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Wall conditions characterization in high vacuum chambers using stainless steel thermal desorption probe

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Abstract

The method and device has been developed and used for operative estimation of impurity level, outgassing rate and molecules layers number on high vacuum chamber surfaces of magnetic confinement fusion devices *in situ*. It is based on the thermal desorption of gases into a vacuum vessel from the surface of a special metal probe during its heating up to temperature of 300ºC. To investigate and use this method, the probe was designed, manufactured and installed into the Uragan-2M plasma device vacuum chamber. During the measurements, decrease released gas amount from the surface by more than one order of magnitude was recorded after Uragan-2M vacuum chamber VHF/RF discharge cleaning in various regimes and pumping. The mass-spectrometric measurements had shown 18 u (H_2O), 28 u ($CO+N_2$) and 44 u (CO_2), as the main gases desorbed from the probe surface during its heating. Heavy hydrocarbon masses (mainly 58 u) were also registered. The method had been also tested in the regime of the measurements of hydrogen gas retained in the wall.

Keywords: wall conditions, thermal desorption diagnostics, outgassing in a vacuum, molecules layers, discharge cleaning, torsatron Uragan-2M

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1. Introduction

In many cases during experiments or technological processes in a high vacuum it is very important to know such vacuum parameters as residual pressure and partial impurity pressures in a volume of vacuum chamber which usually are measured. Besides, the outgassing rate of wall material, numbers of impurity molecules layers on the wall surfaces are also important. In particular, the latter essentially influences on the intensity of impurity influx to plasma and, consequently, on the discharge performance. For example, as it is seen in Fig.1 [1], removing more than 100 molecules layers in the LHD stellarator vacuum vessel due to ECR discharge cleaning resulted in essential increase of plasma stored energy.

In the case of the Uragan-2M torsatron (U-2M, Ukraine, Kharkov, IPP NSC KIPT) the total number of gas molecules in the vacuum vessel with the volume of $\approx 4m^3$ at the pressure of 10^{-6} Torr is $1.4 \cdot 10^{17}$. 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 the residual pressure in a vacuum volume don't give the knowledge on real amount of gas molecules in it. To estimate the real quantity of gas molecules on the surface of vacuum chamber *in situ* is possible using the thermal desorption method [2-4]. In those papers the special probe in the form of W wire or strip preliminary decontaminated at high temperature was placed in the vacuum chamber. Surface contamination level acquired by the probe after its placing into the vacuum chamber in Ref. [4] was quantitatively estimated by the measurement of pressure

increase during short-pulse heating of the probe. Since the main material of most plasma devices is stainless steel, it is interesting to apply above mentioned method using a stainless steel (SS) probe instead of the tungsten one for operative estimation of surface impurity level. Such method had been devised and preliminary tested in Ref. [5].

In the present study, the systematical measurements were carried out of outgassing rate of the stainless steel probe, and estimations were made of the number of molecular layers 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.

2. Experimental investigations

The scheme of the experiment and the photo of device placed on the U-2M flange are shown in Fig. 2. 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 mm x 190 mm x 0.3 mm. Probe was connected to the massive copper contacts (#3) of the electric power supply (#6) providing pulsed Ohmic 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. The retained gases, mainly hydrogen, will start to release from the metal bulk at higher probe

temperatures. The measured value was the pressure increase ΔP in the branch pipe 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 separated into 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, the investigations of this stage are presented in this paper first. When the probe temperature increases to 350°C-700°C, others gases, mainly H_2, are emitted. High temperature stimulates diffusion of the retained hydrogen atoms in the metal bulk. The atoms reach the surface of the probe 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 [6]. 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 [7, 8], because other processes come to play, such as the gas motion through the interface boundary, diffusion in the films material, etc. Note, in our case the latter could be important factor due to possibility of carbonization of stainless steel surface because of presence such residual gases as CO_2, CO and hydrocarbons during the discharge cleaning procedure.

Before SS probe placing in the U-2M chamber it was calibrated on the Ohmic heating voltage and specific outgassing rate at the temperatures of 200-300ºC in the special

stand [9]. Outgassing behavior was investigated by means of thermal desorption and mass-spectrometry methods similar to described in the paper [10].

