ANALYZING THE CONTENTS OF RESIDUAL AND PLASMA-SUPPORTING GASES INSIDE A VACUUM DEPOSITION UNIT CHAMBER*

A.Ye. Mikheev[1], V.A. Kharlamov[2], S.D. Kruchek[2], A.A. Cherniatina[2], I.I. Khomenko[1]
[1]Siberian State Aerospace University named after Academician M.F.Reshetnev
31 "KrasnoyarskiyRabochiy" prospect, Krasnoyarsk, 660014, Russia.
[2]JSC "Academician M.F. Reshetnev Information Satellite Systems"
52 ul. Lenina, Zheleznogorsk, Kranoyarskii krai Russia.

E-mail: michla@mail.ru, vah@iss-reshetnev.ru

Abstract. The paper describes a quadruple mass-spectrometer method, which is used to analyze the content of residual gas in a vacuum chamber of the ARM NTM (Automatised Working Area) ion-plasma unit. This unit is used to perfect the magnetron deposition process for coating radio-reflecting surfaces. The intake of pure argon into the chamber revealed up to 0.3 % of impurities in the plasma-supporting gas, including 0.02 % of water and oxygen. A significant presence of hydrocarbon gases is explained by the presence of solvents sorbed in rubber washers, joints of internal equipment, and other components inside the chamber. In order to decrease the level of impurities in the plasma-supporting atmosphere inside the chamber and improve the composition and properties of the coatings, it is necessary to take additional measures to cleanse and degas the surface of the chamber from condensation products and hydrocarbon compounds. To provide a minimal level of impurities in the coated surfaces it is vital to clean and degas the surfaces of the chamber, removing residual moisture and hydrocarbon compounds.

Target dispersing and coating condensation processes are conducted in a gaseous environment, containing residual components of the vacuum chamber atmosphere. Inleakage, wall desorption, gas emissions from spluttered components and specimens cause the residual atmosphere to intake various chemically active gases, e.g.: water, oxygen, hydrocarbons, hydrogen, and carbon dioxide. The quantity of these elements and compounds depends on a number of factors. In laboratory and industrial apparatuses operating in vacuum not exceeding 10^{-4} Pa the sum of partial water, nitrogen, and oxygen pressures normally comprises a greater part from residual gas pressure [1]. The degree of impurities in the plasma-supporting gas depends on the residual gas pressure; it is individual for each vacuum chamber and depends on its state before deposition, along with the properties and amount of components prepared for vacuum depositing and the operating pressure of the plasma during the process of

*This research has been made possible through the financial support from the Ministry of Education and Science of the Russian Federation. State contact № 02.G25.31.0043.

depositing targets. The typical pressure levels during a magnetron process of depositing aluminum onto a carbon fiber component are as such: the residual gas pressure is $(1–10)\times10^{-4}$ Pa; the operating plasma pressure is 0.07–0.2 Pa. Thus, the expected volume of additives is from 0.05 to 1.5 %. As these active components – including chemically active gases – are drawn into the plasma deposition operating area, the following chemical compounds are formed with the target's material – oxides, nitrides, carbides. These substances might significantly change the properties of the deposited surface [2].

Analyses of the residual and plasma-supporting gases composition were conducted in the chamber of the ARM NTP vacuum unit – previously used for developing multilayer radio-reflecting deposition techniques. A diagram of the unit is shown in Figure 1.

The unit is primarily used for depositing multilayer thin-film coatings onto surfaces of various templates, the ion-flow processing of material specimens, performing ionic-plasma experimentations, and testing technological processes, applied during deposition on different surfaces.

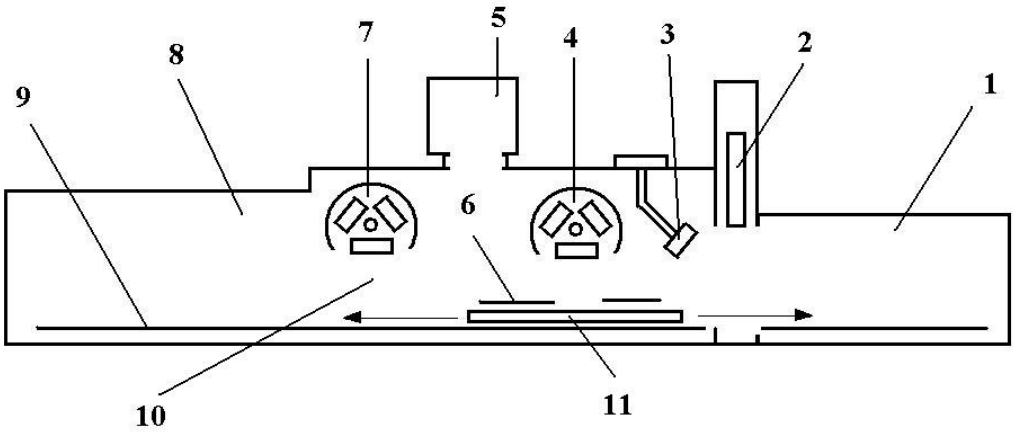

Figure 1. Diagram of the vacuum chamber of the ARM NTP unit:
1 – air lock; 2 – vacuum seal; 3 – ion source with a continuous drift of electrons; 4, 7 – magnetron unit; 5 – source of high-energy gas ions; 6 – limiting slit; 8 – reverse chamber; 9 – rails; 10 – operating chamber; 11 – transposition system surface.

