https://doi.org/10.46813/2024-149-038

ORBITRON PUMP WITH NITROGEN CRYOPANEL

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The article presents the results of a study of the operation of a vacuum orbitron getter-ion pump (OGIP) with a nitrogen cryopanel. The design features of the orbitron pump and its operating modes are described. The composition of the residual atmosphere (MX7304A mass spectrometer) of the vacuum chamber during its pumping out by the OGIP to ultrahigh vacuum has been studied. It has been established that there are no heavy hydrocarbons in the chamber, and methane is not its main component and does not determine the ultimate pressure of the pump. In an insufficiently heated vacuum chamber at $P = 2 \cdot 10^{-8}$ Pa, the main component of the residual atmosphere is water vapor. Therefore, with further thorough degassing of the vacuum chamber, the use of OGIP with a nitrogen cryopanel makes it possible to obtain an ultra-high oil-free vacuum better than 10^{-8} Pa.

PACS: 07.75.+h; 32.10.Bi; 33.15.Ta

INTRODUCTION

Further development of electronics, spintronics, nanotechnology, technologies for obtaining ultrapure substances, etc., puts forward increased requirements for the quality of the vacuum environment and, above all, for the depth and purity of the vacuum. This leads to the improvement of the existing means of obtaining a vacuum and its diagnostics and the creation of fundamentally new ones. In this regard, it should be noted that no “breakthrough” developments have taken place in vacuum technology over the past 20–25 years. The main successes in the development of vacuum technology in these years are associated mainly with the miniaturization of various electronic devices used in vacuum equipment and the widespread use of computer technology. Regarding vacuum pumps for obtaining and maintaining an ultra-high (less than 10^{-7} Pa) vacuum, it should be noted that of the five main types of vacuum pumps inherited from the vacuum technology of the 20th century (diffusion pumps, turbine vacuum pumps, sputter ion pumps, cryo pumps and electrophysical vacuum pumps), only the last two types of pumps most fully meet modern requirements for ultrahigh vacuum technology [1–4]. Cryovacuum pumps remain the leaders among modern vacuum pumps both in terms of the depth of the ultimate vacuum (less than 10^{-10} Pa) and its quality (complete absence of hydrocarbons). However, the high cost, complexity of obtaining and storing the main refrigerant for these pumps, namely liquid helium, limits their application only to large research centers.

Electrophysical (in other words, getter ion) pumps also have quite acceptable vacuum characteristics: the ultimate vacuum is not worse than 10^{-10} Pa in the complete absence of heavy hydrocarbons [5].

The aim of this work was to study the features of obtaining an ultrahigh oil-free vacuum using an orbitron-type getter ion vacuum pump with a nitrogen cryopanel, which we have created as one of the promising types of electrophysical pumps.

1. THEORY/CALCULATION

1.1. SUBLIMATION VACUUM PUMPS

The getter ion vacuum pump is a combination pump. It integrates sublimation vacuum pump and ion vacuum pump. The evacuation effect of sublimation pumps is based on the use of the getter properties of a film obtained by condensation of thermally evaporated atoms of active metals (Ba, Ti, Ta, Mo, Mg, Hf, Zr, Er, Y). To date, more than ten modifications of evaporative pumps have been developed, differing in the initial active metals and methods of their evaporation. A detailed description of the designs and analysis of the features of the pumping mechanisms of such pumps are given, for example, in [5].

In most modern sublimation pumps, titanium is the starting getter metal. Titanium forms strong non-volatile compounds and solid solutions with the atoms of almost all gases that are in vacuum systems, with the exception of inert ones. In 1962, the effect of a significant improvement in the absorbing properties of a titanium film for most gases was discovered when it condenses on a surface cooled to T = 77 K with liquid nitrogen (nitrogen cryopanel) [6].

