IN SITU QUANTIFICATION OF PLASMA FACING SURFACE CONDITIONS IN THE URAGAN-2M TORSATRON

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The method and device has been developed and used for operative estimation of impurity level, outgassing rate and number of impurity gases molecular layers, on the surface of the Uragan-2M (U-2M) vacuum chamber walls. It is based on the thermal desorption of gases into a vacuum vessel from the surface of a special stainless steel probe during its heating up to temperature of 300ºC. The investigations were carried out of an outgassing rate and number of molecular layers on the probe surface *in situ* in the torsatron Uragan-2M. Decrease released gas amount from the surface by more than two orders of magnitude was recorded after Uragan-2M vacuum chamber VHF/RF discharge cleaning in various regimes and pumping. The method had been also tested in the regime of the measurements of hydrogen gas release retained in the probe before and after impact of RF plasma pulse discharges in work regime.

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INTRODUCTION

In many cases during experiments or technological processes in a high vacuum it is very important to know not only such vacuum parameters as residual gas pressure and partial impurity pressures in a volume of vacuum chamber which usually are measured. The outgassing rate of wall material, number of impurity molecular layers on the wall surfaces are also important. In particular, the latter essentially influences on the intensity of impurity influx to plasma at the initial stage of plasma device operation. For example, removing more than 100 molecules layers from the surfaces of the LHD stellarator vacuum vessel (NIFS, Japan) due to ECR discharge cleaning resulted in essential increase of plasma stored energy.

In the case of the Uragan-2M (IPP, NSC KIPT, Kharkov, Ukraine) the total number of gas molecules in vacuum vessel with volume of ≈ 4 m^3 at the pressure of 10^{-6} Torr is $\approx 1.4 \cdot 10^{17}$. At the same time 100 impurity molecular mono-layers on the surface of the U-2M vacuum chamber contain more than 10^{22} molecules. That means the measurements of residual pressure in a vacuum volume don't give the knowledge on real quantity of gas molecules in it. To estimate the real amount of gas molecules on the surface of vacuum chamber *in situ* one can using the thermal desorption method. Such method had been devised and preliminary tested in Ref. [1].

In present study the systematical measurements were carried out of outgassing rate of the stainless steel probe, and estimations were made of the number of molecules layer on its surface in the U-2M torsatron after pulsed VHF/RF wall conditioning procedure in different regimes. Besides the impurity level, the hydrogen retention in the vacuum chamber walls and its evolution during plasma device operation can strongly influence on plasma parameters. So the possibility to use such method for investigations of hydrogen behavior, namely, hydrogen gas outgassing and retention in stainless steel, *in situ* in the U-2M torsatron was addressed too.

1. EXPERIMENTAL CONDITIONS

The scheme of the experiment and the photo of device placed on the U-2M flange are shown in Fig. 1. The strip-like probe (#2) was made of similar to U-2M vacuum chamber (#1) material, i.e. stainless steel (SS) 12Kh18N10T, analogous to St316 steel. It was placed in such a way to be flush with the U-2M wall. The probe dimensions are 10 x 200 x 0.3 mm. Probe was connected to the massive copper contacts (#3) of the electric power supply (#6) providing pulsed heating of the probe to a high temperature. The highest temperature of probe heating used in this experimental series was 300ºC which is sufficient to stimulate release of impurities from its surface. At higher temperatures the gases, mainly hydrogen, will start to release from the metal bulk. The measured value was the pressure increase during probe heating. Then, such characteristics were estimated as the specific outgassing rate q (Torr·l/s·cm^2) and the number of molecules layers N on the probe surface.

