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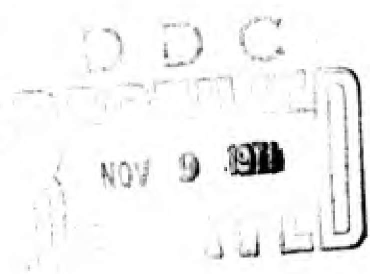
SOME QUESTIONS OF VACUUM TECHNOLOGY DURING THE DEPOSITION OF THIN FILMS

by

B. S. Danilin and V. Ye. Minaychev



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13. ABSTRACT

The structure and properties of thin films obtained by thermal vaporization in a vacuum are determined to a considerable degree by the conditions of condensation and depend on temperature of vaporization, rate of condensation, and angle of incidence of deposited substance on the base layer; nature of the base layer, its degree of purity, and microrelief of the surface; the degree of evacuation and the composition of residual gases during condensation. The parameters of the above categories are discussed briefly in this article. The conclusion is that since the change in the properties of films is influenced decisively in many cases not by the absolute magnitude of the vacuum, but by the magnitude of partial pressures of definite gases and vapors, their removal from the working volume by selective evacuation is a simpler and more real solution to the problem of obtaining pure films than the superhigh vacuum.

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Translator's note: On several occasions, symbols found in formulae and calculations appear to have been rendered incorrectly in the original document. They will be shown exactly as they appear in the original.

U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

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SOME QUESTIONS OF VACUUM TECHNOLOGY
DURING THE DEPOSITION OF THIN FILMS

B. S. Danilin and V. Ye. Minaychev

The structure and properties of thin films, obtained by thermal vaporization in a vacuum, in spite of the seeming simplicity of the process of obtaining them, are determined to a considerable degree by the conditions of condensation and depend on a number of factors, which can be united into the following three groups:

1) the temperature of vaporization, the rate of condensation and the angle of incidence of the deposited substance on the base layer, and also the degree of its purity and conformity of its structure to the structure of the base layer;

2) the nature of the base layer, the degree of its purity, the microrelief of the surface and the temperature of the base layer during the condensation of the volatile substance on it;

3) the degree of evacuation and the composition of residual gases during condensation.

The parameters of the first two groups have been studied well [1-5]. The question about the effect of the degree of evacuation

and the composition of residual gases on the properties of thin films has been studied the least. At the same time this factor determines to a considerable degree the stability and the reproducibility of the structure and physical parameters of films.

The interplay of the volatile substance with residual gases can occur both in the body and on the surface. In the body during transfer from the source to the base layer the molecules and atoms of the volatile substance can, in the first place, be dispersed on the molecules of residual gases, which in certain cases can slow down the growth of the layer and distort the distribution of the condensate, and, in the second place, the vapor can be saturated by gases and carry them into the condensate.

On the other hand, the freshly deposited surface of the layer, continuously undergoing bombardment by the molecules of residual gases, can absorb them, and sometimes form chemical compounds with them.

As it appears from Fig. 1, the processes which proceed in the body in the presence of a high vacuum do not exert a substantial effect on the degree of contamination of the deposited material with residual gases. For example, at a pressure of 10^{-6} mm Hg and d = 5 cm out of each 1000 molecules of volatile substance only one collides with a molecule of residual gas.

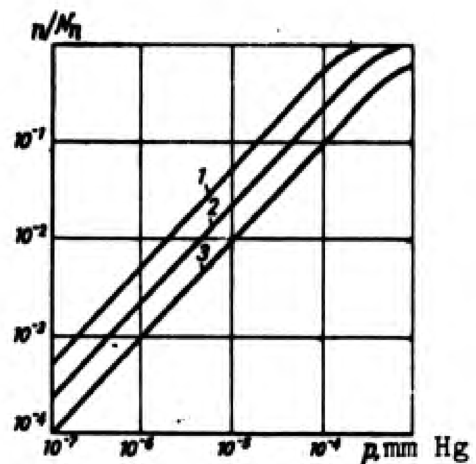

Fig. 1. The dependence of the ratio n/N_n on pressure in the case of various distances between the evaporator and the base layer: (n — number of collisions, N_n — number of molecules of volatile substance in 1 cm^3 of volume): 1 — d = 20; 2 — 10; 3 — 5 cm.