Due to the significant selectivity of the absorption of various gases by the titanium film, the residual atmosphere of titanium sublimation pumps is enriched mainly in inert gases and CH_4 methane. Methane is formed in the pump itself during its operation, its amount determines the ultimate pressure of the pump [5, 7]. The absorption of methane is insignificant even at the temperature of the titanium film T = 77 K. Note that in the case of pumping out a mixture of different gases in the processes of absorption of their molecules, competition and discrimination arise. Due to the complexity of the study of these processes, the data of different authors on the evacuation of a gas mixture is extremely contradictory [5, 8].

1.2. GETTER ION ORBITRON PUMPS

In getter ion pumps, the pumping out of gas molecules by a titanium film is improved due to the ionization of some of them by accelerated electrons.

Historically, the first to be created were the simplest getter ion pumps, in which the titanium evaporator and the gas molecule ionizer operate independently of each other and are not functionally interconnected [4, 5]. Unfortunately, in these pumps the path lengths of ionizing electrons are small, the probability of ionizing gas molecules by them is insignificant, and the rate of pumping out of inert gases is very low. This significant disadvantage of such pumps is overcome in getter ion orbitron pumps.

For the first time the orbitron pump was proposed by Herb in 1965 [9]. The principle of operation of the orbitron pump is described in detail in [2, 5, 9]. In orbitron pumps titanium is sputtered by an electron beam method. The source of electrons is a hot cathode and the electrons emitted by it are used both for ionization of gas molecules and for sputtering titanium. Titanium atoms are deposited on the inner surface of the pump body wall and form an adsorbing titanium film on it. The active gases are evacuated by a pump due to the chemisorption of a continuously renewable titanium film. The electrons in the pump move along complex spatial paths (orbits) around the anode and, as a result, pass a long way before hitting it. Inert gases are pumped out by the pump as a result of ionization of their atoms by electrons moving in the pump cavity, further accelerated movement of the formed positive ions to the negatively charged wall of the pump body, their subsequent implantation into a titanium film and “immure” in it with settling Ti atoms.

The electrons moving in the pump ionize a certain part of the molecules of active gases and molecules of heavy hydrocarbons. The resulting ions fall apart into charged fragments, which are also sorbed by the titanium film according to the above mechanism. Thus, the evacuation mechanism of the orbitron pump combines the chemisorption evacuation mechanism of the sublimation titanium pump and the evacuation mechanism of the ion transfer vacuum pump.

The pumping rate of orbitron pumps is mainly determined by the size of the surface area of the pump wall covered with a titanium film and the capture coefficient [5]. Ultimate pressure of orbitron vacuum pumps with cooling of the pump body (titanium film) with running water is about 10^{-7} Pa. The use of a nitrogen cryopanel ($T = 77$ K), on which a part of the Ti atoms is deposited, makes it possible to obtain the ultimate pressure of vacuum orbitron pumps of the order of 10^{-10} Pa.

The orbitron pumps are UHV pumps that require roughing to $\sim 10^{-2}$ Pa. They produce clean (dry) vacuum, do not generate noise, are vibration free, do not induce magnetic fields, and have small mass. The absence of magnetic field and low mass is their great advantage over the sputter on pumps that need large permanent magnets. Although the orbitron pumps work in any position, the most suitable installation of the pump is a vertical one with the electrical feedthroughs up. In this position, peeling the getter layers cannot cause short circuits between the electrodes and ground. The pumps are simple in design and maintenance. All of their components are easily accessible. This makes it possible to manufacture and operate orbitron pumps in small research laboratories. In 1970–1990, in many countries, several varieties of such pumps, which were produced in small quantities, were created [1, 2, 4, 5]. In Ukraine, pioneering work on the study of the features of the orbitron pump’s residual atmosphere composition was carried out at Kharkov University under the leadership of Z. Zyman [10]. In recent years, there have been reports of the development of miniature orbitron pumps built into various MEMS [11].

