The role of the heating of the vacuum chamber on the water content in plasma and gas

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Abstract. The behavior of the H2O concentration in stainless steel chamber is compared for the cases of gas with and without plasma discharge at different temperatures of chamber wall. The experiments were performed with the use of mixtures of rare gases and water vapor. In absence of plasma discharge the dynamics of the density of the water molecules was determined by their interaction with the surface. In the presence of a gas discharge the concentration of water molecules in the volume of the chamber was determined by optical actinometry method. The role of the chamber wall temperature is discussed.

1. Introduction

Monitoring the content of water molecules in the gas phase in vacuum and plasma discharge installations is well known problem [1]. In vacuum systems it is primarily associated with the adsorption of molecules on vessel surfaces. In this case for the relatively low temperature the chemical transformations of molecules are negligible so the adsorption can be considered as purely physical mechanism. But situation changes dramatically in the presence of the plasma discharge inside the vessel, even if it's not located close to the wall. In this case one has to take into account the non-equilibrium plasma reactions and heterogeneous phase reaction, not only between water molecules, but also between their reaction products. Up until recently, there were no methods of measurement of concentration of water molecules directly in plasma. Modern demand for those caused by the development of technology, especially in powerful electric devices, including the ITER project [2]. The reactor working conditions are limited by water leakage at the rate of 10^{-7} Pa·m^3·s^{-1} [2]. Such small flows can occur both because of the defects of the camera shell or due to the water self release from the surface when the temperature changes. Spectral methods for control of water vapor content in the cold plasma of glow discharge for searching and eliminating the source of subsequent leaks, have been recently proposed in [3, 4].

In the study of dynamics of the water vapor density in the model installation [3], it was observed that the concentration of water molecules in the plasma during the initial period (<1 min) decreases by several times with respect to the original, and then reducing of the concentration slows down. In this paper these stages are studied more detailed.

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2. Experimental setup

The device and its experimental setup were described in [3, 4]. The vacuum chamber, flanges and gas valves are made of stainless steel 12X18H10T. The profiled sealing copper gaskets are used as a vacuum tight connectors. Unlike [3], the volume of the vacuum chamber is reduced to 22000 cm^3 due to the shutdown of the sorption pump. The square of the inner surface of the vacuum chamber is 5900 cm^2. The hollow cathode, which volume is 50 cm^3, was installed to the center of chamber.

Experiments were carried out at different temperatures of the chamber wall. High temperature were obtained by using an external electric heater in the form of a flat tape with a heat-resistant insulation, which allowed to heat chamber to 295-450 K. Heating and temperature consistency (±5 K at 420 K) were controlled by thermocouples in various zones of the chamber surface.

The concentration of water molecules in the plasma discharge was measured using optical actinometry method [4] by comparing the intensities of the lines of the vibrational-rotational spectra of "hot" hydroxyl [5] in the 306 nm and line of Xe (823.2 nm). The gas mixture for discharge consisted of 99%He + 1%Xe + additive of water vapor. The gas temperature in the plasma discharge was determined from the relative intensities of lines from the "cold" hydroxyl vibrational-rotational band (also in the area of 306 nm) [3, 5].

In all cases the vacuum chamber was pumped out for several hours keeping the walls temperature T=(400-420) K before the gas injecting. Measurements were carried out either with pre-heated walls or after their cooling those to room temperature. Results are shown for the case of a mixture gas He:H_2O:Xe=99:33:1 at a total pressure of 0.5-1 mbar, the discharge current of 0.28 A.

3. Experimental results and discussion

The results of measurements are shown in figures. 1.a, 1.b for the wall temperatures 295±3 K and 395±5 K correspondingly. The initial concentration of water molecules was n_0=3.5·10^{15} cm^{-3}.
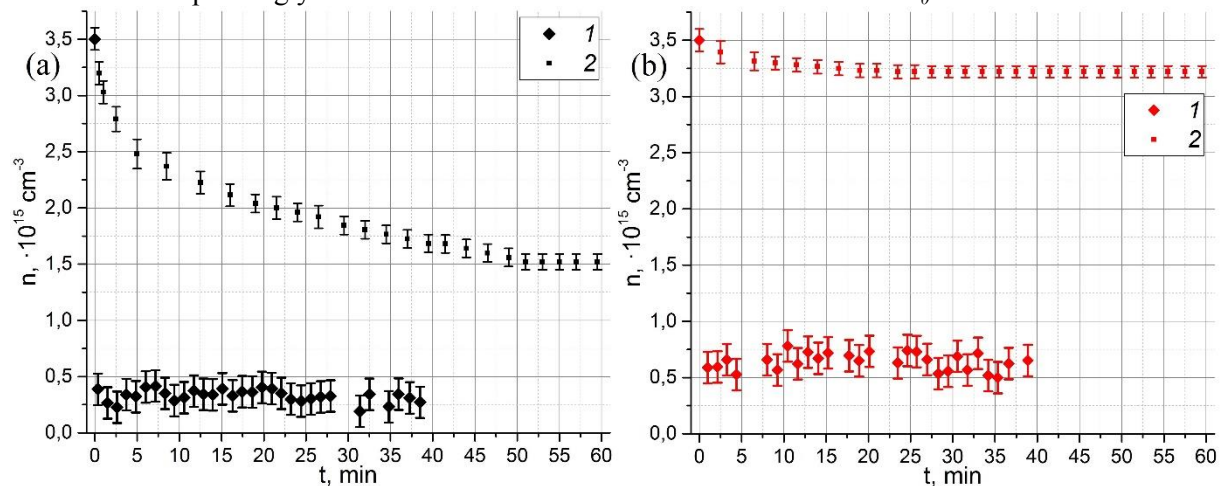