Outgassing from the metal surface into a vacuum volume could be divided on two sub-stages: at the rather low temperatures up to 300°C, the gases adsorbed on the metal surface, mainly H_2O, CO, N_2, CO_2 release to the vacuum volume. Namely investigations of this stage will be presented in this paper first. When a metal temperature increases to 350…700°C, others gases, mainly H_2, are emitted. The retained hydrogen atoms diffuse in the metal bulk, they reach the surface and migrate along it, then recombine, and desorb in the molecular form. 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 [2]. Situation would be more complicated if the metal surface is not clean but

coated with films of matters with strong chemical bond, e.g. oxides, carbides, etc. In this case Fick's law can be not valid and gas behavior may differ from the classic one [3, 4], because other processes come to play, such as the gas motion through the interface boundary, diffusion in the films material, etc.

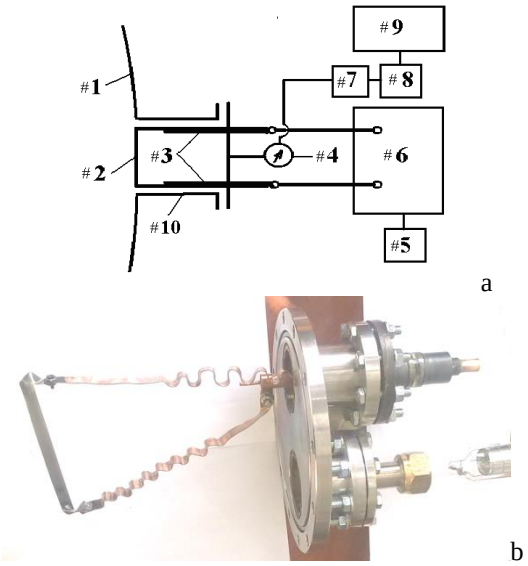

Fig. 1. The scheme of the experiment (a) and photo of device (b): 1 – U-2M vacuum chamber wall; 2 – stainless steel probe; 3 – current contacts; 4 – pressure gauge; 5 – time relay; 6 – electric power supply; 7 – VIT-2 vacuum measurement device; 8 – interface module WAD-AIK-BUS; 9 – computer; 10 – branch pipe

Before SS probe placing in the U-2M chamber it was calibrated on the heating voltage and specific outgassing rate at the temperatures of 200…700ºC in the special stand [5]. Outgassing behavior was investigated by means of thermal desorption and mass-spectrometry methods similar to described in the paper [6]. The SS probe temperature was measured by a W-Re thermocouple attached into the probe centre. It is necessary to note, that in this case the measurements were carried out in stationary regimes of heating. The specific net outgassing rate (q) was calculated from the equation

$$q = (P-P_0)S/F, \qquad (1)$$

where S=100 l/s was the pumping speed, and $F \approx 28$ cm^2 was the area of the heated to 250…300ºC probe surface. The estimated value of the specific outgassing rate of the SS probe in the temperature range 250…300ºC was determined as $\approx 8 \cdot 10^{-5}$ Torr·l·s^{-1}·cm^{-2} and was used to estimate q and N values measured in the U-2M, when the pulsed regime of probe heating was used. The 5 V applied voltage during 4 seconds is sufficient to provide probe heating to the temperature of 250…300ºC.

2. RESULTS, DISCUSSION

Then the probe has been placed in the Uragan-2M branch pipe (see #10 in Fig. 1,b). It is used without the thermocouple to provide minimum parasitic outgassing. U-2M vacuum chamber was pumped to the pressure of ~10^{-6} Torr by the fore-pump and three turbo molecular pumps, each with the net pumping speed 0.5 m^3/s. Then the SS probe measurements are used and the heating time is established as 4 s at the 5 V of applied voltage. The probe temperature increased up to 300ºC and the pressure increase in the branch pipe #10 (see Fig. 1) caused by desorbed gases was measured.