2. MATERIAL AND METHODS

To study the operation of the orbitron pump, an all-metal ultra-high vacuum plant was used. Its appearance is shown in Fig. 1. The main elements of the plant are an orbitron pump, a diode sputter ion pump НМД-0.16 type, a system of three adsorption carbon vacuum pumps for creating a preliminary oil-free vacuum and a cylindrical vacuum chamber with a volume of about 20 l/s.

The design of the plant provides for independent pumping of any of the system elements to a pre-vacuum of 10^{-2} Pa by adsorption pumps. Orbitron pump and sputter ion pump are connected to the vacuum chamber by vacuum valves with diameter of conventional passage from 60 mm. The orbitron pump body, sputter ion pump body, high vacuum valve bodies and the vacuum chamber can be heated up to 300 °C by electric heaters, and the orbitron pumps body and vacuum chamber can also be cooled with running water.

The preliminary vacuum is measured using the thermocouple gauge, the high vacuum in the chamber and pumps is measured using the cold cathode magnetron gauges. The composition of the residual gases in the vacuum chamber is determined by the single-pole mass-spectrometer of the MX7304A type. Ultimate pressure (better than $1 \cdot 10^{-8}$ Pa) in the chamber is created as a result of pumping it out with degassing heating up to 300 °C for 10…15 h.

2.1. DESIGN FEATURES OF THE ORBITRON PUMPS

The orbitron pump we created, shown in Fig. 2, consists of three main units: a body (1) with water cooling, a flange with an electrode system (2), and a compartment (3) for a nitrogen cryopanel. Through the branch pipe (5), the pump is connected to the vacuum system, to the branch pipe (4) the cold cathode magnetron gauge is connected to measure the vacuum in it.

The anode rod (7) is made of a tungsten wire with a diameter of 2 mm; a small cylinder (8) of titanium iodide is fixed to the rod. The tungsten-rhenium filament of the cathode (10) has a diameter of 0.12 mm. The pump has two identical cathodes – working and standby. The screen plate (9) and the screen tube are made of stainless steel. Elements of the electrode system are assembled on vacuum feedthrough of the base (11). The operating position of the pump is vertical with a plinth at the top (see Fig. 2,a,b). In the lower part of the pump, in a special compartment (3), there is a nitrogen cryopanel (6) formed by the inner (facing the anode) surface of the container for liquid nitrogen. The container is made of thin stainless steel sheet.

The Ti atoms evaporated from the heated titanium cylinder (8) are deposited both on the side surface of the inner wall of the pump body (1) (water cooling) and on the inner surfaces of the ontainer with liquid nitrogen (cooling to T = 77 K). The container for liquid nitrogen has an inner diameter slightly larger than the diameter of the pump body.