The chamber of the ARM NTP unit consists out of an air lock chamber and an operating chamber. The operating chamber is used for conducting magnetron deposition and plasma treatment of specimens. The chamber is equipped with magnetron and ion sources, reciprocating and rotatory systems for moving templates, and screens for limiting the deposition area for the magnetron targets. Mechanical and turbomolecular pumps are used to pump out the chambers.

The operating chamber has a significant internal surface area; as a result, during deposition operations, the surfaces on the chamber walls, screens, and components inside the chamber become covered in condensation products from the deposited substances. Replacement of targets and maintenance operations inside the chamber expose it to the atmosphere. As a result, the condensates become a source of water and atmospheric components such as nitrogen, oxygen, carbon dioxide. When added to the gases generated by templates during the initial treatment and deposition processes, these substances are found in the volume of gas, pumped from the chamber.

Prior to conducing radio-reflecting aluminium coating deposition treatment, the ARM NTP unit undergoes certain maintenance. During this process, the internal surfaces, components and screens of the chamber are cleaned of condensation products and other foreign materials. The surfaces are then wiped clean using oil solvent and ethyl alcohol.

In order to provide the monitoring of residual gas, an HPQ-2S quadruple mass-spectrometer has been installed on one of the flanges. The device resisters mass spectrum within a range from 1 to 100 Da with a maximum working pressure at 1 Pa.

The spectra mass of the chamber had been registered prior to operation and after the installation of necessary targets into the magnetrons and cleaning the screens and surfaces of the chamber, performed with high quality argon (Russian state standard nomenclature: GOST 10157-79) being pumped into the chamber to enable a plasma-supporting environment.

Fig. 2 depicts the mass spectra of residual gas after vacuum pumping to a pressure of 1.5×10^{-4} Pa. Fig. 3 shows the spectra of plasma-supporting gas in the presence of argon.

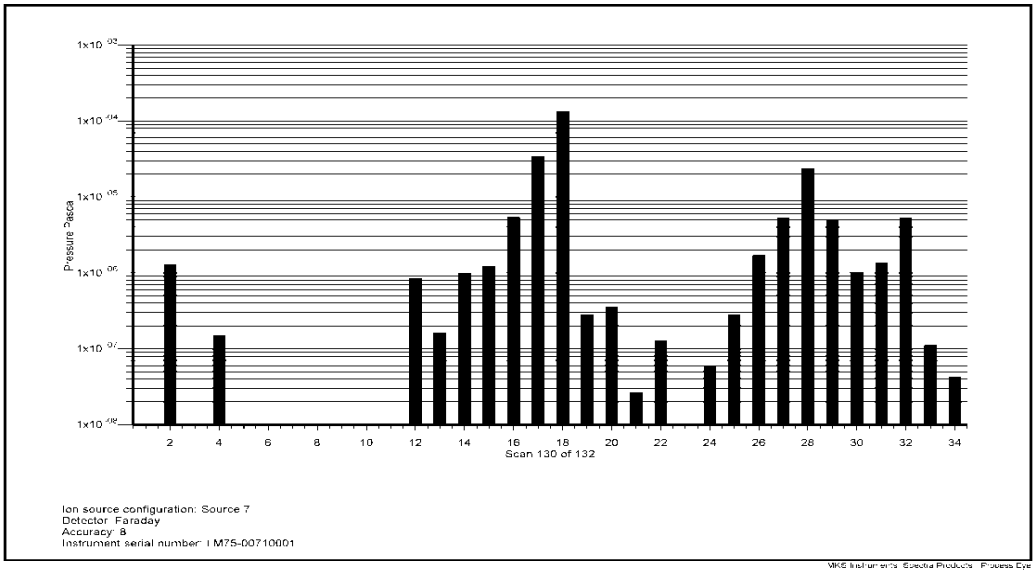

Figure 2. Mass spectrum of residual gas after pumping the ARM NTP chamber to a preparatory high vacuum.

