

LAWS OF NANOSIZE MOLYBDENUM (VI) OXIDE LAYERS OPTICAL PROPERTIES CHANGE AS A RESULT OF HEAT TREATMENT

N.V. Borisova, E.P. Surovoy

Kemerovo State University

E-mail: epsur@kemsu.ru

The spectrophotometric method determines two absorption and reflection spectral areas of nanosize MoO₃ layers – short-wave $\lambda < 330$ nanometers and long-wave $\lambda > 330$ nanometers. It is stated by spectrophotometric, gravimetric and microscopic methods that in atmospheric conditions MoO₃ layers transformation degree ($d=10...130$ nm) grows at time (1...140 minutes) and heat treatment temperatures ($T=373...600$ K) (at constant layer thickness) increasing as well as at reduction of layers thickness. The reduction of absorption maxima at $\lambda=350$ nm and increase at $\lambda=870$ nanometers at heat treatment of MoO₃ layers is revealed. The colour centers formation model is offered. It includes the center formation – anionic vacancy with one seized electron ($[(Va)^{++}e]$) during preparation of MoO₃ layer, thermal electron transition from a valent zone on a level of the center, the second electron capture by the center ($[(e Va)^{++}e]$).

Obtaining nanosize layers of various materials, ascertaining the nature and laws of the processes occurring under the influence of different energy factors are of great interest both for solid state physics and chemistry and general theory of heterogeneous catalysis and in connection with the necessity of developing real systems with controlled susceptibility level to various external influences. Among various inorganic materials the molybdenum oxide (VI) takes a special place. Molybdenum oxide (VI) and systems on its base attract attention of researchers of different profiles [1–18]. MoO₃ is used for obtaining molybdenum (its alloys, many other molybdenum compounds) applied as a constituent part of ceramic clays, glazes, enamels, colors. It is used as a catalyst in organic synthesis at oil processing (cracking, hydrofining, reforming) it is added as an admixture to motor oil. Molybdenum oxide (VI) coated on different carriers (titanium dioxide, silica) causes photostimulated conversion of methane and methane-containing gas mixtures (in different gas compositions) with rather high yield of methanol, formaldehyde, CO, CO₂ [13–14]. Devices on the basis of molybdenum oxide (VI) may be recommended for being used as electrochrome and photochrome monitors [5, 13, 17], electrochrome glasses or light redistributing filters [4–6], sensors for controlling gas content in atmosphere [10–12]. The main regulating (recording) elements in these devices are thin layers (films) of molybdenum oxide (VI). It is also known that optical and electrophysical properties of different materials thin films depend significantly on their thickness, conditions of obtaining, backing material [19, 20]. Mentioned practical value as well as absence of information in domestic and foreign literature by the present moment about systematic investigations of dimensional effects influence on optical properties of molybdenum oxide (VI) films make the task of complex investigation of optical properties in nanosize layers of molybdenum oxide legitimate and timely.

The results of investigation cycle intended for ascertaining nature and laws of the processes occurring in the conditions of atmosphere in nanosize layers of MoO₃ of different thicknesses depending on temperature and time of thermal effect are presented in the given paper.

Objects and methods of investigation

Samples for investigation were prepared by the method of thermal evaporation in vacuum ($2 \cdot 10^{-3}$ Pa)

by coating thin (10...130nm) MoO₃ layers on glass substrate using vacuum universal post VUP-5M. Boats made of molybdenum with the thickness of $d=3 \cdot 10^{-4}$ m were used as vaporizer. Optimal distance from the boat-vaporizer to the substrate is 8...9 cm.

Glasses from photo plates with the thickness of $1 \cdot 10^{-3}$ m and area of $2 \cdot 10^{-4}...4 \cdot 10^{-4}$ m² were served as substrates. They were subjected to preliminary treatment in concentrated nitric acid, in potassium dichromate solution in concentrated sulfuric acid, in boiling soapsuds, washed out in distilled water and dried [21,22]. Treated substrates are optically transparent in the range of 300...1100 nm.