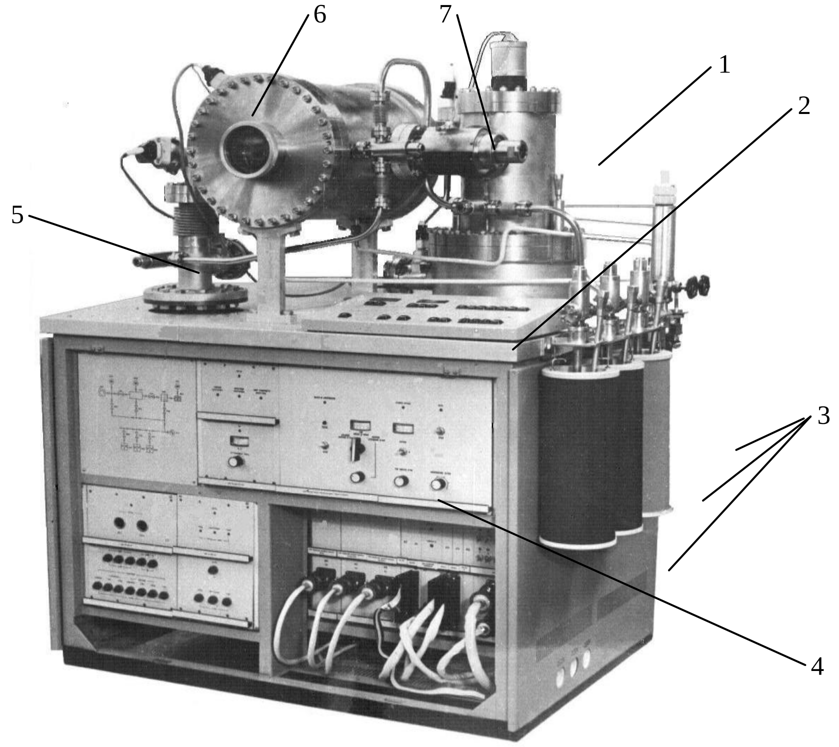


Fig. 1. External view of the ultra-high vacuum plant: 1 – orbitron pump; 2 – control panel and gauge control unit; 3 – adsorption vacuum pumps; 4 – power supply of orbitron pump; 5 – vacuum pipework of sputter ion pump; 6 – vacuum chamber; 7 – vacuum valve

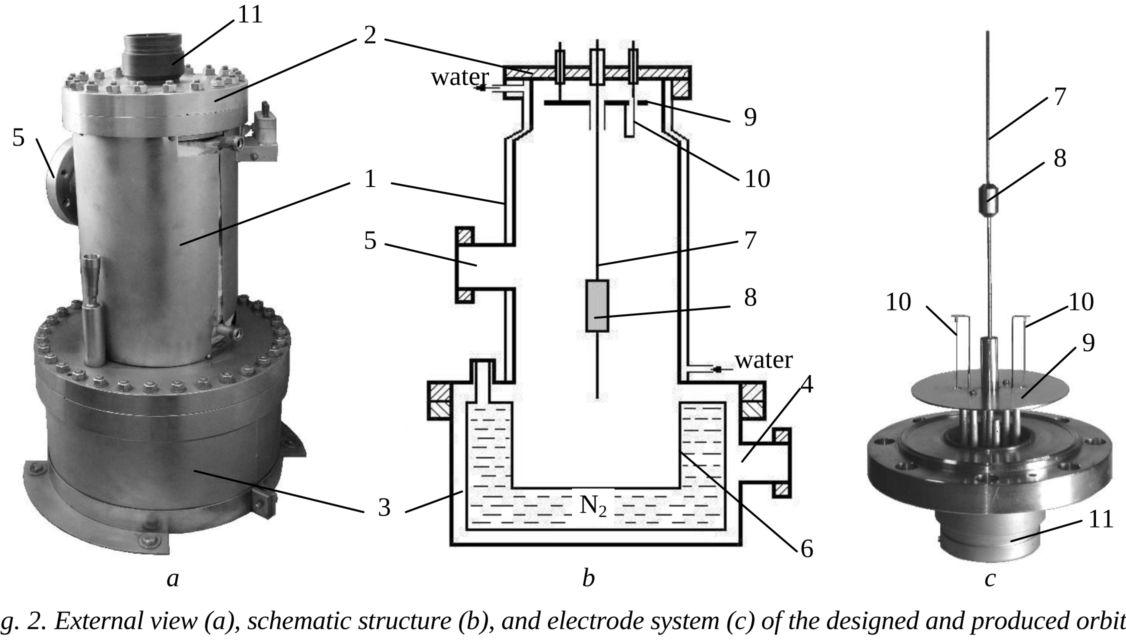


Fig. 2. External view (a), schematic structure (b), and electrode system (c) of the designed and produced orbitron pump: 1 – body; 2 – electrode flange; 3 – compartment for nitrogen cryopanel; 4 – pipe for cold cathode magnetron gauge; 5 – connecting pipe; 6 – nitrogen cryopanel; 7 – anode; 8 – titanium cylinder; 9 – screen plate; 10 – cathode; 11 – base

Then pieces of titanium film, which over time can peel off from the wall of the pump body, fall to its bottom and are there at a low temperature (thereby reducing their gas evolution).

