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MATHEMATICAL MODEL OF DETERMINING HEAT CONDITION IN DESUBLIMATION PROCESS OF VOLATILE METAL FLUORIDES

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To optimize the technological parameters of the process and to create the automatic regulation system of desublimation process the mathematical model for desublimation process of volatile metal fluorides in surface devices (by the example of uranium hexafluoride) has been developed. Optimal conditions for desublimation process designed with the use of the model developed were tested and showed good agreement with the experimental data.

Introduction

Not only stability and efficiency of desublimation equipment operation but also manufacturing environmental safety often depend on proper organization of heat conditions in desublimation process of volatile substances in surface desublimators. So obtaining uranium hexafluoride (UHF) by uranium oxides fluorination, UHF at desublimation may be accumulated on certain areas of device surface where the most efficient conditions for the given process are formed [1]. It often results in device packing or breakthrough of residual phase through the device therefore the maximal degree of filling the industrial desublimators with desired product (G_{max}) not often exceeds 80 % and for the devices of complex geometry, for example, ring desublimators this value is only 30...50 %.

As the layer of solid product is formed a free cross section of desublimation device decreases and gas velocity correspondingly increases, temperature of desublimation surface changes, as well as the conditions of heat transfer from desublimated product to the device surface [2]. This results in changing desublimation surface temperature that may cause gaseous UHF breakthrough through the device therefore gases containing UHF, oxygen and fluorine excess after the fluoridation stage pass two or more serially connected desublimator. In each next device the desublimation surface increases in the same direction as gas is moving.

Therefore determination of optimal conditions of UHF desublimation from gas-vapor mixture is urgent. It is necessary for controlling desublimation front in the device and increasing the degree of desublimator filling-up with solid product due to its uniform distribution over the volume of the device excluding product losses connected with inefficient operation of desublimation surface, prevention of sudden packing of the device with solid UHF.

These tasks may be solved using the developed mathematical model of desublimation process describing mass, heat and hydrodynamic flows inside the device. This mdel allows determining:

- mass flows of gas-vapor mixtures and solid UHF inside the device (material flows);
- coefficients of heat- and mass transfer from gas-vapor mixture to solid surface;
- cooling time of gas-vapor mixture from initial temperature to the temperature of UHF desublimation;
- velocity and mass of UHF segregated from gas flow into solid phase per unit surface;
- change of heat- and hydrodynamic flows inside the device occurring due to increasing thickness of product layer on its walls in desublimation process.

Computation of the device having annular shape or the shape of flat planes is rather complex therefore let us consider mathematical model for determining optimal heat conditions of desublimation for annular devices.

1. Material computation of UHF desublimation process

Material computation was carried out on the basis of the scheme of annular desublimator flows entering and escaping it subject to uranium hexafluoride desublimation (Fig. 1). The obtained data will be further used at thermal design of cooling process of gas-vapor mixture containing UHF.

Gas-vapor mixture escaping the reactor of uranium oxides fluoridation has a complex composition. Together with desired product of the reaction (UHF) there is an excess of F_2 and HF forming as a result of interaction of fluorine with moisture inevitably attending in gaseous phase (before entering into fluoridation reactor initial fluorine is purified from HF). Besides, mixtures attending in uranium oxides as well as vapors of OF_2 and NOF enter into gaseous mixture. Due to low content of the latter in gas-vapor mixture HF, OF_2 and NOF concentrations may be neglected.

At specified mass efficiency of desublimator by the main product and reaction gases composition **before desublimation** the following things are determined subsequently:

1) volume flow of UHF

$$V_{UF_6} = \frac{M^{мас}_{UF_6}}{\rho_{UF_6}},$$

where $M^{мас}_{UF_6}$ is the volume flow, kg/h; ρ_{UF_6} is the density of vapors at the temperature of entering gas-vapor mixture, kg/m^3; V_{UF_6} is the volume flow, m^3/h;