The measurements of the SS outgassing rate and number of monolayer estimations were carried out after such a wall conditioning daily procedure: 1.5…2 hours VHF/RF steady state or pulsed discharge cleaning (standard VHF and RF regimes of discharges cleaning were described in [7-9]) in atmosphere of H_2, N_2, gases, and their mixture, then pumping during 1.5…2 hours with 3 turbo molecular pumps, then 1.5..2.5 hours VHF/RF pulsed discharge cleaning again and long time pumping during 18 hours with three turbo molecular pumps. The VHF steady state discharge characteristics were: VHF (f=140 MHz) generator power was about 3 kW and applied to T-like antenna both in regime with magnetic field and without. The pressure of working gas during regime without magnetic field was $2 \cdot 10^{-2}$ Torr , and in the regime with low magnetic field (~ 0.01 T) it was $1 \cdot 10^{-4}$ Torr.

During RF pulse discharge cleaning regime, two RF-generators with power about 50 kW of each operated at low magnetic field (up to 0.01 T). Power of the one generator (f=5 MHz) applied to the frame antenna. Second generator power (4.8 MHz) applied to the three-half-turn antenna. Pulse duration was 20 ms and the pulse repetition rate was 4 pulses/min.

The typical apparatus curves of pressure temporal behavior are shown in Figs. 2, 3. The small signal in Fig. 3 measured 1 min later after switch off the first pulsed heating, corresponds to less than one monolayer and it means that practically all gases adsorbed on the probe surface had been desorbed during first thermal pulse. Note, that the time of formation of one monolayer on the surface at room temperature in vacuum conditions of 10^{-6} Torr is only about 5 seconds [10].

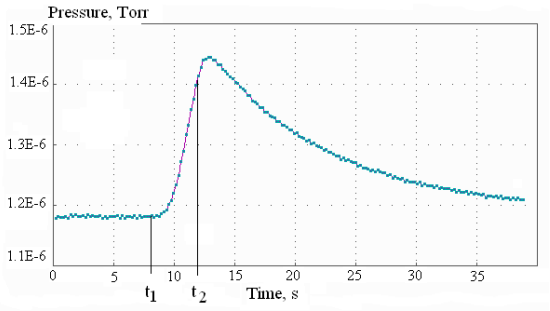

Fig. 2. Apparatus curve of pressure increase in the Uragan-2M during SS probe heating to the temperature of 300ºC: t_1 and t_2 are the times of switch on and switch off the heating, initial pressure is $1.18 \cdot 10^{-6}$ Torr

The specific outgassing rate was estimated from the above mentioned equation (1).

The estimation of monolayer number on the probe surface was carried out with the use of the equation:

$$N=V\cdot L/N, \qquad (2)$$

where V (normal cm^3)= $q\Delta t$ – the amount of the gas desorbed from the unitary probe surface; Δt (s) – time of the gas desorption; L – number of molecules in the gas volume of the 1 cm^3 (Loshmidt's number); N_w – the number of molecules in the monolayer on the unitary surface. In the calculations it was supposed the water vapor as the main adsorbed substance and, according to Ref. [11] data, $N_w \approx 5\cdot10^{14}$ cm^{-2}. In fact, the mass-spectrometric measurements made during SS probe heating to 300ºC temperature in the U-2M-vacuum chamber and also measurements in the above mentioned special stand confirmed this assumption. Noticeable increasing of desorbed CO_2 (44 u), 28 u and hydrocarbons (58 u) was observed, too.

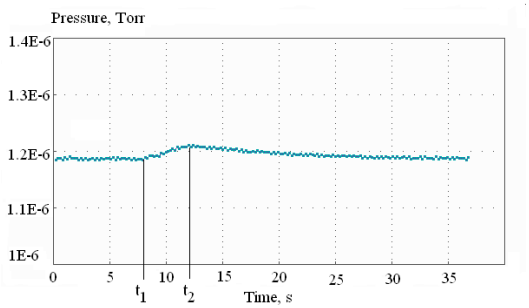

Fig. 3. Apparatus curve of pressure increase in the U-2M during SS probe heating to the temperature of 300ºC after 1 minute of the first pulsed heating: t_1 and t_2 are the times of switch on and switch off the heating