After the SS probe placing in the vacuum chamber #4 (Fig.3) of the stand, the last one was pumped to the pressure of 10^{-6}-$2\cdot10^{-7}$ Torr. After that, the investigated SS probe #3 was heated up to a required temperature value (250-300ºC), and the maximum increase in the total pressure which was caused by gases desorbed from the sample was measured. At the same time, a mass-spectrum of the cumulate desorbed gas was registered (Fig.4). The registered mass spectra showed that the major desorbed gases were those with atomic masses A = 18 u (H_2O), 28 u ($CO+N_2$), 44 u (CO_2) and 58 u (supposingly hydrocarbon). 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

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

where S is the pumping speed, and F is the area of the probe surface heated to 250-300ºC. P_0 is the initial pressure and P is the maximum pressure after heating. The values S=100 l/s and $F\approx$28 cm^2 which are characteristic for the stand and the probe are used in calculations.

The estimated value of the specific outgassing rate of the SS probe in the temperature range 250-300ºC was determined as $\approx8\cdot10^{-5}$ Torr·l·s^{-1}·cm^{-2}. The pulsed regime of probe heating is also investigated. In Fig. 5 the dependence of SS probe temperature on the time of heating at the 5 V applied voltage is shown. It is seen that 4 seconds is sufficient to provide probe heating to the temperature of 250-300ºC.

Then the probe has been placed in the U-2M port (Fig.2b). It is used without the thermocouple to provide minimum parasitic outgassing. U-2M vacuum chamber was pumped to the pressure of ~10^{-6} Torr (Fig.6) 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 5V of applied voltage. According to the experiments on the stand, the probe temperature increased up to 300ºC and the pressure increase in the branch pipe #10 (Fig. 2) caused by desorbed gases was measured using of the ionization gauge (#4), the vacuum measuring apparatus VIT-2 (#7), the interface module WAD-AIK-BUS (#8) and the computer (#9).

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 [11-13]) 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 approximately 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 discharge pulsing cleaning regime, two RF-generators with power about 50 kW of each operated at low magnetic field (up to 0.1T). Power of the one generator (f=5MHz) 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.

The typical apparatus curves of pressure temporal behavior are shown in Figs. 7, 8. The small signal in Fig.8 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 [14].

The specific outgassing rate was estimated from the above mentioned formula (1). The estimation of monolayer number on the probe surface was carried out with the formula:

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

where $V = q\Delta t$ – 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. [15] data, $N_w \approx 5 \cdot 10^{14}\,cm^{-2}$. In fact, the mass-spectrometric measurements made during SS probe heating to 300ºC temperature in the U-2M-vacuum chamber (Table.1) and also measurements in the above mentioned special stand confirmed this assumption, see (Fig. 4). Noticeable peaks of desorbed CO_2 (44 u), 28 u and hydrocarbons (58 u) was observed, too.

A numerical calculation was applied to the measurement displayed in Fig. 7. The analysis was based on the gas balance equation

$$\frac{d(pV)}{dt} = Q - pS \ , \qquad (3)$$

where Q is the cumulate outgassing (Torr·l/s). In the equation, the vacuum vessel V_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. 7, after heat pulse to the probe the pressure decreases with the characteristic time t_0~10 s. However, it does not return to the initial value. It is reasonable to suggest that additional prolong outgassing appears in the branch pipe. It induces the pressure $p_g \approx 7 \cdot 10^{-8}$ Torr. The residual pressure $p_{p*}=p_p-p_g$ starting certain time after heat pulse should obey Eq. (3) with zero outgassing term. In this case the equation has an analytical solution $p_{p*}=p_1\exp[(t-t_{ar})/t_0]$, where t_{ar} is an arbitrary time moment. The result of numerical fitting of analytical solution to the experimental curve is shown in Fig. 9.

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 $t_0=V_v/S$=9.4 s. The corresponding pumping speed is S=0.42 m^3/s. Knowing pumping speed, the cumulate outgassing is calculated using Eq. (3), see Fig. 10. The curve in Fig. 10 has a narrow peak of outgassing. The interesting feature is the outgassing acquires small negative values shortly after a while of the heat pulse. This could be explained so, that released gas contains a component which, after leaving the probe, very quickly redeposited to the vacuum vessel walls.