2) volume flow of each component in gas mixture

$$V_i = \frac{V_{UF_6} M_i}{M_{UF_6}},$$

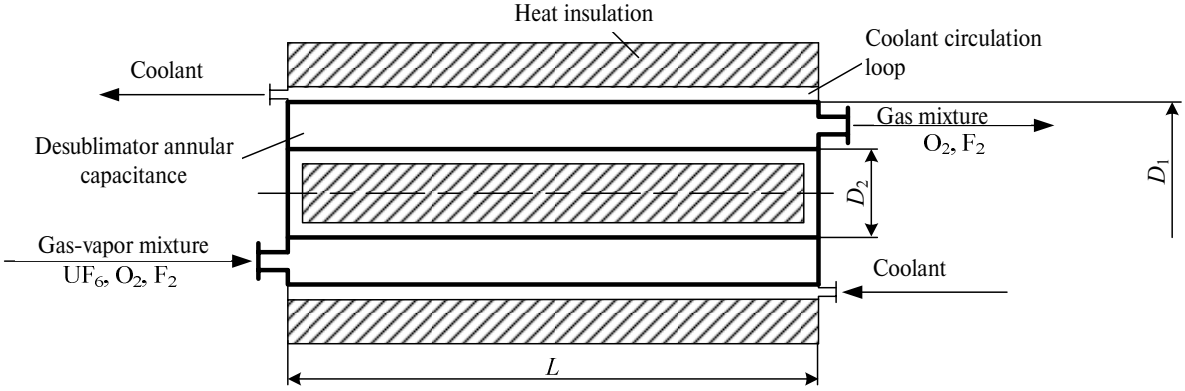

Fig. 1. *Scheme of flows entering into desublimator and escaping from it. L is the length of desublimator annular capacitance; D_1, D_2 are the diameters of inner and outer ring of the device*

where V_i is the volume flow of i mixture component in composing, m³/h;

3) mass flow of each component in gas mixture

$$M_i^{мас} = V_i \cdot \rho_i,$$

where ρ_i is the density of i mixture component at specified temperature T and pressure P, kg/m³ [3] determined as

$$\rho_i = \frac{M_i}{22,4} \cdot \frac{273 \cdot P}{T_г^{вх} \cdot P_0},$$

where M_i is the molar mass of i gas component, kg/mole; P is the partial pressure of components in the system, Pa; P_0 is the total pressure of gas mixture, Pa; $T_г^{вх}$ is the temperature of i gas component, K.

Mass flows of the gas mixtures components **after desublimator** are determined from the equation of material balance

$$\sum M_{прих} = \sum M_{расх},$$

where $\Sigma M_{прих}$ and $\Sigma M_{расх}$ are the mass flows of substances before and after desublimation, kg/h, equal to

$$\sum M_{прих} = M_{UF_6}^{мас} + M_{F_2}^{мас} + M_{O_2}^{мас};$$

$$\sum M_{расх} = (M_{F_2}^{мас}) + (M_{O_2}^{мас}) + M_{UF_6\,(тв)}.$$

Total mass flow of gas mixture after desublimator equals to

$$\sum M_г^{мас} = (M_{F_2}^{мас}) + (M_{O_2}^{мас}) = \sum M_{прих} - M_{UF_6}.$$

Taking into consideration the complicity and ambiguity of explanation of UHF desublimation process and admixtures behavior at it let us accept he following assumptions:

- UHF is desublimated for 100 %;
- Mixtures contained in UHF (F_2 and O_2) do not undergo chemical transformations and stay in gaseous phase.

2. Heat and hydrodynamic computations

Calculating UHF cooling and desublimation the following things are determined subsequently for the flows, showed in Fig. 1:

1) integral physical-chemical characteristics of gas-vapor mixture at input and output of desublimator;
2) heat transfer for axially symmetric problem.

Density of gas-vapor mixture $\rho_{см}$ may be found from the equation

$$\rho_{см} = x_{UF_6}\rho_{UF_6} + x_{O_2}\rho_{O_2} + x_{F_2}\rho_{F_2},$$

where x_i is the mass fractions of mixture component, equal

$$x_{UF_6} = \frac{M_{UF_6}}{M_{см}}; \quad x_{O_2} = \frac{M_{O_2}}{M_{см}}; \quad x_{F_2} = \frac{M_{F_2}}{M_{см}}. \qquad (1)$$

Coefficient of dynamic viscosity $\mu_{см}$, specific mass heat capacity at constant pressure $c_{p\,см}$ and coefficient of heat conductivity $\lambda_{см}$ of gas-vapor mixture are calculated by the similar equations

$$\mu_{см} = x_{UF_6}\mu_{UF_6} + x_{O_2}\mu_{O_2} + x_{F_2}\mu_{F_2}; \qquad (2)$$

$$c_{p\,см} = x_{UF_6}c_{p\,UF_6} + x_{O_2}c_{p\,O_2} + x_{F_2}c_{p\,F_2}; \qquad (3)$$

$$\lambda_{см} = x_{UF_6}\lambda_{UF_6} + x_{O_2}\lambda_{O_2} + x_{F_2}\lambda_{F_2}. \qquad (4)$$

Average rate of moving gas-vapor mixture along the section of the device W, m/s is determined by the formula

$$W = \frac{Q}{S},$$

where S is the cross-sectional area in the device, m².