Numerical calculation is applied to the measurement displayed in Fig. 2. The analysis is based on the gas balance equation:

$$d(pV)/dt = Q - pS, \qquad (3)$$

where Q is cumulate outgassing (Torr·l/s). In the equation, the vacuum vessel V volume and the pumping speed S are assumed to be constant. The outgassing and the pressure could be split into two parts $Q=Q_0+Q_p$ and $p=p_0+p_p$, where Q_0 and p_0 are the stationary solutions of Eq. (3), and Q_p and p_p are probe induced variations (non-stationary). As it is seen from Fig. 3, after heat pulse to the probe the pressure decreases with the characteristic time t_d~10 s. However, it does not return to the initial value. The residual pressure $p_{p*}=p_p-p_g$ starting certain time after heat pulse should obey Eq. (1) with zero outgassing term. In this case the equation has an analytical solution

$$p_{p*}=p_1\exp[(t-t_1)/t_0], \qquad (4)$$

where t_1 is an arbitrary time moment. The result of numerical fitting of analytical solution to the experimental curve is shown in Fig. 4.

The fact that fitting is successful could be explained by domination of one gas sort in the released gas mixture. Fitting gives the value of $t_0=V/S$=9.4 s. The corresponding pumping speed is S=420 l/s. Knowing pumping speed, the cumulate outgassing is calculated using Eq. (3) Fig. 5.

The above described procedure of measurements and simplified data processing is applied to the different conditions in the U-2M vacuum chamber that gives the dynamics of decreasing of outgassing rate from the vacuum chamber walls and number of molecules layers during U-2M chamber wall conditioning (Fig. 6).

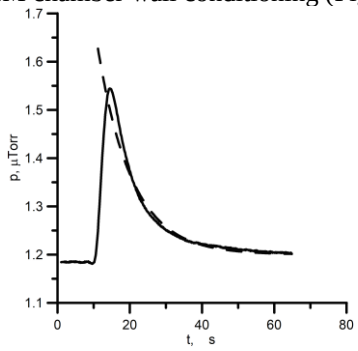

Fig. 4. Pressure curve from Fig. 3 (solid line) and fitted analytical curve (dashed line)

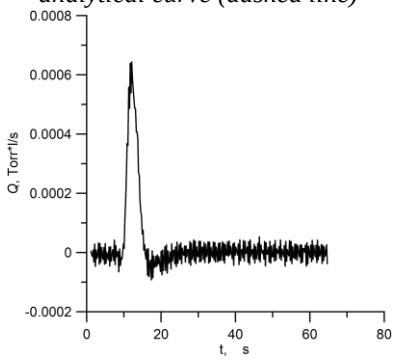

Fig. 5. Cumulate outgassing calculated for the measurement displayed in Fig. 7

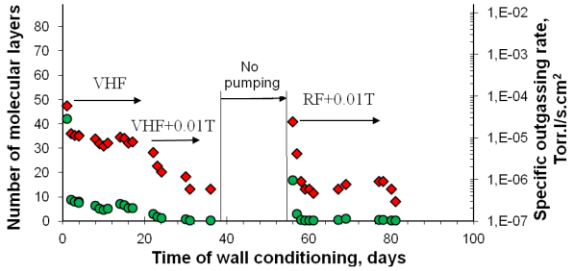

Fig. 6. Uragan-2M wall conditions: green points correspond to number of molecular layers on the surface of SS probe; red rhombs correspond to SS probe outgassing rate at 300ºC after VHF or RF discharge cleaning and pumping

The figure reflects, in general, positive wall conditioning tendency with different rates for different wall conditioning scenarios. It is seen in Fig. 6 (points 21…36) that steady state VHF discharge cleaning in low (0.01…0.02 T) magnetic field is more effective than the regime without magnetic field (points 1…17). In turn, the RF pulse discharge wall conditioning in these conditions seems more effective than the VHF (points 52…82). The

number of molecules layers was decreased up to less than one layer for both VHF and RF discharge cleaning. Drastic increase of the outgassing rate in the point 56 is caused by pumping void during 16 days. In this time the U-2M vacuum chamber was filled with nitrogen at the atmosphere pressure.