2. Results and discussion

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 (see Fig.11). The figure reflects, in general, positive wall conditioning tendency with different rates for different wall conditioning scenarios. It is seen in Fig.11 (points 21-36) that steady state VHF discharge cleaning in low (0.1-0.2 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 not after 18 hour pumping, but one hour after the RF/VHF discharge cleaning, the SS outgassing rates are essentially, in about one order of magnitude higher, as it is seen in Fig. 12. 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 $7 \cdot 10^{-7}$ 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~40 at the pressure of ~$7 \cdot 10^{-6}$ Torr at the beginning of the wall discharge cleaning campaign.

Finally, some words on the possibility to use the probe for hydrogen behavior (retention and release from stainless steel) investigations *in situ* in the U-2M torsatron. It is known [16] that 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 the Ohmic heating voltage or time duration of the electric pulse), the hydrogen atoms in the metal bulk will diffuse to the surface, where recombine into molecules and then desorb. Special mass-spectrometric measurements in the stand (Fig.3) and in U-2M vacuum chamber have confirmed that in the temperature range 350-700°C hydrogen is the one of the main gases desorbing from SS probe.

It is shown in Fig.13 hydrogen release from the SS probe during its heating to temperatures of 500-700°C. The first measurement (500ºC, point 1 in Fig.13) 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 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 $6 \cdot 10^{-6}$ Torr 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 $1\text{-}2 \cdot 10^{-6}$ Torr. Then the second

measurement (point 2 in Fig.13) of hydrogen release from the SS probe was carried out at the same 500ºC temperature. It is seen that hydrogen release rate increased in more than one order. As the diffusion flow from the metal is proportional to hydrogen concentration in it, this results means essential increase of hydrogen content in the SS probe.

Then a few high heating pulses (5V, 10 s, points 3-6 in Fig.13) were applied to the probe to remove hydrogen from the probe bulk. Pulses off-duty time was two minutes. From pulse to pulse the SS probe temperature increased from 600°C (first pulse, point 3) to 700°C (fourth pulse, point 6). Then, after the probe cooling down to the room temperature, the measurement of hydrogen release rate at the temperature of 500C was carried out (point 7 in Fig. 13). It is seen that after only 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 in the vacuum chamber wall material, too.

4. Summary and conclusion

The thermal desorption method has been developed for operative diagnosing impurity level on Uragan-2M vacuum chamber surfaces *in situ*. To perform the experiments the device was designed, manufactured and installed in the U-2M vacuum chamber, which gives possibility to register low flows of gases desorbed from the 12KH18N10T stainless steel strip-like probe during its pulsed heating up to temperature 250-300ºC.

The investigations were carried out of outgassing rate and estimation of the number of molecules layers, in the Uragan-2M torsatron *in situ* after plasma 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 are 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 ≈40 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. Mass-spectrometric measurements has shown domination of H_2O (18 u), presence of CO_2 (44 u) and 28 u, as the main gases desorbed from the SS probe surface during its heating. Heavy hydrocarbon masses (58 u) were also registered.

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 RF discharges in the regular regime. It means that the hydrogen content in the SS probe also increased after plasma-probe surface interaction. So, using thermal desorption probe method one can effectively monitor not only the surface conditions but the hydrogen retention in the vacuum chamber wall material, too.

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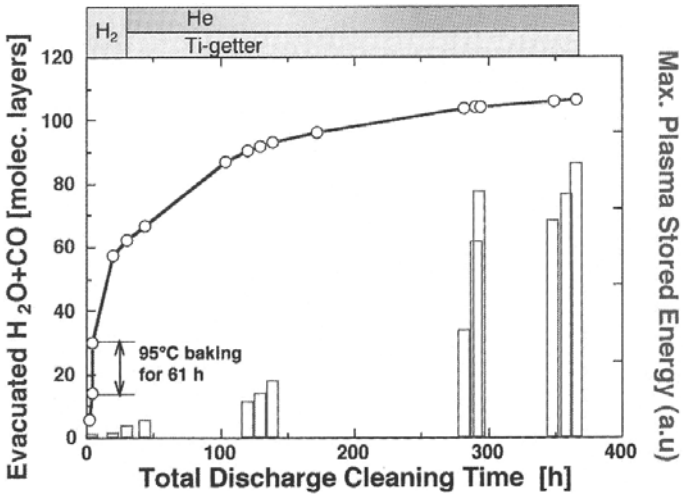