For an estimation of the possible degree of contamination of a layer by residual gases during its condensation it is sufficient to compare the number of molecules of the residual gases N, colliding on the surface of condensation, with the number of molecules of volatile substance N_0 which reach the base layer in a unit of time.

Figure 2 illustrates the possible degree of contamination of a layer of aluminum by residual gases at various pressures depending on the rate of condensation. Thus, at a rate of condensation 10 Å/s and pressure of 10^{-6} mm Hg the number of the molecules of gas hitting about the base layer and volatile material is found in a ratio of approximately 1:10. If the coefficient of adhesion of nitrogen was equal to a unit, then the layer of aluminum could contain up to 10% nitrogen, however, even a comparatively small quantity of molecules, which can contain up to 10% nitrogen, however, even a comparatively small quantity of molecules, which can be retained on the surface of a freshly deposited film, will substantially change its physical properties, inasmuch as many materials (even at room temperature) possesses a high chemisorption capacity. The classification of these materials are given in the table (in parenthesis those materials are shown, which possibly belong to the given group, but the sorption properties of which have not been properly studied).

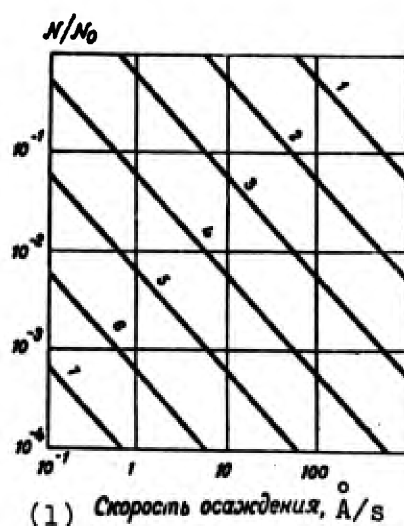

Fig. 2. The dependence of the ratio N/N_0 on the rate of deposition of aluminum at various pressures.

1 — $p = 10^{-4}$; 2 — 10^{-5}; 3 — 10^{-6}; 4 — 10^{-7}; 5 — 10^{-8}; 6 — 10^{-9}; 7 — 10^{-10}

KEY: (1) Rate of deposition, Å/s.

(1) Группа	(2) Материал	• Газ (3)						
		O_2	C_2H_2	C_2H_4	CO	H_2	CO_2	N_2
I	Ca, Sr, Ba, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Fe (Re)	+	+	+	+	+	+	+
II	Ni, Co	+	+	+	+	+	+	−
III	Rh, Pd, Pt, (Ir)	+	+	+	+	+	−	−
IV	Al, Mn, Cu, Au*	+	+	+	+	−	−	−
V	K	+	−	−	−	−	−	−
VI	Mg, Ag, Zn, Cd, In, Si, Ge, Sn, Pb, As, Sb, Bi	+	−	−	−	−	−	−
VII	Se, Te	−	−	−	−	−	−	−

*Gold does not absorb oxygen.

KEY: (1) Group; (2) Material; (3) Gas.

Apart from the gases shown in the table, the majority of materials also absorb intensely the water vapors and the hydroxyl group. At the same time inert gases in a neutral form are not absorbed in a noticeable quantity by freshly deposited films even of such an active material as titanium [7].

The probability of adhesion (gripping) of the molecules of gas during their collision on the surface of this or that material depends on the energy of the bond between them. The majority of materials selectively absorb gases. Therefore the composition of the gases which are absorbed by the deposited film substantially differs from the composition of residual gases in the vacuum system [8].

During the vaporization of the material and its condensation the pressure p in the working volume of the device is determined by the following relationship:

$$p = \frac{Q + (1 - a) G}{s + Ak\alpha}, \qquad (1)$$

where s — the effective rate of evacuation of the pump which is utilized; Q — the quantity of gas which enters the system as a result of gas evaluation from the walls and inleakage: G — the quantity of gas which enters the system as a result of gas evolution from the volatile material; A — the area of condensation; k — gas absorbing capacity (the rate of evacuation) of a unit of area of condensation α — the coefficient which characterizes the part of molecules interacting with the film and remaining in it in the form of gas inclusions.

If k and α have significant magnitude, but $G \ll Q$, then a reduction in pressure in the vacuum system is observed because of the gettering action of the freshly deposited film. This process is accompanied by the intense contamination of the film with residual gases and by change of its physical properties.