Figure 1. Concentration of water molecules changes with time. The initial concentration of water molecules was n_0=3.5·10^{15} cm^{-3}. (a) chamber without heating; (b) chamber is warmed to 395 K. *1* – with discharge, actinometrical measurements; *2* – without discharge, manometeric measurements.

Manometric measurements were carried out without the discharge. The required number of water molecules were injected to the chamber and the dynamics of water vapor density with time was investigated. The concentration of water molecules in the vacuum chamber was determined and controlled by pressure sensor. Figures 1.a.2, 1.b.2 shows that the molecular density is decreasing with time and the drop saturates after 10 minutes in warm case and after 40 min in case of cold walls. For cold case the reduction of molecules is deeper (~45%) than for the heated one (~8%). The rate and magnitude of water molecules loss depends on the wall temperature, which agrees with the assumption that the physical mechanism of adsorption of molecules on surfaces is taking place.

The measurement results confirm the results of calculations [6, 7], compared with those given in table 1. Wherein: T – the temperature of the vacuum chamber walls; N – concentration of water molecules in a state of dynamic equilibrium with the adsorbed molecules; P – partial pressure of water molecules; N_A – number of water molecules adsorbed on the chamber walls; θ – number of monolayers of adsorbed particles in the saturation stage (degree of coating); T^*, P^*, θ^* – relevant parameters from [6, 7].

Table 1. A comparison of the measurements with the calculations of the work [6, 7], no discharge in chamber. The partial initial density of water vapor n_0=3.5·10^{15} cm^{-3}.

T, K	N·10^{19}	P, Pa	N_A·10^{19}	θ	T^*, K	P^*, Pa	θ^*
295	4.8	7	2.9	3.9	295	7	4
395	7	12.1	0.7	0.9	395	12	0.7

In actinometrical measurements we take into account the thermal pushing of actinometer particles out of the discharge area, whose role in this case played a xenon [4]. The gas temperature in the discharge was measured by the intensity distribution in the rotational structure of the spectrum of "cold" hydroxyl [3]. Additional heating of the surface of the hollow cathode was controlled by radiometer, and taken into account during analysis of the results.

The behavior of water molecules in time with the discharge switched on changes significantly compared to the previous case. The concentration of molecules drops much faster with characteristic time t1 and deeper in initial phase (figures 1.a.1, 1.b.1). By the times of more than tens of minutes concentration of molecules is practically maintained in the second phase. The wall temperature does not affects on water concentration essentially on the first phase. In our opinion electron impact is responsible for the fast drop of water concentration in the first phase the dissociation. In the work [3] the rate constant $<v_e·\sigma_{e,diss}>$ of the process of dissociative excitation of the H_2O molecule (v_e – electron velocity, $\sigma_{e,diss}$ – cross section of dissociative excitation of the molecule H_2O) was measured under similar conditions and the corresponding characteristic dissociation time was found equal $\tau_{diss}=(<v_e·\sigma_{e,diss}>)^{-1}$~5 s. This time up to a factor 2-3 coincide with those that we observed in the first phase $t_1 \approx \tau_{diss}$.

The second quasi stationary phase also reveals some features which does not agree with predictions of thermodynamic adsorption theory. In the framework of this theory the total balance of particles remain constant and the amount of particles split up between the free volume molecule and the molecules captured by the surface. It becomes obvious if one compare the data from figures 1.a.1, 1.b.1 and from the table 1. In the absence of discharge the change of the wall temperature from 295 K to 395 K leads to releasing ~2·10^{15} cm^{-3} molecules from the surface to the bulk. In the presence of discharge the same wall heating leads to releasing ~0.3·10^{15} cm^{-3} molecules which about 10 times less. It definitely means that the structure of surface layers get modified by discharge despite the fact that the low power (50 W) discharge in our experiments is located quite far from the walls. The probable explanation is that the discharge generate the active particles which reacts with surface particles thus transform the adsorption mechanism from pure physical to more complicated one so formally $\theta_T \neq \theta_P$, where θ_T, θ_P – numbers of absorbed monolayers in case of thermal equilibrium and in case of plasma discharge influence respectively.

4. Conclusion

The behavior of water vapor concentration with time in vacuum chamber with stainless steel walls was studied. The measurement results are correspond to the physical adsorption theory [6, 7] at different temperature conditions. The local plasma source in vacuum chamber far from its wall significantly changes the dynamics of the density of the water vapor. That is caused by the process of emergence of a new active particles in a plasma-chemical decomposition of the starting molecules which is faster than the adsorption process. Measurements lead to the conclusion that the presence of these particles can significantly change the particle desorption mechanism on the surface.

Acknowledgements

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