The pump is powered from an electronic unit, the circuit diagram of which is similar to that described in [9]. The anode voltage U_a can vary within 0…8 kV, the cathode filament current I_c is within 0…3.2 A. The calculated volume flow rate of the vacuum pump for air is about 500 l/s when the pump body is cooled with running water and about 600 l/s when using a nitrogen cryopanel. The ultimate pressure of vacuum pump when cooling the pump body with running water is 10^{-7} Pa, when using a nitrogen cryopanel – no worse than 10^{-9} Pa.

3. RESULTS AND DISCUSSION

Vacuum parameters of the pump (volumeflow rate and ultimate pressure) significantly depend on the operating modes of its electrode system. Sublimation of titanium atoms from the surface of a titanium cylinder occurs at a certain power of the electron beam, which can be changed in three ways: by changing the cathode filament current (I_c), changing the anode voltage (U_a), and changing the bias voltage (U_b). At low filament currents of the cathode, the number of electrons is insufficient for noticeable heating of the titanium cylinder. In this mode, titanium does not evaporate and the orbitron pump works like an ion vacuum pump. A similar situation can be created with the optimal filament current of the cathode by reducing the anode voltage to $U_a = 1…2$ kV. Thus, if necessary, the orbitron pump can be switched to “watch” mode of operation, when it is only necessary to maintain a high vacuum in the recipient with low gas evolution. The power released at the anode can also be controlled by changing the bias voltage U_b. In this case, the place of focusing of the electron beam on the anode changes and the shape of the electron orbits changes. The latter significantly affects the efficiency of ion pumping.

The orbitron pump, switched off and cut off from the vacuum system, maintains a high vacuum for many weeks, which allows it to be started without pumping out to the preliminary vacuum.

Before discussing the results of mass spectrometric studies of the composition of the residual atmosphere (RA) of the vacuum chamber when it is pumped out by the orbitron pump (Fig. 3), the following should be noted. Since the gas that is evacuated from the vacuum chamber is air, then its RA will contain the main components of air – nitrogen, oxygen, argon and water vapor. The air mass spectrum contains seven main mass spectrometric peaks with masses of 28 amu (100%), 32 amu (27%), 14 amu (6%), 16 amu (3%), and 40 amu (1%), which belong to the N_2^+, O_2^+, N^+, O^+, and Ar^+ ions, respectively (the percentages of peak intensities relative to the N_2 peak intensity are shown in parentheses), and, in addition, peaks with masses of 18 and 17 amu, which belong to the H_2O^+ and HO^+ ions, respectively. In the absence of a leak in a vacuum system in the mass spectrum of its RA, the intensity ratios of these peaks will depend, first of all, on the type of high-vacuum pumps used. The presence of other mass spectrometric peaks will depend on the degree of degassing of the walls of the vacuum chamber, on the materials used in the design of the vacuum plant, on the temperature of the walls of the chamber and other units of the vacuum system [12].

Fig. 3,a shows, for illustration, the mass spectrum of the composition of the RA of a metal vacuum chamber when it is pumped out by a diffusion oil-steam pump. The most intense peaks with M > 39 amu, presented in the Table, correspond to fragments of organic molecules of working fluids of diffusion and mechanical vacuum pumps.

The mass spectrum of RA when evacuating the vacuum chamber with a sputter ion pump (see Fig. 3,b) at $P = 2·10^{-7}$ Pa in the chamber is quite simple. The main mass spectrometric peaks are the peak of molecular ions of water vapor H_2O with M = 18 amu and peaks of fragments of its molecule with M = 17 amu (HO) and M = 16 amu (O). Since sputter ion pumps have significant pumping selectivity, in the mass spectrum the peak with M = 32 amu (O_2) is completely absent, but there are peaks with M = 4 amu (He) and M = 40 amu (Ar). Peak with M = 2 amu (H_2) mainly appears in the mass spectrum due to the fragmentation of the H_2O molecule in the ion source of the mass spectrometer. Mass spectrometric peaks with M = 28 amu and M = 44 amu belong to CO and CO_2, respectively. These gases appear in the composition of the RA vacuum chamber mainly as a result of their desorption from its walls. N_2^+ ions make a small contribution to the peak with M = 28 amu.