Fig.1. Quantity of gas removed with ECR discharge cleaning and plasma stored energy in LHD device [1].

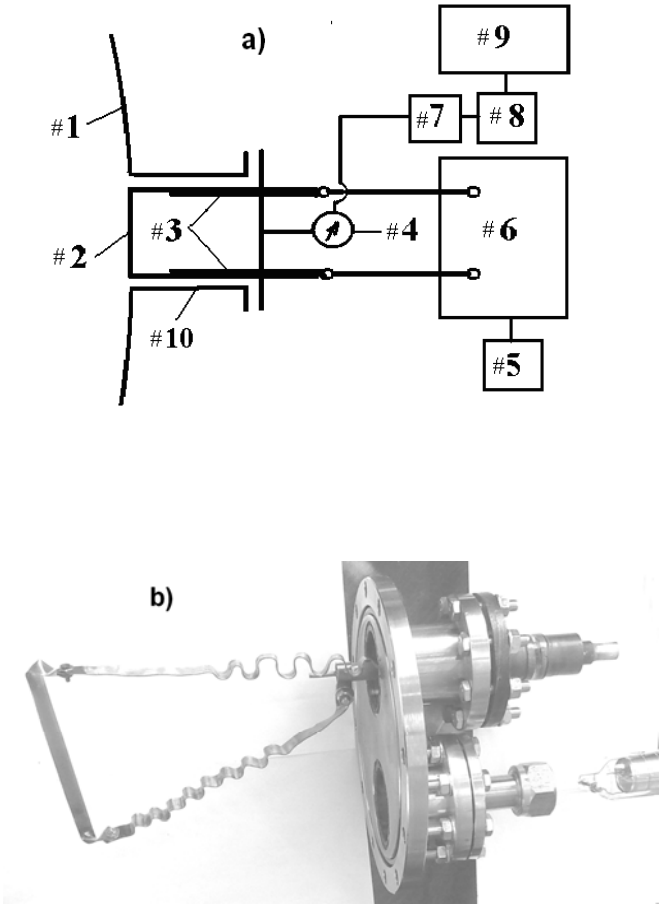

Fig.2. Scheme of the experiment (a) and photo of device (b): 1- U-2M vacuum chamber wall, 2- stainless steel probe, 3- current contacts, 4-ionization gauge, 5- time relay, 6-electric power supply, 7- VIT-2 vacuum measurement device, 8- interface module WAD-AIK-BUS, 9 - computer, 10 - branch pipe.

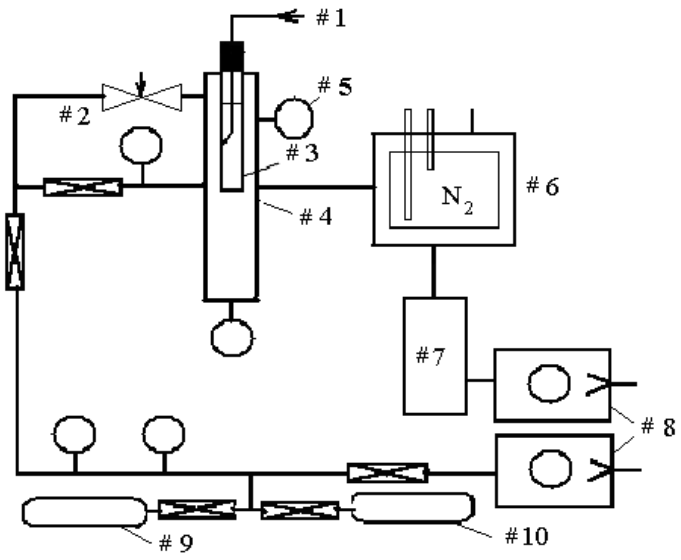

Fig.3. Scheme of the experimental stand for outgassing, hydrogen retention and release researches: 1- thermocouple, 2- leak valve, 3- SS probe, 4- vacuum chamber, 5- mass-spectrometer, 6- nitrogen condensation pump, 7- oil pump, 8- rotary pumps, 9- hydrogen balloon, 10-helium balloon, 11- vacuum gauges.