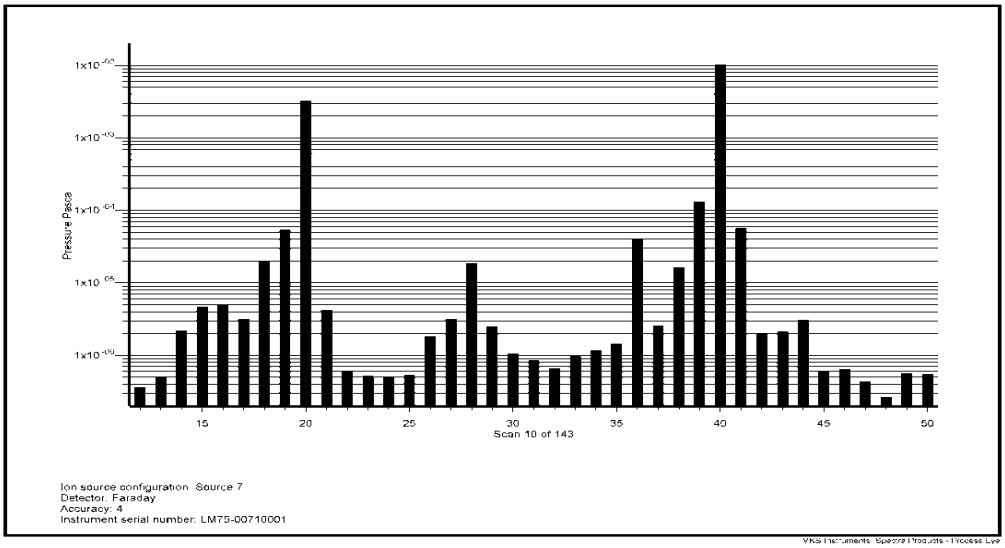

Figure 3. Mass spectrum of plasma-supporting gas in the ARM NTP chamber during the intake of argon.

The mass spectra diagrams show that the primary components of residual gas are water (peaks at 16, 17, and 18) – partial pressure at 1.2×10^{-4} Pa, nitrogen (peaks at 28 and 14) – partial pressure at 2.2×10^{-5} Pa, oxygen (peaks at 32 and 16) – partial pressure at 4.5×10^{-6} Pa, light hydrocarbons (peaks at 26, 27, 29, 30, and 31) – with a total partial pressure at 1×10^{-5} Pa. Other substances include hydrogen, helium, carbon, and hydrocarbon particles with a total partial pressure at 1×10^{-6} Pa

After the intake of argon into the chamber (masses 40 and 20) to a pressure of 1×10^{-2} Pa, the pressure of residual gas components (including impurities in the argon) became as follows: for water (peaks at 16, 17, and 18) – partial pressure at 2×10^{-5} Pa, for nitrogen (peaks at 28 and 14) – partial pressure at 2×10^{-5} Pa, for oxygen (peaks at 32 and 16) – partial pressure at 6×10^{-7} Pa, for compound of carbon (peaks at 15, 19, 21, 26, 27, 29, 35, 36, 37, 38, 39, 41–44) – total partial pressure at 2.7×10^{-4} Pa. The pressure of argon isotopes with mass numbers 36 and 38 (Ar^{36} and Ar^{38}) have been excluded from this figure. Components, having a partial pressure of 1×10^{-6} Pa and less than this figure have also not been considered. Thus, the result shows that the total percentage of impurities in the plasma-supporting gas is 2.9 %, including 0.2 % water and oxygen. Further vacuum pumping in the chamber to an operating pressure of 0.1 Pa revealed that the impurities proportionally decreased to 0.3 %, including 0.02 % water and oxygen.

In comparison we can see that the high quality GOST 10157-79 argon, used in the test has the following percentage of impurities: under 0.0007 % oxygen, under 0.005 % nitrogen, under 0.0009 % water, under 0.0005 % hydrocarbon compounds. The total percentage of argon is over 99.99 % [3]. Thus, the percentage of water and total sum of impurities in the plasma-supporting gas is significantly greater than required by the state standard for argon intake.

The analysis of the mass spectra show the following: after cleaning the internal surfaces of the chamber and high vacuum pumping, the residual gas in the chamber and plasma-supporting gas continue to have relatively high percentages of chemically-active gases, e.g.:

water (masses 16, 17, 18), oxygen, nitrogen, hydrocarbons (masses 32, 16, 28, 14, 12, 12, etc.). The presence of hydrocarbon gases indicate residue from solvents sorbed in rubber washers, joints of internal equipment, and other components inside the chamber.

In order to decrease the level of impurities in the plasma-supporting atmosphere inside the chamber and improve the composition and properties of the coatings, it is necessary to take additional measures to clean and degas the surface of the chamber from condensation products and hydrocarbon compounds.

During the process of magnetron deposition the percentage of chamber residual gas components in the plasma-supporting gas can significantly increase the percentage of impurities in high quality GOST 10157-79 argon.

Thus, to provide a minimal degree of impurities in the coated surfaces it is vital to clean and degas the surfaces of the chamber, removing residual moisture and hydrocarbon compounds. Another recommendation is to conduct a mass-spectrometric analysis of residual and plasma-supporting gases before beginning the vacuum evaporation treatment of surfaces.

References

[1] Holland L 1962 Nanesenie tonkikh plenok v vakuume [Eng. trans. Thin-layer Coating in Vacuum] (Moscow: Gosenergoizdat)
[2] Platnik L S, Sorokin V K 1977 Materialovedenie v mikroelektronike [Eng. tans. Material Sciences in Microelectronics] (Moscow: Energia)
[3] GOST [Russian State Standard] 10157-79 Gaseous and Liquid Argon. Technical Specifications