Mass numbers and chemical formulas of fragments of molecules of vacuum oils [12]

M, amu	Chemical formula of fragment
39	C_3H_3
41	C_3H_5
43	C_3H_7
55	C_4H_7
57	C_4H_9
68	C_5H_3
71	C_5H_4
81	C_6H_9
83	C_6H_{11}
95	C_7H_{11}
97	C_7H_{13}

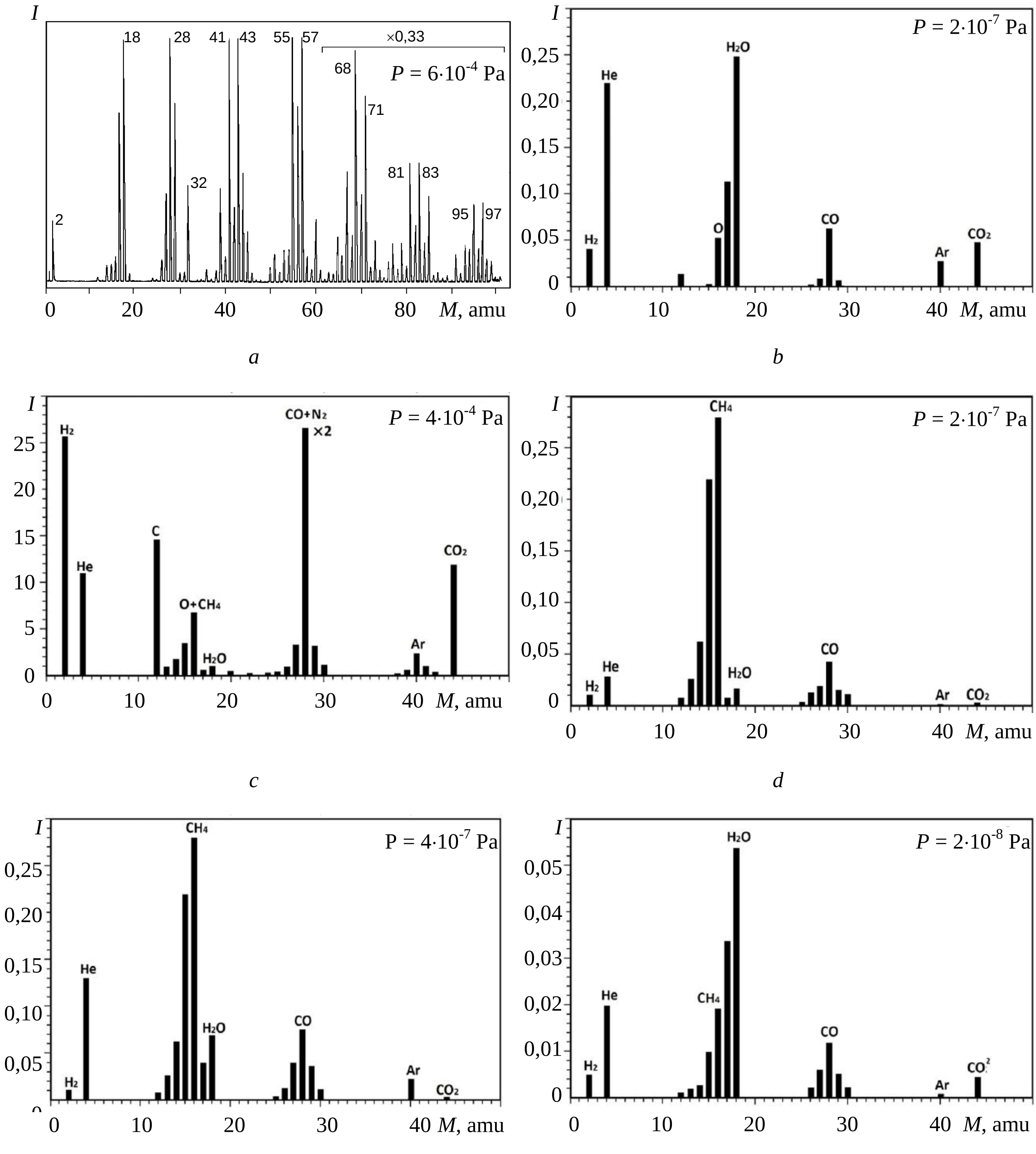
In Fig. 3,c shows the mass spectrum of the RA of a heated vacuum chamber, cut off from the pumps in the absence of a leak. The main mass spectrometric peaks belong to the molecular hydrogen ion H_2, carbon monoxide and carbon dioxide CO and CO_2. The peaks of the inert gases He and Ar with an insignificant amount of water vapor H_2O are significant. Such a composition of RA is the result of both gas evolution from the walls of the vacuum chamber (H_2, CO, and CO_2) and the selectivity of pumping out air components by pumps.