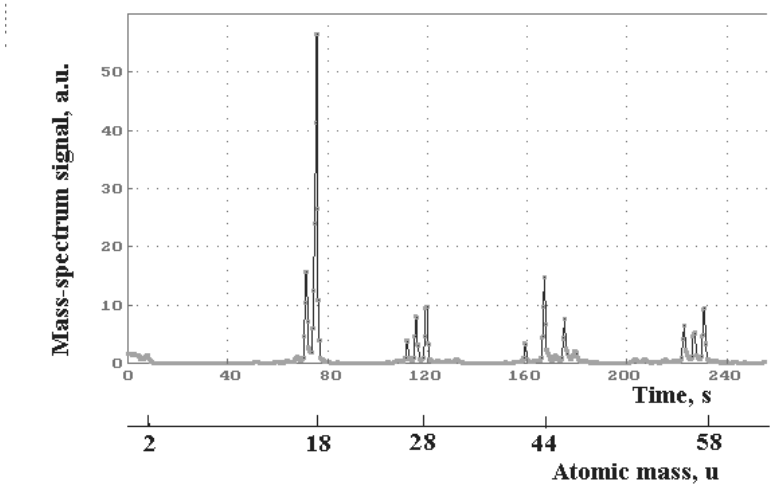

Fig.4. Mass-spectra of desorbed gases under SS probe heating to the temperature of 300^{o}C.

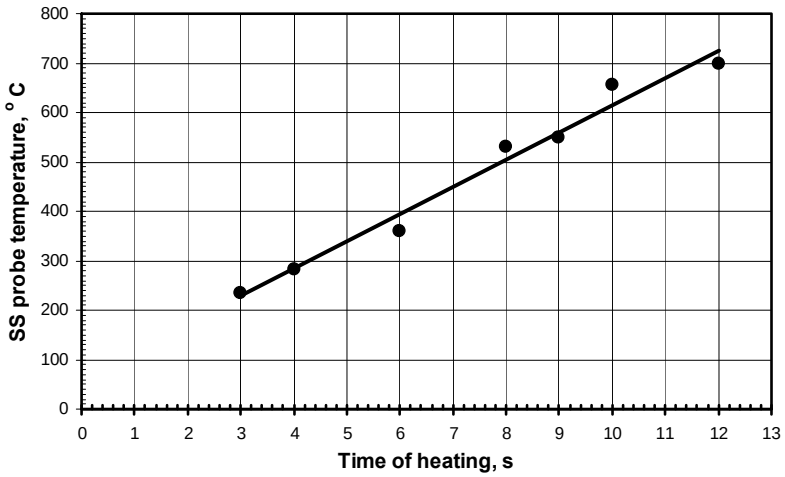

Fig.5. SS probe temperature vs time of heating with 5 Volts applied voltage.

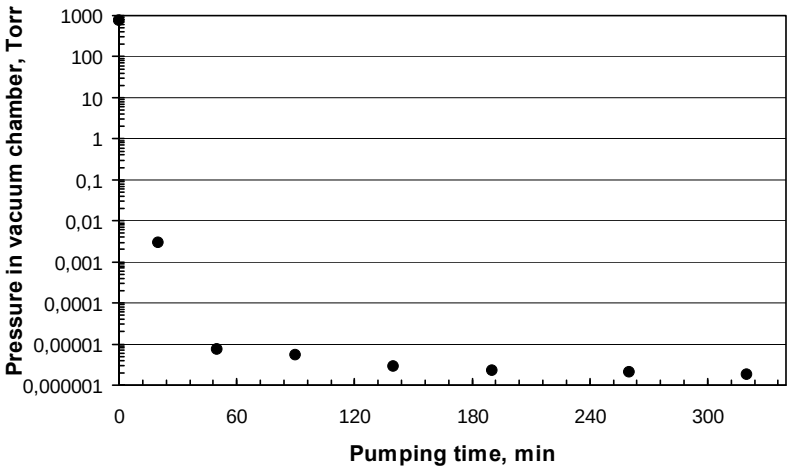

Fig.6. U-2M vacuum pumping curve.