The main criteria of heat and hydrodynamic similarity (Reynolds Re, Prandtl Pr and Peklet Pe) equal [4, 5]:

$$\mathrm{Re} = \frac{W\rho_{см}d_э}{\mu_{см}}; \quad \mathrm{Pr} = \frac{c_{p\,см}\mu_{см}}{\lambda_{см}}; \quad \mathrm{Pe} = \mathrm{RePr}.$$

where $d_э$ is the equivalent diameter of the device cross-section, m.

Physical-chemical properties of gas-vapor mixture at the device output are calculated similarly by the formulas (1)–(4).

Thermal-transport equation for the case of axial-symmetric laminar flow has the form [6, 7]:

$$\frac{\partial T}{\partial \tau} + W\frac{\partial T}{\partial x} = a\left(\frac{\partial^2 T}{\partial x^2} + \frac{1}{r}\frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial r^2}\right).$$

Let us write down approximately:

$$W = \text{const}, \quad \frac{\partial T}{\partial x} = \frac{T - T_0}{x}, \quad \frac{\partial^2 T}{\partial x^2} = \frac{T - T_0}{x^2}$$

and consider the ratio:

$$\left(W\frac{\partial T}{\partial x}\right)\Big/\left(a\frac{\partial^2 T}{\partial x^2}\right)=\frac{Wx}{a}=\frac{Wd_э}{\nu}\frac{\nu}{a}\frac{x}{d_э}=$$

$$=\mathrm{RePr}\frac{x}{d_э}=\mathrm{Pe}\frac{x}{d_э}>>1,$$

where $W\frac{\partial T}{\partial x}$ is the convective heat transport; $a\frac{\partial^2 T}{\partial x^2}$ is the diffusion heat transfer; $a=\frac{\lambda_{см}}{c_{см}\rho_{см}}$ is the coefficient of gas mixture temperature conductivity, m²/s.

The carried out estimation shows that the ratio of the convective heat transport to the heat transfer by temperature conductivity >>1 in the whole rated area. Therefore, the heat flow transfer along the longitudinal axis (x) by heat conductivity may be neglected in comparison with the transfer by convection.

Ultimately the equation of boundary layer turned out approximate. Taking into account the ratio between the values of the coefficients of heat conductivity (λ) and heat transfer (α) as well as geometry of the flow area of gas-vapor mixture in the annular desublimator the assumption about the fact that gas flow is cooled to the constant temperature equal to the temperature of UHF desublimation and the whole heat flow extracted in desublimation process is discharged through the lateral surfaces may be made (Fig. 2). The following notations are accepted: T_0, T_1, T_s, T_w are the temperatures of gas-vapor mixture at the input to the desublimator refrigerated zone, desublimation front, surfaces of desublimation and desublimator cold wall; q_1, q_2 are the heat flows discharged from gas flow to desublimation surface and through desublimate layer to the device wall.

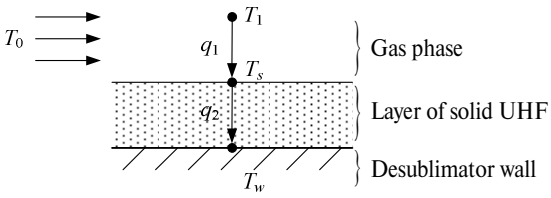

Fig.2. *Scheme of cooling process and UHF desublimation from gas-vapor mixture*

To estimate the length of initial nonsteady area of gas-vapor mixture flow l_i the following formula may be used [6–8]:

$$\frac{l_i}{d_э}=0,055\,\mathrm{Re}.$$

Heat balance in the system is described by the equation

$$dQ=dm\Delta H=\alpha dTdFd\tau,$$

where ΔH is the heat of UHF desublimation, kJ/mole; dF is the area of a unit cell of desublimation (surface), m², equal

$$dF=\frac{\pi}{4}(D_1^2-D_2^2)dx,$$

where D_1, D_2 are the diameters of outer and inner ring in cylindrical device, Fig. 1, m; dx is the length of the unit cell, m.

