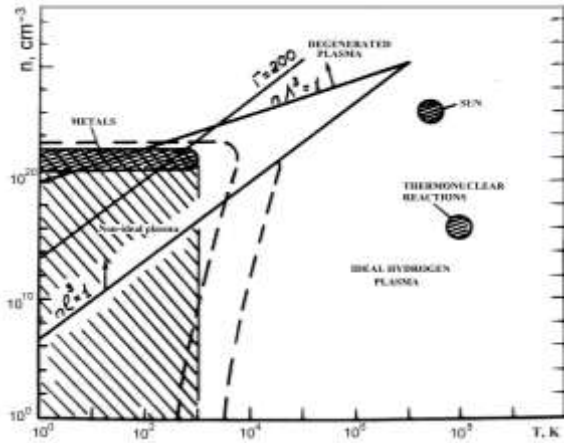

Fig. 7. Fields of the different states of hydrogen plasma on density-temperature coordinate plane [22]

In a general case the hydrogen plasma in metalcontains different particles, such as H^+(proton), e

(electron), H (atom), H^-, H_2^+, H_2. For the concentrations $n > 10^{18}$ cm^{-3} and temperature more than ≈1 K there will be only protons (or D^+, T^+) and electrons in plasma, i.e. the hydrogen plasma in metal is fully ionized.

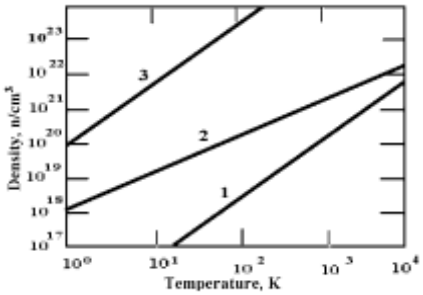

Fig. 8. Electron degeneracy line: 1 – ($n_eL_e^3$=1); 2 – Mott line (r_D=0.84r_B); 3 – proton degeneracy line ($n_pL_p^3$) [24]

Really, as it is seen in hydrogen phase diagram in Fig. 8, the Mott criterion [21] ($n_p = 1.2\cdot10^{18}\,T$(K); $r_D = (kT/4\pi e^2\cdot n_p)^{1/2} = 0.84r_B$, where n_p and n_c – proton concentration and critical density, accordingly; r_D and r_B – Debye and Bor radiuses, accordingly), which determines the hydrogen metal plasma transition to stripped plasma state, is released at above mentioned parameters (see curve 2, Fig. 8). At 1 atm the hydrogen pressure over palladium surface the temperature of Mott transition is 520 K that is near of 565 K critical temperature of gas-liquid type ($\alpha \leftrightarrow \beta$) phase transition in palladium [20]. The electron component of plasma with the indicated parameters is degenerated, and the condition of non-degeneracy of protons does not look strong enough. Indeed, the non-degeneracy parameter: $n\lambda^3 = 1/n\,(2\pi mkT/A^2)^{3/2} \gg 1$*, for hydrogen in palladium is equal ≈ 15 if $n=10^{22}$ cm^{-3} and T=300 K. Thus, when considering the proton gas in palladium from the plasma standpoint, it turns out to be strongly non-ideal plasma, close to degeneracy. As shown in [25], in a strongly non-ideal plasma, quantum effects can appear themselves before degeneracy begins. Therefore, under certain conditions, the possibility of changing the energy spectrum of such plasma could realize which will lead to the appearance of interesting phenomena and effects: phase transitions, the emergence of long-range interactions between protons, through lattice phonons, etc. A number of experimentally observed phenomena, superconductivity of β-phase of Pd, anomalies of thermal capacity at 55 K, the disappearance of paramagnets, the gas-liquid phase transition [20], shows of the quantum nature of hydrogen behavior in Pd.

2.2. HYDROGEN OUT GASSING FROM Pd-CATHODE

Hydrogen release from the palladium volume occurs through a series of parallel reactions in successive stages: $H_{me}(H^+ + e^-) \leftrightarrow \varsigma_1 \leftrightarrow H_s \leftrightarrow \varsigma_2 \leftrightarrow H_{2s} \leftrightarrow \varsigma_3 \leftrightarrow H_{2g}$, where ς_1 is the probability of an atom coming to the surface; ς_2 is the probability of atom recombination; ς_3 is the probability of molecular desorption.

