

INFLUENCE OF DIFFERENT TYPES OF HYDROGEN TREATMENT ON HYDROGEN RETENTION AND RELEASE FROM 12Kh18N10T STEEL

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Hydrogen outgassing from stainless steel was investigated after sample treatment by different plasmas: VHF/RF pulse discharges during wall conditioning in the Uragan-2M torsatron, with RF pulse plasma heating regime in Uragan-2M, with steady state plasma of magnetron type discharges in DSM-1 device, with pulse plasma in the plasma accelerator QSPA Kh-50. For comparison the rate of hydrogen release from SS samples was measured after their saturation in molecular hydrogen gas at the low pressure of $\sim 10^{-2}$ Torr at the room temperature. The measurements of hydrogen release rate were carried out with thermal desorption method and mass-spectrometry. It was observed the significant differences in hydrogen outgassing behavior for samples treated with different hydrogen plasma. The possible reasons of distinctions in kinetics of hydrogen interaction with stainless steel are discussed.

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INTRODUCTION

Hydrogen retention in the vacuum chamber walls of plasma devices and its evolution during plasma device operation can strongly influence on plasma parameters, so investigations of hydrogen behavior, namely, hydrogen gas outgassing and retention in stainless steel, as the main wall material, are very important. During operation of Uragan-2M (U-2M) stellarator wall material (12Kh18N10T stainless steel, analog to St316) may be exposed to different hydrogen plasmas: plasma of VHF/RF pulse discharges of various cleaning regimes, plasma of RF work regime [1, 2].

In this work, using earlier developed impulse thermal desorption method [3], the influence was examined of different plasmas treatment of samples in U-2M in situ on hydrogen retention and release from 12Kh18N10T stainless steel. In addition the measurements of hydrogen outgassing rate in the special stand had been carried out after plasma treatment of SS samples in the plasma accelerator QSPA Kh-50, with steady state plasma of magnetron type discharges in DSM-1 device. Also, for comparison, saturation in molecular hydrogen gas was used at the low pressure of 1.3 Pa ($\sim 10^{-2}$ Torr) and at room temperature.

1. EXPERIMENTAL

For all experiments the samples for studies were the strips made of similar to U-2M vacuum chamber material, i.e. stainless steel (SS) 12Kh18N10T with such dimensions of 10x190x(0.3...0.5) mm. Before SS sample placing in plasma device it was cleaned with such procedures: fine sandpaper cleaning, wiping with special fabric wetted in clean branded gasoline, drying, wiping with special fabric wetted in 96 % ethanol, drying.

Then the initial hydrogen outgassing q from the SS sample was measured in the special device GAS (Fig. 1)

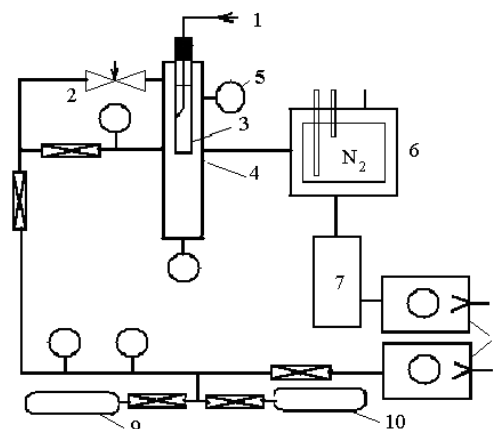


Fig. 1. Scheme of the experimental device for the study of gas release, hydrogen sorption, hydrogen permeability: 1 – W-Re thermocouple; 2 – flow; 3 – probe; 4 – vacuum chamber; 5 – mass spectrometer; 6 – nitrogen condensation pump; 7 – diffusion pump; 8 – for-vacuum pump; 9 – hydrogen balloon; 10 – helium balloon

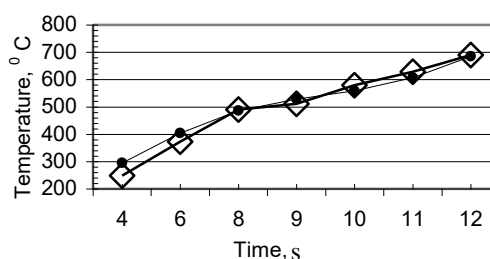


Fig. 2. Temperature dependence on the heating time: circles – thickness of SS sample is 0.3 mm, squares – sample thickness is 0.5 mm

Direct current impulse heating of the samples was used during experiments in the GAS and in U-2M. The dependence of probe temperature on the time of heating at the 5 V applied voltage is presented in Fig. 2. The SS

probe temperature was measured by a W-Re thermocouple attached into the probe centre.