Mass loss of UHF at desublimation may be determined by the formula

$$dm=dV\rho_{тв}=dFd\delta\rho_{тв},$$

where $d\delta$ is the change of UHF layer thickness on desublimator cooling surface for the time $d\tau$, m; dV is the volume of extracted UHF into solid phase, m³, equal

$$dV=dFd\delta.$$

Hence:

$$\Delta Hd\delta\rho_{тв}dF=\alpha dTdFd\tau,$$

$$\frac{dm}{d\tau}=\frac{\alpha dTdF}{\Delta H}=\dot{m},$$

$$\frac{\dot{m}}{\rho_{тв}}=\dot{V}=dF\beta=\frac{\alpha dTdF}{\Delta H\rho_{тв}}.$$

where $\dot{m}$ is the rate of mass release, kg/s; $\dot{V}$ is the rate of UHF volume release, m³/s.

Then

$$\beta=\frac{d\delta}{d\tau}=\frac{\alpha dT}{dH\rho_{тв}},\qquad(5)$$

where β is the coefficient of mass transfer from gas to cool surface, m/s [4–6].

Having integrated the equation (5) by the temperature the equation for mass transfer from gas to desublimation surface may be obtained:

$$\beta=\frac{\alpha(T_1-T_s)}{\Delta H\rho_{тв}}.$$

Heat flow transferred by desublimation surface gas may be expressed from Newton law for heat transfer:

$$q_1=\alpha(T_s-T_1),$$

and heat flow transferred through the layer of solid UHF to the device wall may be stated by Fourier law for heat-transfer:

$$q_2=-\lambda_{тв}\frac{T_s-T_w}{\delta}=-\frac{\lambda_{тв}}{\delta}(T_s-T_w).$$

On the basis of the above we accept $q_1=q_2$, then

$$\alpha(T_s-T_1)=-\frac{\lambda_{тв}}{\delta}(T_s-T_w).\qquad(6)$$

Having sated the temperature of desublimation surface from the equation (6) we have:

$$T_s=\left(\alpha+\frac{\lambda_{тв}}{\delta}\right)=\alpha T_1+\frac{\lambda_{тв}}{\delta}T_w.$$

Then

$$T_s=\frac{\alpha T_1+\frac{\lambda_{тв}}{\delta}T_w}{\alpha+\frac{\lambda_{тв}}{\delta}}=\frac{T_w}{1+\frac{\alpha\delta}{\lambda_{тв}}}+\frac{\frac{\alpha\delta}{\lambda_{тв}}T_1}{1+\frac{\alpha\delta}{\lambda_{тв}}}.\qquad(7)$$

As Nusselt number equals [4–6]

$$\mathrm{Nu}_{тв}=\frac{\alpha\delta}{\lambda_{тв}},\qquad(8)$$

then substituting equation (8) into expression (7) we may obtain:

$$T_s = \frac{T_w}{1+\mathrm{Nu}_{тв}} + \frac{\mathrm{Nu}_{тв}\,T_1}{1+\mathrm{Nu}_{тв}}.$$

Hydrodynamic regime in the effective volume of the device was calculated on the basis of the system of Navies-Stocks complete steady-state axial-symmetric averaged differential equations of elliptic type [6, 7, 9] written in variables of vorticity Ω, stream function Ψ. Equations (9), (10) were written in cylindrical coordinate system, where r is the radial and z is the axial:

$$u_r \frac{\partial \Psi}{\partial r} + u_z \frac{\partial \Psi}{\partial z} = \\ = \frac{1}{\mathrm{Re}} \frac{1}{r}\left[\frac{\partial}{\partial r}\left(r\nu_t \frac{\partial \Psi}{\partial r}\right) + \frac{\partial}{\partial z}\left(r\nu_t \frac{\partial \Psi}{\partial z}\right)\right] + F_j, \qquad (9)$$

$$\frac{\partial^2 \Psi}{\partial r^2} + \frac{\partial^2 \Psi}{\partial z^2} = \Omega r^2 + \frac{1}{r}\frac{\partial \Psi}{\partial r}, \qquad (10)$$

where $\Omega = \frac{1}{r}\left[\frac{\partial u_r}{\partial z} - \frac{\partial u_z}{\partial r}\right], u_r = \frac{1}{r}\frac{\partial \Psi}{\partial z}, u_z = -\frac{1}{r}\frac{\partial \Psi}{\partial r}.$