*The explanation of the sense of physical parameters described in [21].

The indices s, g, and *me* mean the states of hydrogen in the metal surface, gas phase and metal volume. Often the additional stage, atoms migration on the surface before recombination, includes. The double arrows mean that reactions take place in two directions – degassing and absorption.

The amount of hydrogen released from the metal volume in each specific case will determine by the parameters of the initial state of the gas and metal, the rate of reactions during all three stages, and the hydrogen-metal phase diagram. If diffusion is the most slow part of the process then the gas flow from the metal obeys to the first Fick's law, i.e., is proportional to the gas concentration in the metal. For a clean metal surface the law of the amount of hydrogen dissolved in metal $C \sim a\cdot P^{1/2}$ is usually satisfied, where C is hydrogen concentration in metal; P is the pressure of molecular hydrogen, and a is the coefficient. The process of gas release would be more complicated if the metal surface is not clean, but coated with films of substances with strong chemical bond, e.g. oxides, carbides, etc. In this case, Fick's law can be not valid, and hydrogen behavior may differ from the classic one. In the presence of impurities on the palladium surface, especially carbon, the rate of hydrogen absorption-degassing processes can significantly decrease. Perhaps, some decrease the amount of hydrogen with increasing sorption time (≈ 10%, see Fig. 3b) is associated with the accumulation of impurities on the Pd sample surface.

As for the kinetics of hydrogen interaction with the palladium surface under the influence of electrolysis current, in this case impurities can also affect the rates of surface reactions. At least, the energy of palladium degassing after electrolysis ≈ 40 kJ/mole is much higher than that for pure Pd (≈ 15 kJ/mole). From the other hand, impurities on the surface and changes in the surface morphology can play the role of barrier for hydrogen desorption, providing it trapping in metal volume.

CONCLUSIONS

The current-voltage characteristics of the electrolysis process measured in various regimes with the different polarity of electrodes. It had shown the current-voltage characteristics are similar to each other, despite the fact that cathode surface facing to electrolyte changed in three times. The reasons of such behavior had analyzed.

Mass specter of the mixture of gases, generated during electrolysis, and hydrogen out gassing in a vacuum, measured. At an electrolyte temperature of ~ 50 °C and a pressure in the electrolyze chamber of ~ 1 atm, the generated gas is 92 vol.% hydrogen. After hydrogen absorption by Pd-cathode during electrolysis, and with subsequent heating in a vacuum, pure (better than 99.999 vol.%) hydrogen are generated.

The out gassing rate of hydrogen from palladium cathode after exposure to ion current during electrolysis process in the electrolyte measured in dependence on temperature and the time of exposure. The activation energy of the hydrogen out gassing from the cathode after one-hour exposure to electrolysis current calculated to be E ≈ 40 kJ/mole.

The amount of hydrogen desorbed from the cathodes (productivity) estimated. For the Pd-cathode, it was about 850 Ncm^3H_2 (56 Ncm^3H_2, H/Pd ~ 0.48) and hydrogen out gassing rate at the maximum temperature 650 °C was 0.3…0.5 $cm^3 H_2/s$.

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Article received 11.09.2025

МОЖЛИВИЙ МЕТОД ОТРИМАННЯ НАДЧИСТОГО ВОДНЮ

Г.П. Глазунов, М.М. Бондаренко, О.Л. Конотопський

Описано можливий метод отримання надчистого водню (чистота вища, ніж 99,999 об.%). Чистий водень утворюється як побічний продукт одночасно з технічним отриманням технічного водню в процесі електролізу. Використання Pd-катодів забезпечує їх високе насичення воднем під час процесу електролізу, а потім, при нагріванні у вакуумі, – отримання потоку надчистого водню. Було показано, що Pd-катод масою 14,5 г поглинає близько 1 л водню протягом 1 год процесу електролізу в 1 ваг.% розчині соди у воді. Швидкість насичення воднем у процесі електролізу приблизно у шість разів вища, ніж в експериментах з молекулярним воднем. Енергія активації процесу дегазації, що розрахована за температурною залежністю швидкості виділення водню від температури, становить ≈ 40 кДж/моль. Мас-спектрометричні вимірювання підтвердили високу чистоту отриманого водню. Проаналізовано фізико-хімічні механізми для пояснення отриманих результатів.