In Fig. 3,d shows the mass spectrum of the RA of the vacuum chamber at a pressure in it $P = 2·10^{-7}$ Pa when it is pumped out by the orbitron pump in its main operating mode ($U_a = 7.5$ kV, $I_a = 25$ mA, $I_c = 2.6$ A, $U_b = 50$ V, power consumption in the anode circuit is about 200 W) with water cooling of its body. The main component of RA is methane CH_4 with M = 16 amu. Its quantity determines the ultimate pressure of the pump.

When the orbitron pump operates in the “watch” mode ($U_a = 2$ kV, $I_a = 5$ mA, $I_c = 2.4$ A, power consumption in the anode circuit is about 10 W), the pump maintains a vacuum in the chamber $P = 4·10^{-7}$ Pa. From the mass spectrum of RA (see Fig. 3,e), we see that its main constituent is again methane, as well as inert helium, which is practically not pumped out in this operating mode of the pump.

The most interesting is the mass spectrum of the RA vacuum chamber when it is pumped out by the orbitron pump in the same main operating mode and the pressure in it P = $2\cdot10^{-8}$ Pa (see Fig. 3,f), but using a nitrogen cryopanel. Now the gas that determines the ultimate pressure in the chamber is water vapor H_2O, not methane CH_4.

In our opinion, the reason for this is a significant decrease in the intensity of the chemical reaction of methane synthesis on the surface of the sorbing titanium film when it is cooled to T = 77 K compared to T = 290 K, since pumping out methane with a titanium film at T = 77 K is ineffective.



a

b

c

d

e

f

Fig. 3. Mass spectra of the residual atmosphere of the vacuum chamber during its evacuation: with a diffusion pump (a); sputter ion pump (b); water-cooled orbitron pump (d); orbitron pump in "watch" mode (e); orbitron pump with nitrogen cryopanel (f). Mass spectrum of the residual atmosphere of the vacuum chamber cut off from the pumps (c)

CONCLUSIONS

Based on the results of the studies carried out on the operation of the orbitron pump with a nitrogen cryopanel, important practical conclusions can be note:

1. In the mass spectra of the residual atmosphere of the vacuum chamber when it is pumped out by the orbitron pump with a nitrogen cryopanel, the mass spectrometric peaks of heavy hydrocarbon molecules and their fragments are completely absent.
2. Methane CH_4 in the composition of the residual atmosphere of the vacuum chamber during its pumping out orbitron pump with a nitrogen cryopanel is not its main component and its amount does not determine the ultimate pressure of the pump.
3. In an insufficiently heated vacuum chamber at $P = 2 \cdot 10^{-8}$ Pa, when it is pumped out by an orbitron pump with a nitrogen cryopanel, he main component of its residual atmosphere is water vapor H_2O. With a decrease in the amount of desorbed water vapor molecules from the walls of the vacuum chamber by prolonged heating to 250…300 °C while pumping it out by orbitron pump with a nitrogen cryopanel, further improvement of the vacuum in it is possible.