Left part of equation (9) describes convective transport of a substance (gaseous UHF) due to the medium speed (gas-vapor mixture). The first member of the right part of equation (9) describes diffusion substance transfer due to medium viscosity ν_t by the coordinates r and z. Member F_j describes additive source constituent stipulated by the change of volume medium flow by the section as a result of UHF mass loss from gas-vapor mixture in the process of desublimation. Poisson equation (10) for determining stream function Ψ is obtained from the equation of continuity [6, 7].

Using the above mentioned equations the program of calculation of cooling process and uranium hexafluoride desublimation process from gas-vapor mixture is developed.

3. Numerical implementation of mathematical model

Numerical implementation of mathematical model is carried out in the following order:

1) specified with initial data: initial geometry of computed area (D_1, D_2, L); physical properties and consumable parameters of each component in initial gas-vapor mixture $(\rho,\mu,\lambda,c_p,dm)_{UF_6,O_2,F_2}$, crystal UHF ($\rho_{тв}$, $\lambda_{тв}$, ΔH); rate and temperature boundary conditions (Q_0, T_0, T_w), technological process time ($\tau_{кон}$);
2) global calculation cycle is organized by the time with a specified stage δ and values of mass flow corresponding to this interval are searched out dm_{UF_6} [10];
3) inner calculation cycle is organized by the condition ($x \le L$ and $dm_{UF_6} > 0$), where x is the current coordinate by calculated area changing in the range of $0 \le x \le L$; L is the length of desublimator cooled part [10].

Physical-chemical characteristics of gas mixture, similarity criteria for the flow process of mixture Re, Pr, Nu are computed and heat-transfer coefficient α is determined inside this cycle.

Taking into account the fact that at the initial cooling area of gas-vapor mixture the process occurs in unstable conditions the calculation of desublimated UHF mass is found out subject to the ratio:

$$K = \frac{T_0 - T_i}{T_0 - T^*},$$

where T_i is the current temperature, K; T^* is the temperature of desublimation, K;

K deduction is taken into consideration to the moment while $T_i > T^*$ in the sequel it is supposed that K=1 and gas mixture temperature remains steady and equal to T^* while $dm_{UF_6} > 0$.

On the basis of the obtained parameters the mass-transfer coefficient β is calculated subject to K. According to the values W and $d\tau$ the released UHF material is distributed on the lateral surface of calculated area and dm_{UF_6}, is decreased by this value and x is increased.

Inner loop of the program continues computing subject to UHF layer growth and change of temperature on the surface of this layer T_s (Fig. 2).

4) after finishing outer loop the function graphs $\delta = f(x)$ are plotted.

Temperature curves of gas-vapor mixture depending on transverse (y) and longitudinal (x) coordinates in annular desunlimator space in initial nonsteady cooling area for the case of feeding base mixture with mass rate of $M_{UF_6}^{мас}$ to desublimator are presented in Fig. 3, 4 as an example. It is seen from the curves that the length of nonsteady cooling zone of gas-vapor mixture to UHF desublimation temperature along the device axis does not exceed 0,1L.

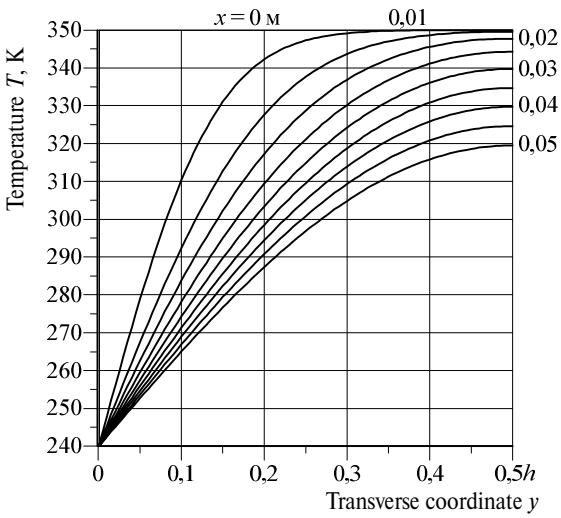

Fig. 3. *Dependence of gas-vapor mixture temperature on transverse coordinate at different values of x*

Due to low gas velocity (w=0,06 m/s) and laminar-flow conditions of gas-vapor mixture in desublimator ring section (Re=20) gas mixing does not occur in transverse direction (along axis y). Therefore, in transverse direction heat transfer in gas flow occurs only due to heat conductivity.