The thickness of MoO₃ films was determined by spectrophotometric, microscopic and gravimetric methods [21]. Gravimetric method of quartz microweighing is based on determining mass increment (Δm) per unit of quartz resonance surface (with the thickness of $h=0,1$ mm) after coating MoO₃ film on it. Resolving power at thermal stabilization of resonators on the level of $\pm 0,1$ K amounts to $\Delta m=1 \cdot 10^{-8}...1 \cdot 10^{-9}$ g/cm². Average thickness of the film after weighing was calculated by the formula:

$$d_n = \Delta m / F_n \cdot \rho_m,$$

where Δm is the increment of quartz resonator mass after MoO₃ film coating, F_n is the film area on the substrate, ρ_m is the specific weight of coated substance [21, 22].

Samples were subjected to thermal treatment in drying chambers BE 300» and «SPT-200», in muffle furnace «Tulyachka-3P» in the temperature range of 373...600 K. In this case samples were placed on a porcelain plate heated to the proper temperature and subjected to thermal treatment during 1...140 min in atmospheric conditions. Effects of the researched samples before and after thermal treatment were recorded by gravimetric, microscopic and spectrophotometric (in the range of wave lengths 190...1100 nm using spectrophotometer «Shimadzu UV-1700») methods.

Results and discussion

Researching optical properties of nanosize MoO₃ layers coated on glass substrates before and after thermal treatment in atmospheric conditions it was stated that absorption and reflection spectra of the samples before

thermal treatment depend considerably on their thickness. Representative absorption and reflection spectra of MoO₃ layers of different thicknesses in the range of ($d=20\dots130$ nm) are presented in Fig. 1 and 2 as an example. It is seen that two spectral absorption and reflection regions typical for MoO₃ films and monocrystals may be singled out for the samples of different thicknesses [4, 6, 9, 10, 17] – the short-wave $\lambda < 330$ nm and long-wave $\lambda > 330$ nm. Determining optical width of forbidden zone of MoO₃ layers is substantially complicated due to absorption band in the range of $\lambda=330\dots400$ nm with the maximum at $\lambda=350$ nm. After preliminary thermal treatment of the samples in the temperature range of 373...600 K during $\tau=1\dots120$ min the absorption band with the maximum $\lambda=350$ nm practically disappears. The edge of the absorption band of MoO₃ films was estimated by the formula [23] using absorption spectra of the samples subjected to thermal treatment. It is stated that the edge of the absorption band of MoO₃ layers is found out at $\lambda \approx 320$ nm. This value coincides satisfactorily with the edge of absorption band determined by the diffuse reflection spectra of fine-crystalline powders and by the results of measuring transmission spectrum of fine MoO₃ films coated on quartz substrate [8].

At layer thickness of $d \approx 10\dots20$ nm the structureless absorption is observed on absorption spectra. At increasing MoO₃ layers thickness the absorption increases and diffused absorption band with the maximum at $\lambda=500$ nm starts forming in the region of absorption edge. As the MoO₃ layers thickness is increased ($d \approx 20\dots70$ nm) the shift of the diffused absorption band with the maximum at $\lambda=500$ nm into long wave spectrum region with the maximum at $\lambda=1020$ nm is observed at simultaneous formation of absorption band in the range of $\lambda \approx 400\dots600$ nm. At MoO₃ layers thickness of $d \approx 70\dots90$ nm the maximum absorption at $\lambda \approx 500$ nm is appeared and at $\lambda > 750$ nm the absorption increase is observed. At MoO₃ layers thickness in the range of $d \approx 90\dots120$ nm two diffused maximums appear on absorption spectra at $\lambda \approx 450$ nm and 700 nm. Absorption and reflection bands identification for the initial MoO₃ layers in long wave spectrum region requires thorough research of surface state, determination of presence and ascertainment of admixtures parts, structural and intrinsic defects.