Thus, the orbitron pump with a nitrogen cryopanel developed by us is an effective pumping device for creating an ultra-high (better than 10^{-7} Pa) oil-free vacuum in heated metal vacuum installations.

REFERENCES

1. I. Bello. *Vacuum and Ultravacuum: Physics and Technology*. London: “CRC Press Taylor & Francis Group”, 2018, 1062 p.
2. K. Jousten. *Handbook of Vacuum Technology*. Weinheim: “Wiley-VCH Verlag GmbH & Co”, 2016, 1050 p.
3. N. Yoshimura. *Vacuum Technology Practice for Scientific Instruments*. Berlin: “Springer-Verlag”, 2008, 353 p.
4. К.Е. Демихов, Ю.В. Панфилов. *Вакуумная техника:* Справочник. М.: «Машиностроение», 2009, 590 с.
5. Г.Л. Саксаганский. *Электрофизические вакуумные насосы*. М.: «Энергоатомиздат», 1988, 280 с.
6. R.A. Haefer. *Kryo-Vakuum technik: Grundlagenund Anwendungen*. Berlin: “Springer-Verlag”, 1981, 332 p.
7. D. Edwards, Jr. Methane outgassing from a Ti sublimation pump // *J. Vac. Sci. Technol.* 1980, v. 17, issue 1, p. 279-281; https://doi.org/10.1116/1.570412
8. D.J. Harra. Review of sticking coefficient sand sorption capacities of gases on titanium films // *J. Vac. Sci. Technol.* 1976, v. 13, issue 1, p. 471-474; https://doi.org/10.1116/1.568900
9. R.A. Douglas, J. Zabritski, R.G. Herb. Orbitron Vacuum Pump // *Review of Scientific Instruments*. 1965, v. 36, issue 1, p. 1-6; https://doi.org/10.1063/1.1719315
10. З.З. Зыман, В.Г. Горожанкин, Б.Г. Сафронов. О путях понижения давления легких углеводородов в орбитронном насосе // *Вопросы атомной науки и техники. Серия «Физика и техника высокого вакуума»*. 1976, в. 1(5), с. 38-41.
11. T. Grzebyk, A. Gyrecka-Drzazga. Field-emission electron source for vacuum micropump // *Vacuum*. 2011, v. 86, issue 1, p. 39-43; https://doi.org/10.1016/j.vacuum.2011.04.010
12. G.S. Anufriev, B.S. Boltenkov, A.I. Ryabinkov. High-resolution mass spectra of the residual gas in a metallic vacuum system // *Technical Physics*. 2006, v. 51, p. 100-111; https://doi.org/10.1134/S1063784206010154

Article received 03.05.2023

ОРБІТРОННИЙ НАСОС З АЗОТНОЮ КРІОПАНЕЛЛЮ

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Наведено результати дослідження роботи вакуумного орбітронного геттерно-іонного насоса (ОГІН) з азотною кріопанеллю. Описано конструктивні особливості насоса та режими його роботи. Досліджено склад залишкової атмосфери (мас-спектрометр МХ7304А) вакуумної камери при її відкачуванні ОГІН до надвисокого вакууму. Встановлено, що в камері відсутні важкі вуглеводні, метан не є її основним компонентом і не визначає граничний тиск насоса. У недостатньо прогрітій вакуумній камері при $P = 2 \cdot 10^{-8}$ Па основним компонентом залишкової атмосфери є водяна пара. Тому при подальшій ретельній дегазації вакуумної камери використання ОГІН з азотною кріопанеллю дозволяє отримати надвисокий безмасляний вакуум краще 10^{-8} Па.