Note, if to measure the SS probe outgassing just after the short time RF/VHF discharge cleaning, the SS outgassing rates are essentially, in about one order of magnitude higher, as it is seen in Fig. 7.

It is the evidence that very high impurity flow desorbs under plasma-surface interactions and that the pumping facilities do not cope with impurity pumping. As the result these impurities redeposit on the walls of the vacuum chamber and branch pipes after finishing of the discharge cleaning. The subsequent pumping of the U-2M vacuum chamber during 18 hours leads to obtaining rather high vacuum with $7\cdot10^{-7}$ Torr pressure and to decreasing of the number of molecules layers on the SS probe surface up to N=0.5…1 instead of N~100 at the pressure of ~$7\cdot10^{-6}$ Torr at the beginning of the wall discharge cleaning campaign.

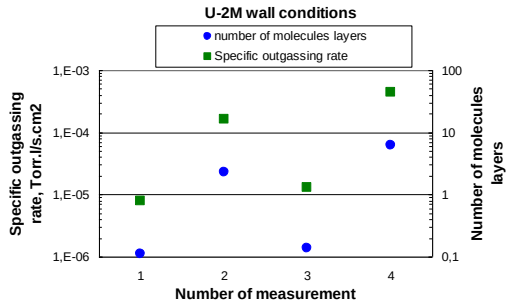

Fig. 7. U-2M wall conditions in different times of measurements: points with index 1 (RF) and 3 (VHF) were measured for standard procedure of discharge cleaning just after 18 hours pumping; points 2 and 4 were obtained one hour shortly after the discharge cleaning end both for the VHF(2) or RF(4)

Finally, some words on the possibility to use such methods for hydrogen behavior (retention and release from stainless steel) investigations *in situ* in the U-2M torsatron. The effective hydrogen diffusion and desorption starts at the temperatures above 350°C. If after the SS probe heating to the temperature of 300°C to heat it once more, but to the temperature of 500°C (by increasing of voltage or time of electric pulse), the hydrogen atoms in the metal bulk will diffuse to the surface, where recombine into molecules and then will desorb.

It is shown in Fig. 8 hydrogen release from the SS probe during its heating to temperatures of 500…700°C. The measurement at 500ºC temperature (green point 1) was carried out after standard cycle of discharge cleaning: 1.5…2 hours RF pulsed discharge cleaning in H_2, then pumping during 1.5…2 hours, then 1.5…2.5 hours RF pulsed discharge cleaning and long time pumping during 18 hours. After hydrogen release measurement the operation regime switched 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 $6\cdot10^{-6}$ Torr to $2\cdot10^{-5}$ 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 work in such a regime, it was switched off and U-2M vacuum chamber was pumped to the pressure of about $(1…2)\cdot10^{-6}$ Torr. Then the measurement of hydrogen release from the SS probe was carried out at the same 500ºC temperature (green point 2, Fig. 7). It is seen that hydrogen release rate increased in more than one order. Then a few high heating pulses (5 V, 10 s, brown points in Fig. 8) were applied to the probe to remove hydrogen from the probe bulk. Pulses off-duty time was two minutes.

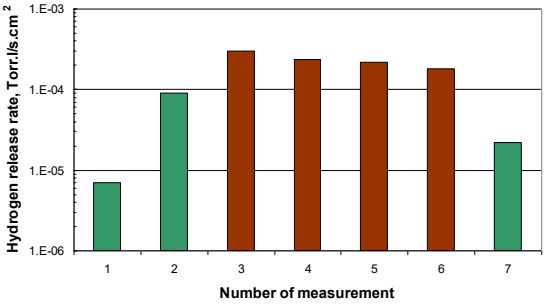

Fig. 8. Hydrogen release from the SS probe: green color – sample temperature under the measurement was 500ºC; brown color – sample temperature under the measurement was 600…700ºC

From pulse to pulse the SS probe temperature increased from 600°C (first pulse) to 700°C (fourth pulse). Then, after the probe cooling down to the room temperature, the measurement of hydrogen release rate at the temperature of 500°C was carried out (point seven in Fig. 7).