Since the films of a number of materials actively absorb gas, then the most contaminated prove to be the inner layers, which absorb the basic mass of residual gases. If as a result of the interaction of the freshly deposited film with residual gases oxides appear in the metal, thus "cementing" the zone between the metal and the glass, then the adhesion of the film to the glass base layer is considerably improved.

For some materials the partial oxidation of the films sometimes improves their properties (for example, partial oxidation of the Nichrome film during its formation reduces its temperature coefficient of resistance, thereby improving the parameters of thin-film resistors). The excessive reduction in the partial pressure of the oxidizing agent can be undesirable also in the case when the film of metallic oxides are prepared which are reduced at high temperature as well as in the case of low pressure of oxygen.

One of the most sensitive methods for the determination of the influence of residual gases on fine films is the measurement of

the specific electrical resistance, which usually increases in proportion to the contamination of the film by residual gases, moreover this influence is exhibited most sharply in the case of small thicknesses of films [9].

An increase in the rate of condensation with the same magnitude of vacuum decreases the probability of contamination of the film by residual gases. In this case, its specific electrical resistance ρ is decreased, which with an increase in the rate of condensation strives for a maximum value ρ_0, depending on the thickness of the film.

The index of the degree of contamination of the film by extraneous gas inclusions is the value

$$\beta = \frac{\rho - \rho_0}{\rho}. \quad (2)$$

By assigning the maximally possible magnitude of β it is possible from the dependence of specific electrical resistance on the reverse value of the rate of condensation to find the value ρ_0 and the minimum permissable rate of condensation [10].

The increase in specific electrical resistance is accompanied by a change in orientation and structure. It is characteristic that on the change in electrical properties and structure in films of a number of metals and alloys the most noticeable effect among the ordinary residual gases is exerted only by oxygen and water vapor. Thus, at low partial pressures of oxygen polycrystalline, strongly oriented films of indium and tin are obtained, but at significant partial pressures of oxygen the films are not oriented at all; their surface has a considerably coarser structure. Films of antimony, condensed in a conventional vacuum device with a rate of condensation less than 100 Å/s, are heterogeneous and consist of amorphous islets submerged into a matrix made up of crystallites. At high rates of

deposition purely metallic films are obtained. Furthermore, uniform metallic films can be obtained even at very low rates of condensation (0.1-1 Å/s) in a ultrahigh vacuum device.

The adsorption of residual gases by semiconductors, on the one hand, can cause irreversible changes in the surface of the semiconductor, and on the other – change the distribution of the carriers of the charge in the solid body. Oxygen exerts a great influence on the surface properties of germanium [11]. In adsorbing on the surface of germanium, it dissociates [12] and, depending on the pressure of the gas and the properties of the material, can manifest its influence either as an acceptor [13] or a donor [14].

Residual gases exert a substantial influence on the change in the Hall coefficient. This change is expressed particularly sharply during the initiation and growth of the film, where the influence of the residual gases is most intense [15]. The interaction of residual gases (in the first place of oxygen, water vapor, and of carbon dioxide) is exhibited especially strongly on the characteristics of fine films which are utilized as superconductors. For example, for obtaining ultrapure films of tin and indium with sharp boundaries of transition from a normal state into the state of superconductivity it is necessary that at a rate of condensation of 50 Å/s the partial pressure of oxygen does not exceed $5 \cdot 10^{-8}$ mm Hg [8]. With an increase in the oxygen content of films of tin there is an increase in the critical field, necessary for disruption of the superconducting state and a smoother transfer of films from one state into another is observed. No less sensitive to the influence of a number of gases are superconducting layers made from indium, niobium, vanadium, tantalum, the carbide of tantalum and nitride of niobium [16, 17], and also films made from ferromagnetic materials [18, 19].

The sources of a number of gases and vapors, actively affecting the properties of films, apart from being volatile materials are also the structural elements of vacuum systems and the pumps utilized in them.

In the technology of deposition of thin films the greatest distribution is received by diffusion oil-vapor pumps. Their advantages lie in the comparatively low cost, simplicity of construction, reliability and convenience in operation, the constancy of the speed of action in a sufficiently wide interval of pressures, and the capacity to rapidly reduce to working volume the required vacuum in the case of sharply changed gas loads. These pumps operate basically on mineral oils VM-1, VM-2, and VM-5. The pressure of the vapors of these oils is comparatively high, which makes them the basic sources of hydrocarbons, which contaminate the residual atmosphere of deposition devices.