It is seen that 4 seconds is sufficient to provide probe heating to the temperature of 250...300°C and 8 seconds to 500°C. Before the treatment, each sample to be analyzed was placed in the GAS and after pumping to $2 \cdot 10^{-7}$ Torr was baked at the temperature of 700°C during 1 hour. It is enough to remove the principal amount of hydrogen dissolved in the sample. To control the degree of degassing, after cooling the samples to room temperature, impulse heating was first carried out to 300°C, in order to remove surface gases. Then after cooling to room temperature, the sample was heated once more, but to the temperature of 500°C at which the initial specific rate of hydrogen outgassing (hydrogen release) q from the metal volume was measured (Fig. 3, Fig. 5 - point 1) by thermal desorption method earlier described in [3]. Outgassing rate was calculated according to equation: $q = (P - P_0)S/F$, where S is the pumping speed, and F is the area of the probe surface heated to 500°C. P_0 is the initial pressure after sample heating. Using mass-spectrometry it was shown (Fig. 4) that mainly hydrogen desorbs from SS sample at this temperature.

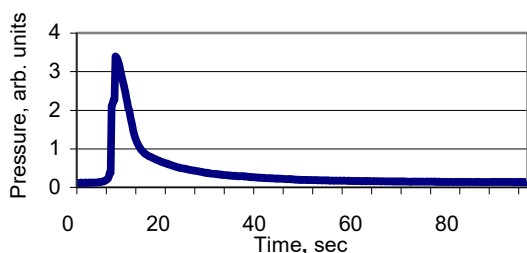


Fig. 3. Apparatus signal during impulse heating of the SS sample to the temperature of 500 °C

Then such samples were installed in the U-2M stellarator [1, 2], DSM-1 [4] or in the plasma accelerator QSPA Kh-50 [5] for hydrogen plasma treatment.

In the U-2M SS sample was treated during RF discharge pulsing cleaning regime when two RF-generators with power about 50 kW of each operated at low magnetic field (up to 0.1 T). Power of the one generator ($f = 5$ MHz) was applied to the frame antenna. Second generator power (4.8 MHz) was applied to the three-half-turn antenna. Pulse duration was 20 ms and the pulse duty cycle was 4 pulses/min. After two hours of such plasma impact the long time pumping during 18 hours was carried out. Then the measurements of hydrogen release rate were carried out accordingly to above described procedure (see point 2 in Fig. 5). After hydrogen release measurement the operation regime switches to the following: RF power from two generators was applied to two antennas. One of them ($f = 4.8$ MHz, 50 kW) was launched to the frame antenna and the second one ($f = 5$ MHz, 120 kW) was connected to three half-turn antenna. The pressure of working gas (hydrogen) was within the range $6 \cdot 10^{-6}$ to $2 \cdot 10^{-5}$ Torr, the magnetic field was ~ 0.36 T. Plasma pulses duration was 5...25 ms, in series of one pulse per 2 min. After two hours operation in such a regime,

U-2M vacuum chamber was pumped to the pressure of about $1 \dots 2 \cdot 10^{-6}$ Torr. Then the second measurement (see point 3 in Fig. 5) of hydrogen release from the SS probe was carried out at the same 500°C temperature in U-2M.. It is seen that hydrogen release rate increased in more than one order of magnitude. As the diffusion flow from the metal is proportional to hydrogen concentration in it, this result means essential increase of hydrogen content in the SS probe.

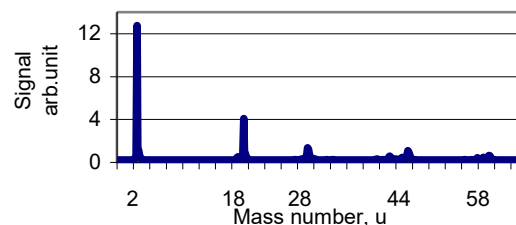


Fig. 4. Mass spectrum at a sample temperature of 500 °C

The treatment in DSM-1 was carried out in hydrogen plasma of stationary magnetron discharge (ion energy 0.7 keV and fluence $6 \cdot 10^{24}$ ions/m²). In this case the sample served as a cathode. After plasma treatment the sample was installed in GAS to measure hydrogen release rate according to above described method (see points 4, 5 in Fig. 5).

The SS sample treatment in the quasi-stationary accelerator QSPA Kh-50 was carried out at a power of 0.1...0.6 MJ/m². The fluence was $5 \cdot 10^{24}$ ion/m², pulse duration 0.25 ms, ion energy 0.4 keV [2]. At 0.6 MJ/m² power, the surface of the sample was melting, and at 0.3 MJ/m² and lower power the sample temperature was below the melting point. After plasma treatment SS samples was installed in stand GAS to measure hydrogen release rate (see points 6-12 in Fig. 5).

For comparison, hydrogen release rate was measured for SS sample saturated in a molecular hydrogen atmosphere at a pressure of $\sim 10^{-2}$ Torr during 24 hours (see point 13 in Fig. 5). These measurements were carried out in situ in the GAS.