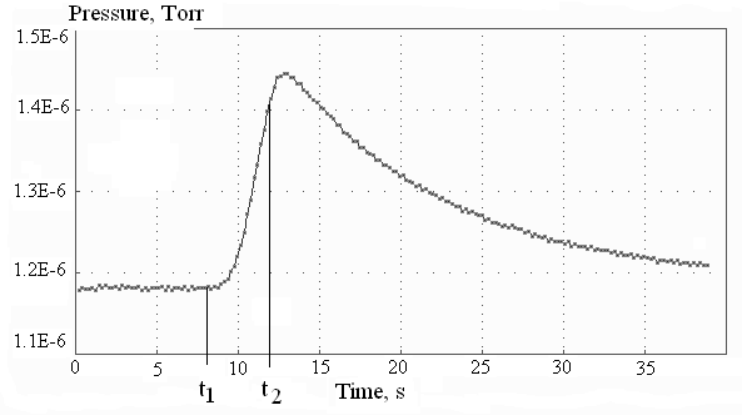

Fig.7. Apparatus curve of pressure increase in the U-2M during SS probe heating to the temperature of 300ºC: t_1 and t_2 are the times of switch on and switch off the heating, initial pressure is $1.18 \cdot 10^{-6}$ Torr.

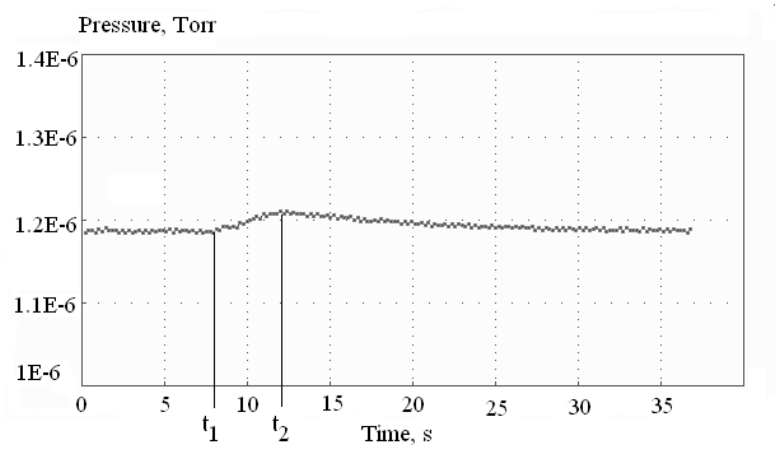

Fig.8. Apparatus curve of pressure increase in the U-2M during SS probe heating to the temperature of 300ºC after 15 minute of the first pulsed heating: t_1 and t_2 are the times of switch on and switch off the heating.

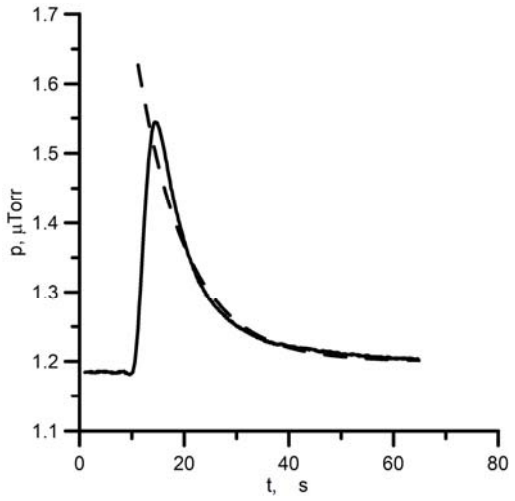

Fig.9. Pressure curve from Fig. 7 (solid line) and fitted analytical curve (dashed line).