It is seen that after four high heating pulses hydrogen gas release (hydrogen outgassing rate) decreased in about four times. So, using proposed method one can effectively monitor not only the surface conditions but hydrogen retention with the vacuum chamber wall material, too. Note, not only hydrogen particles in different states impact on the U-2M wall surface. It may be nitrogen, oxygen, carbon, etc. Study of recycling process for those is also important. In general case this is of a great interest not only for fusion device but for different plasma installations, e.g., plasma accelerators, devices for vacuum-plasma or magnetron deposition, glow discharge plasma facilities etc.

SUMMARY AND CONCLUSIONS

Using thermal desorption method the investigations were carried out of the wall conditions, outgassing rate and estimation of the number of molecules layers, in the Uragan-2M torsatron *in situ* after discharge cleaning in different regimes and pumping. It had been indicated that the VHF and RF discharge cleaning in low magnetic fields of 0.01…0.02 T is more effective than the regimes

without magnetic field. After preliminary short time VHF/RF discharge cleaning and long time pumping the number of impurity molecules layers was decreased from 100 up to less than one layer. The analysis of the obtained data allows saying that such method could be used to monitor the quality of wall conditioning processes during preparing to plasma experiments.

The proposed method was tested in the high temperature regime (400…700ºC) to measure hydrogen outgassing from the SS probe. It was observed the essential (one order of magnitude) increase of hydrogen outgassing after two hours exposure by plasma discharge in working regime. It means that hydrogen concentration in the SS probe also increased.

It is necessary to note, that using thermal desorption probe method one can effectively monitor not only surface conditions but hydrogen retention in the vacuum chamber wall material, too.

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IN SITU КОЛИЧЕСТВЕННЫЙ АНАЛИЗ УСЛОВИЙ НА ПОВЕРХНОСТИ СТЕНОК ВАКУУМНОЙ КАМЕРЫ ТОРСАТРОНА УРАГАН-2М

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Для оперативной оценки количества примесей на поверхности вакуумной камеры торсатрона Ураган-2М использован метод, основанный на термодесорбции газов в вакууме с поверхности зонда из нержавеющей стали при его нагреве до температуры 300°С. Проведены исследования газовыделения и оценка числа монослоев примесей на поверхности зонда в непосредственно торсатроне Ураган-2М. После откачки и чистки ВЧ/УКВ разрядами в различных режимах количество примесей на поверхности снизилось более чем на два порядка. Метод испытан также в режиме измерения скорости выделения водорода из зонда до и после воздействия плазмы импульсных ВЧ-разрядов в рабочем режиме.

IN SITU КІЛЬКІСНИЙ АНАЛІЗ УМОВ НА ПОВЕРХНІ СТІНОК ВАКУУМНОЇ КАМЕРИ ТОРСАТРОНА УРАГАН-2М

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Для оперативної оцінки кількості домішок на поверхні вакуумної камери торсатрона Ураган-2М використано метод, що грунтується на термодесорбції газів у вакуумі з поверхні зонда з нержавіючої сталі при його нагріві до температури 300°С. Проведено дослідження газовиділення і оцінка числа моношарів домішок на поверхні зонда безпосередньо в торсатроні Ураган-2М. Після відкачування і чищення ВЧ/УКВ розрядами в різних режимах кількість домішок на поверхні знизилася більш ніж на два порядки. Метод випробувано також у режимі виміру швидкості виділення водню із зонда до і після дії плазми імпульсних ВЧ-розрядів у робочому режимі.