2. RESULTS AND DISCUSSION

It is seen in Fig. 5 that hydrogen release rate in U-2M after operation regime increased in more than one order in comparison with exposure to RF discharge cleaning regime. It one could explain by essential difference in hydrogen particles energy in different regimes of operation. As the diffusion flow from the metal is proportional to hydrogen concentration in it, this result means essential increase of hydrogen content in the SS sample (walls) after operation in work regime.

Hydrogen outgassing from SS samples saturated in steady state discharges in DSM and in the plasma accelerator QSPA Kh-50 is close in value (see points 6-12 in Fig. 5). Behind of essential difference in treatment times (8 hours for DSM and 2.5 ms for QSPA) it means that only particle energy and fluence play the main role. Note, the changes in the sample surface state (melted or not) can cardinaly influence on kinetics of hydrogen interaction with stainless steel (Fig. 6).

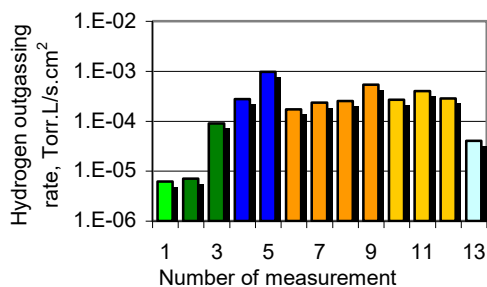


Fig. 5. Rates of hydrogen release from SS samples at the temperature of 500 °C

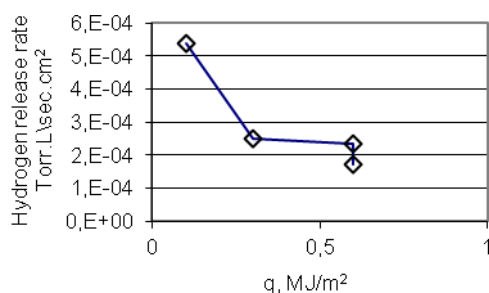


Fig. 6. Hydrogen release rate q dependence on heat load to SS sample surface

CONCLUSIONS

The significant difference was observed in hydrogen outgassing behavior for samples treated with different hydrogen plasmas. The minimum hydrogen release was measured after RF discharge cleaning regime. After RF pulse plasma heating regime hydrogen release rate increased in more than one order. It one could explain by essential difference in hydrogen particles energy. Hydrogen outgassing from SS samples saturated in steady state discharges in DSM and in the plasma accelerator QSPA Kh-50 is close in value, but decrease of heat load to surface (lower than SS melting point) leads to essential increase of hydrogen saturation.

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ВЛИЯНИЕ РАЗЛИЧНЫХ ТИПОВ ВОДОРОДНОЙ ОБРАБОТКИ НА ПОГЛОЩЕНИЕ И ВЫДЕЛЕНИЕ ВОДОРОДА ИЗ СТАЛИ 12Х18Н10Т

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Газовыделение водорода из образцов нержавеющей стали проводилось после обработки различной плазмой: импульсных разрядов УКВ/ВЧ во время чистки стенки в торсатроне Ураган-2М, в режиме нагрева плазмы с ВЧ-импульсом в Урагане-2М, в стационарной плазме разрядов магнетронного типа в устройстве ДСМ-1 с импульсной плазмой, в плазменном ускорителе КСПУ Х-50. Кроме того, для сравнения использовалось насыщение в молекулярном газообразном водороде при низком давлении ($\sim 10^{-2}$ Торр) и комнатной температуре. Измерения скорости выделения водорода проводились методом термической десорбции с участием масс-спектрометрии. Наблюдались существенные различия в характере выделения водорода для образцов, обработанных различной водородной плазмой. Обсуждаются возможные причины различий в кинетике взаимодействия водорода с нержавеющей сталью.

ВПЛИВ РІЗНИХ ТИПІВ ВОДНЕВОЇ ОБРОБКИ НА ПОГЛИНАННЯ ТА ВИДІЛЕННЯ ВОДНЮ ІЗ СТАЛІ 12Х18Н10Т

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Газовиділення водню із зразків нержавіючої сталі проводилося після обробки різною плазмою: імпульсних розрядів УКВ/ВЧ під час чистки стінки в торсатроні Ураган-2М, у режимі нагріву плазми з ВЧ-імпульсом в Урагані-2М, у стаціонарній плазмі розрядів магнетронного типу в пристрої ДСМ-1 з імпульсною плазмою, у плазмовому прискорювачі КСПП Х-50. Крім того, для порівняння використовувалося насичення в молекулярному газоподібному водні при низькому тиску ($\sim 10^{-2}$ Торр) та кімнатній температурі. Вимірювання швидкості виділення водню проводилися методом термічної десорбції за участю мас-спектрометрії. Спостерігалися істотні відмінності в характері виділення водню для зразків, оброблених різною водневою плазмою. Обговорюються можливі причини відмінностей в кінетиці взаємодії водню з нержавіючою сталлю.