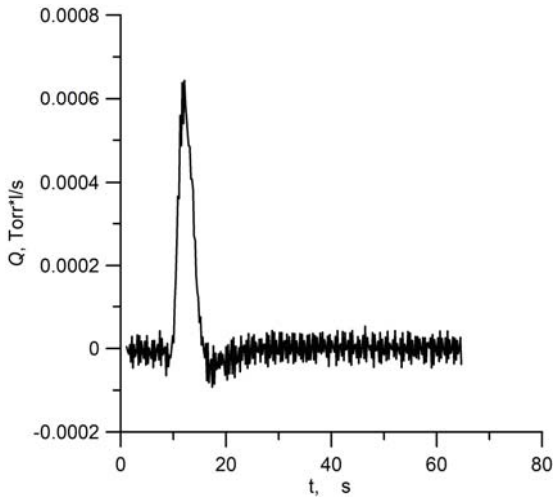

Fig.10. Cumulate outgassing calculated for the measurement displayed in Fig. 7.

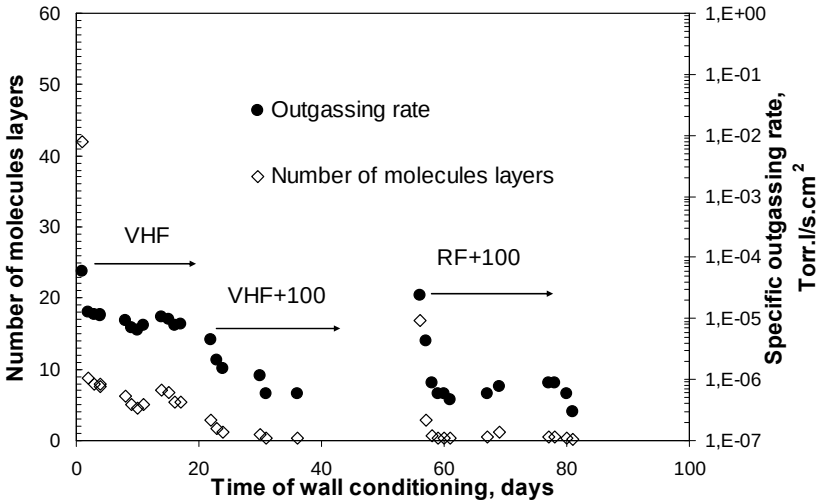

Fig.11. Uragan-2M wall conditions: black points correspond to SS probe specific outgassing rate q at 300ºC after VHF or RF discharge cleaning in H_2, N_2 or N_2+H_2 mixtures, rhombs correspond to number of molecules layers N on the SS probe.

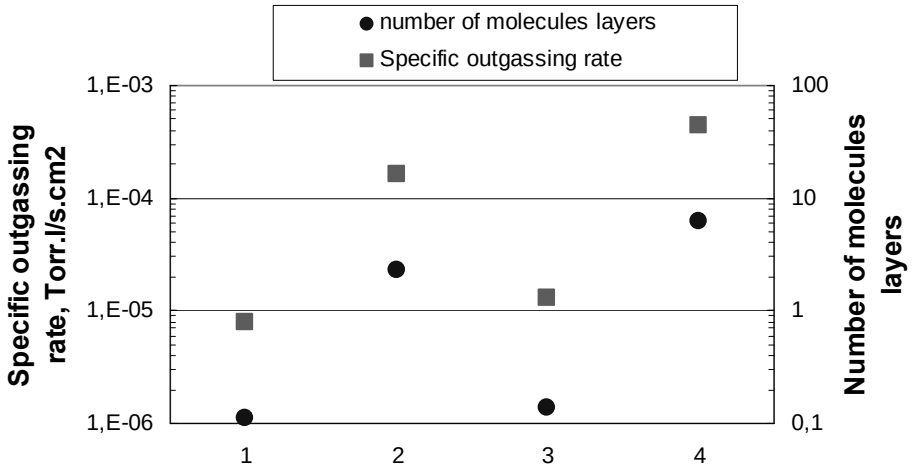

Fig.12. U-2M wall conditions in different times of measurements: points with index 1 (RF) and 3 (VHF) were measured for standard procedure of discharge cleaning just after 18 hours pumping; points 2 and 4 were obtained one hour shortly after the discharge cleaning end both for the VHF(2) or RF(4).