The length of the device zone in which all UHF is desublimated at temperature of desublimator wall of 243 K and desublimator UHF load to 0,5G_{max} amounted

to 0,5L without nonsteady cooling area of gas-vapor mixture (Fig. 5) and 0,8L with it (Fig. 6) and the total length of the device desublimation part is L. In this case the thickness of UHF layer on both walls of annular cavity amounts to 0,5h and 0,4h correspondingly.

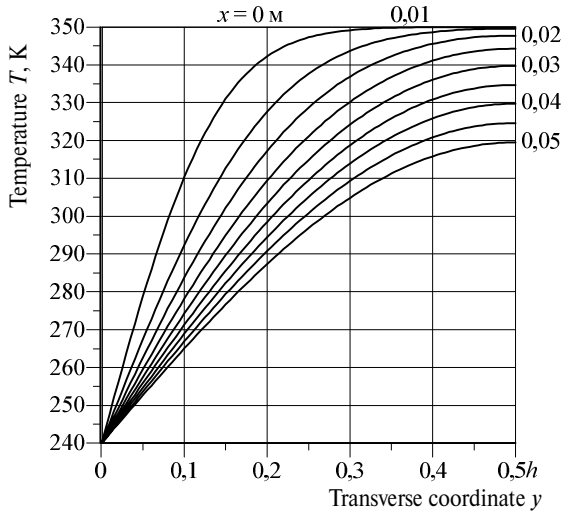

Fig. 3. *Dependence of gas-vapor mixture temperature on transverse coordinate at different values of x*

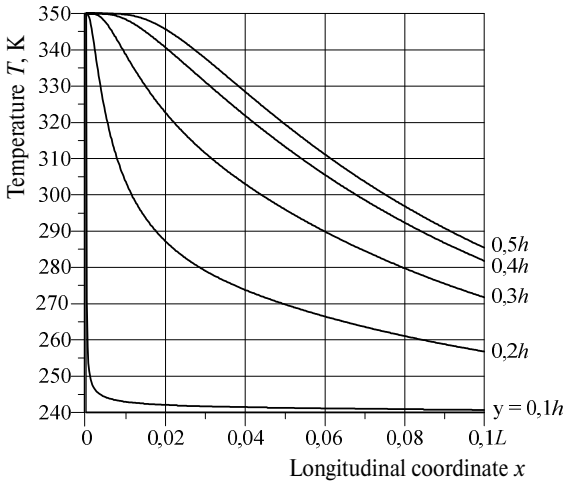

Fig. 4. *Dependence of gas-vapor mixture temperature on longitudinal coordinate at different values of y*

The curves of desublimator zones in which all UHF is desublimated at temperature of the device wall of 243, 253, 263 and 273 K with the area of nonsteady cooling of gas-vapor mixture are presented in Fig. 6.

It is seen from Fig. 6 that the length of the device area in which all UHF is desublimated at desublimator UHF load to 0,5G_{max} with the area of nonsteady cooling at temperatures of desublimator walls of 253, 263 and 273 K amounts to 0,83L, 0,88L and 0,98L correspondingly at total length of the device desublimation part L. At such desublimation temperatures the thickness of UHF layer on both walls of annular cavity equals to 0,77h, 0,70h and 0,65h, 0,77h, 0,70h and 0,65h correspondingly at the width of annular gap h.