Two ways exist for reducing the migration of vapors of working liquid from the pump into the volume being evacuated: the introduction of the highly effective means of protection (traps); the utilization of thermoresistant working liquids with a low vapor pressure.

The designs of cooled and adsorption traps should be such that the rectilinearly moving molecules of vapors of working liquid have to collide repeatedly with the active (cooled) surface of the traps and we could pass into the volume being evacuated. This is attained by means of application of multistage traps with the simultaneous utilization of antimigrators, which prevent the penetration of oil into the volume being evacuated over the inner walls of the trap housings. However, it is necessary to keep in mind that protection with the help of such traps sharply lowers the effectiveness of the pumps, the speed of action of which is used altogether only by 20-30%. This makes it compulsory to use for deposition devices large-scale pumps with a nominal speed of operation of no less than 2000 l/s [20], in order to obtain an effective rate of evacuation of the working volume of the device of an order of 400-600 l/s, which is necessary for the compensation of abundant generation of gas during the atomization of this or that material.

The quantity of hydrocarbons can also substantially lower the series incorporation of two oil-vapor pumps. The second booster pump lowers the rate of antidiffusion of gases and vapors from the side of the line of preliminary evacuation. Furthermore, the low exhaust pressure, created by the booster pump, ensures favorable conditions for the purification of the working liquid of the basic pump from the products of cracking.

Today instead of vaseline oil all the wider use is being made of silicoorganic liquids. Because of high thermal stability and low elasticity of vapor at room temperature ($3 \cdot 10^{-10}$ mm Hg) these liquids make it possible to obtain in the pump a maximum vacuum of 10^{-9} mm Hg without the use of low-temperature traps and 10^{-10} mm Hg with traps cooled from -20° to -30°C. Approximately the same vacuum can be obtained on polyphenyl ethers. The sum of the partial pressures of heavy hydrocarbons in these cases does not exceed 5×10^{-10} mm Hg [21]. If necessary for a further reduction in the level of heavy hydrocarbons when using these working liquids it is recommended to also use the series connection of pumps and freezing traps.

Furthermore it follows to keep in mind that the mechanical pump is also a substantial source of hydrocarbons. Especially intense contaminations occur during the preliminary evacuation of the working volume through a forevacuum main line connected directly to it. Therefore on the forevacuum main line between the working volume and mechanical pump it is also compulsory to install an effective trap.

Another component of residual atmosphere which actively influences the films are the water vapors which are intensely absorbed by the inner surface of the working body of the device during admission into it of humid atmospheric air, and then are continuously liberated from the walls during evacuation. It should be noted that in unheated deposition devices the water vapors are

the basic component and their content reaches 90% of the overall composition of residual gases.

For the purpose of reducing the level of water vapors in the working volume it is necessary to have at least a brief warming up of its internal surfaces at temperature above 100°C. Thus, for this goal a KI-220-1000 infrared heating lamp was used. It was placed inside the working chamber. After a hour of warming up of a chamber with a volume of 130 l an evacuation of $7 \cdot 10^{-7}$ mm Hg was obtained in it (Fig. 3).

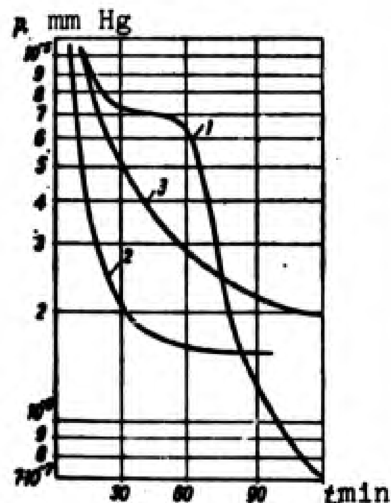

Fig. 3. The evacuation curves of the industrial deposition device UVM-2M: 1 – after a hour of warming up the walls at a temperature above 100°C; 2 – after raising the hood in an external helmet with dried air; 3 – without warming up the hood after the admitting atmospheric air into it.