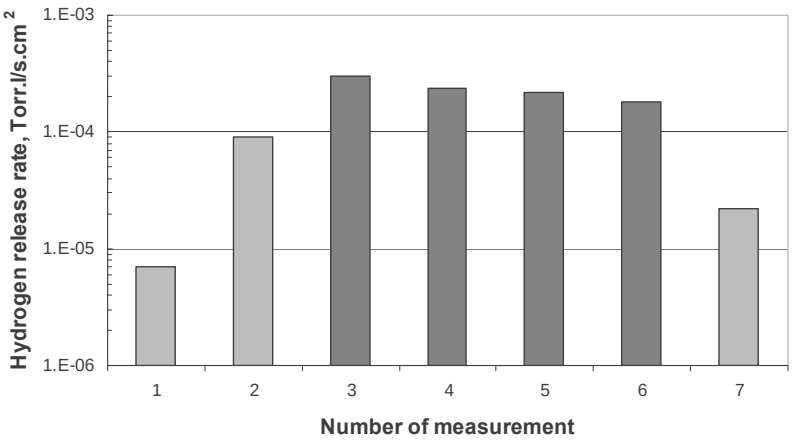

Fig.13. Hydrogen release from the SS probe:

Figure captions

Fig.1. Quantity of gas removed with ECR discharge cleaning and plasma stored energy in LHD device [1].

Fig.2. Scheme of the experiment (a) and photo of device (b): 1- U-2M vacuum chamber wall, 2- stainless steel probe, 3- current contacts, 4-ionization 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.

Fig.3. Scheme of the experimental stand for outgassing, hydrogen retention and release researches: 1- thermocouple, 2- leak valve, 3- SS probe, 4- vacuum chamber, 5- mass-spectrometer, 6- nitrogen condensation pump, 7- oil pump, 8- rotary pumps, 9- hydrogen balloon, 10-helium balloon, 11- vacuum gauges.

Fig.4. Mass-spectra of desorbed gases under SS probe heating to the temperature of 300°C.

Fig.5. SS probe temperature vs time of heating with 5 Volts applied voltage.

Fig.6. U-2M vacuum pumping curve.

Fig.7. Apparatus curve of pressure increase in the U-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.

Fig.8. Apparatus curve of pressure increase in the U-2M during SS probe heating to the temperature of 300ºC after 15 minute of the first pulsed heating: t_1 and t_2 are the times of switch on and switch off the heating.

Fig.9. Pressure curve from Fig. 7 (solid line) and fitted analytical curve (dashed line).

Fig.10. Cumulate outgassing calculated for the measurement displayed in Fig. 7.

Fig.11. Uragan-2M wall conditions: black points correspond to SS probe specific outgassing rate q at 300ºC after VHF or RF discharge cleaning in H_2, N_2 or N_2+H_2 mixtures, rhombs correspond to number of molecules layers N on the SS probe.

Fig.12. 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).

Fig.13. Hydrogen release from the SS probe:

Table 1. Maximum partial pressures of 18 u, 28 u, 44 u and 58 u during SS probe pulsed heating in U-2M torsatron.

Desorbed gas, mass	Initial pressure P_o, Torr	Maximum pressure P, Torr	ΔP, Torr
H_2O (18 u)	$3.3 \cdot 10^{-7}$	$3.9 \cdot 10^{-7}$	$6 \cdot 10^{-8}$
$CO+N_2$ (28 u)	$8.18 \cdot 10^{-9}$	$1.87 \cdot 10^{-8}$	$1.6 \cdot 10^{-8}$
CO_2 (44 u)	$4.9 \cdot 10^{-9}$	$1.68 \cdot 10^{-8}$	$1.19 \cdot 10^{-8}$
58 u	$7.72 \cdot 10^{-10}$	$1.4 \cdot 10^{-9}$	$6.28 \cdot 10^{-9}$

Author's data

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