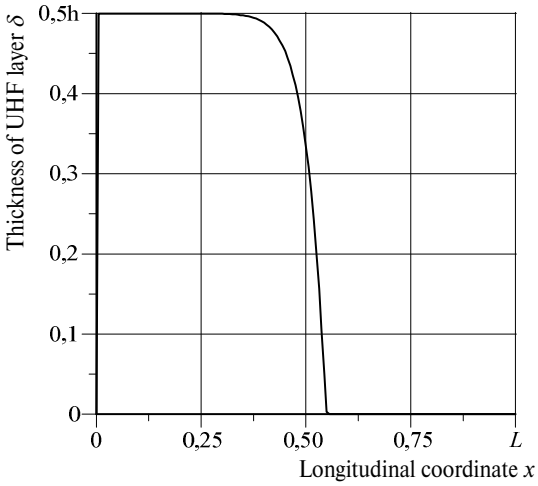

Fig. 5. *Dependence of thickness of desublimated UHF layer on longitudinal coordinate without area of nonsteady cooling of gas-vapor mixture at temperature of device wall of 243 K*

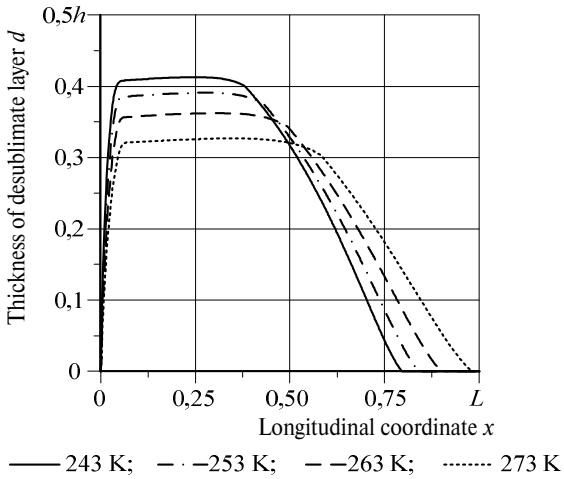

Fig. 6. *Dependence of thickness of desublimated UHF layer on longitudinal coordinate with the area of nonsteady cooling of gas-vapor mixture at different temperatures of the device wall*

Conclusion

The mathematical model of UHF cooling and desublimation processes from gas-vapor mixture entering to desublimator from the fluoridation reactor which allows qualitatively and quantitatively defining the influence of mass- and heat exchange, hydrodynamic characteristics on the examined processes, thereby optimizing technological parameters of the process and creating the system of automatic controlling the desublimation process was developed.

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ADSORPTION REFINEMENT OF WASTE TRANSFORMER OIL USING INDUSTRIAL MONTMORILLONITE-CONTAINING SORBENTS

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The possibilities of adsorption contact refining of waste transformer oil with active montmorillonite-containing sorbents of «Filtrol» series of BASF Catalysts LLC corporation and Zikeevsk M-80 deposit sorbent have been investigated. Usage of F-160 sorbents of «Filtrol» series in the refinement process allows achieving high quality degree of lean transformer oil, permitting its further use in the equipment with operating voltage to 750 kV.

Mineral oils step-by-step undergo deep changes in operating process as the result of accumulation oxidation products and degradation fragments of hydrocarbon base, products of resins firming, as well as products of wearing and corrosion of structural materials and fouling in them [1]. Oil purification and regeneration are the most perspective areas of resources recycling solving both economical and environmental problems.

For waste oils purification the same ways as for base oils purifications are used. They are distillation, acid-base purification, purification with solvent refining agents, contacting (adsorption refining), hydrofining. Treatment of oils, containing dissolved aging products, with adsorbents, for example natural or activated discoloring clays, is technologically efficient and simple way of oil purification [2, 3].

Samples of waste mineral transformer oils, weakly-contaminated (OST-1) and strong-contaminated (OST-2) ones, unsuitable for further use by such quality indices as acid number, chromaticity, loss-angle tangent, breakdown voltage have been chosen as the objects of investigation.

On the basis of literary data analysis and products availability on domestic market the sorbents of domestic and imported manufacturing have been chosen for oils contacting investigation (Table 1).

Table 1. *Base sorbents characteristic*

Name	Grade	Mineral composition	Manufacturee	Packed density, kg/m^3	Specific surface, m^2/g
Blanch soil of Zikeevsk deposit	M-80	Opal-cristobalite, montmorillonite, minor admixtures of silica and clinoptilita	«Sorbent» Russia	No data	80...200
Activated adsorbent of Filtrol series	F-1 F-160 F-105SF F-24 Mb LVM	Main component – montmorillonite	BASF Catalysts LLC corporation	500...800	250...400

Optimal process variables of waste oils adsorption refining, recommended by sorbent manufacturer, are the following: after water removal by centrifuging 0,5...10 % of adsorbent is entered into oil at temperature 80...120 °C and the mixture is stirred during 30...60 min. Adsorbent is separated from oil by filtra-