A reduction in the level of water vapors can also be achieved with the utilization of external safety helmets with dried air. In this instance upon lifting the hood inside the safety helmet the internal surfaces of the working volume do not make contact with the humid atmosphere, as a result of which the time for pumping out of the device to the required evacuation is decreased by several times (Fig. 3, curves 2, 3).

The results of the experiments, presented in Fig. 3, were

obtained on a standard series device of the design UVM-2M [22]. In this case liquid nitrogen is not poured into the trap.

A reduction in the level of hydrocarbons, water vapors, and carbon dioxide is also facilitated by the introduction directly into the working volume of the device of surfaces, which are found at the temperature of liquid nitrogen (so-called Meissner traps). However, this is combined with a number of inconveniences, induced by the necessity for the periodic warming of the traps before the next lifting of the hood. One of the effective means for reducing the partial pressures water vapors and chemically active gases is their supplementary evacuation with the help of a titanium evaporation pump, installed in the working volume in direct proximity to the source of generation of gas [23] (Fig. 4). From Fig. 4 it is evident that with "combined" pumping out the partial pressure of such "harmful" components as heavy hydrocarbons, water vapors, and carbon monoxide is considerably lowered, and in essence the basic component of residual gases is hydrogen.

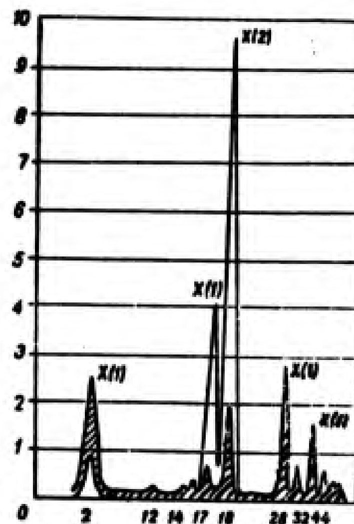

Fig. 4. The mass spectrogram of residual gases during the evacuation of the working volume of a device of an oil-vapor diffusion pump (clear sections) and the joint evacuation by this and titanium evaporation pumps (shaded sections).

The inconveniences, which appear during the high-temperature warming up of vacuum deposition devices, stimulated the searches

for new ways of purifying the internal surfaces. These include, in particular, "washing" (scavenging) of vacuum devices by light gases (hydrogen, helium, and others). Such a "washing" manages sufficiently rapidly to move away from the walls and weakly connected molecules of gases and contaminations while the molecules with powerful sorption bonds which remain on the walls no longer are a serious obstruction to obtaining a high vacuum. The effectiveness of gas flushing increases when it is combined with the warming up of the walls of the object being washed, and also when using atomic or ionized gas. In this case the washing gas becomes the basic component of residual atmosphere [24].

For reducing the gas inclusions in films of those materials which possess an increased sorption capacity it is expedient prior to the beginning of their condensation on the base layer to produce a preliminary deposition of a film of this material on the cooled surface which is located in the working volume. The film, which is deposited on the cooled surface and is absorbing chemically active gases, simultaneously lowers their content in the residual atmosphere, as a result of which the gas inclusions in the films which are deposited on the base layer are decreased.

Conclusions

Since the investigations conducted by various authors speak for the fact that the change in the properties of films is influenced decisively in many instances not by the absolute magnitude of the vacuum (at $p < 10^{-6}$ mm Hg), but by the magnitude of partial pressures of definite gases and vapors, their removal from the working volume by selective evacuation is a simpler and more real solution to the problem of obtaining pure films than the superhigh vacuum.

The lowering in the level of these gases and vapors can be achieved by the protection of the working volume from the penetration

of the vapors of working liquids from the high-vacuum pump and the pump for preliminary evacuation; by the replacement of vaseline oils by silicoorganic working liquids and polyphenyl ethers, which have a low pressure of vapors; by the warming up of the internal walls of the device (for example, with the aid of infrared heating lamps); by the utilization of external safety helmets with dried air, thus preventing the contact of the internal surfaces of the working volume with the humid atmosphere; by the introduction into the working volume of supplementary exhaust means (titanium evaporation pumps, Meissner traps, and others); by the preliminary "getter" atomization of the vaporized material, which possesses an increased sorption capacity.

The enumerated means for reducing the level of harmful gases and vapors can be carried out on deposition devices of industrial design with the help of comparatively simple supplementary devices.

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