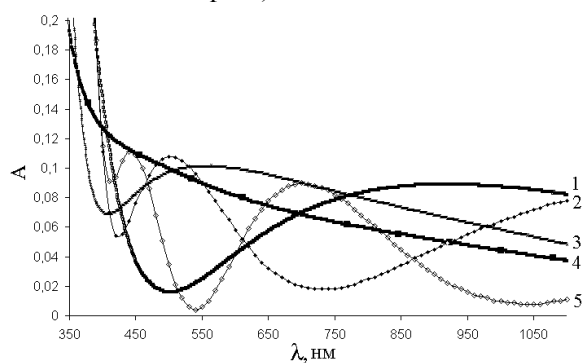


Fig. 1. Absorption spectra of molybdenum oxide (VI) films with the thickness of: 1) 58, 2) 94, 3) 30, 4) 23, 5) 122 nm

As a result of thermal treatment of MoO₃ layers of different thickness in temperature range of 373...600 K

in atmospheric conditions the absorption and reflection samples spectra undergo significant changes. And the observed changes of absorption and reflection spectra as well as limiting values of optical density in absorption bands maximums after samples thermal treatment depend on initial MoO₃ film thickness, temperature and time of treatment. Absorption spectra of MoO₃ films with thickness $d=46$ nm before and after thermal treatment at 423 K are given in Fig. 3 as an example.

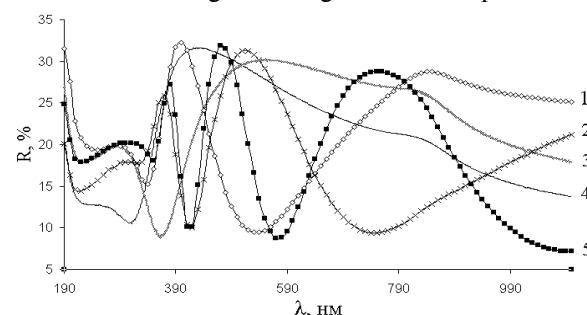


Fig. 2. Reflection spectra of molybdenum oxide (VI) films with the thickness of: 1) 58, 2) 94, 3) 30, 4) 23, 5) 122 nm

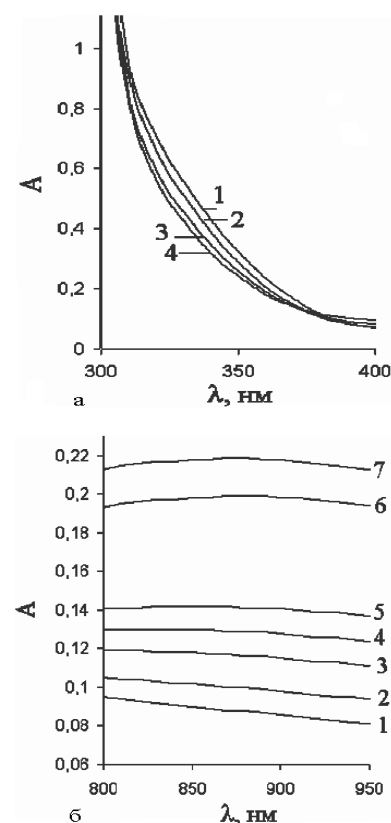


Fig. 3. Absorption spectra of molybdenum oxide (VI) layer with thickness of 46 nm before and after thermal treatment at 423 K: (a) 1) without thermal treatment, 2) 10, 3) 60, 4) 160 min, (b) 1) without thermal treatment, 2) 2, 3) 5, 4) 10, 5) 20, 6) 60, 7) 160 min

At thermal treatment the following changes are observed on absorption spectra of MoO₃ layers: first-of all optical density of the sample in the range of $\lambda=330\dots400$ nm with the maximum $\lambda=350$ nm (centre 1) decreases (that results in the shift of absorption band edge to the short-

wave spectrum region) and increases in the range of $\lambda=400\dots 1000$ nm with the $\lambda=870$ nm (centre 2). At increasing or decreasing thermal treatment temperature the laws of absorption spectra changes are remained regardless of MoO_3 layers initial thickness – the decrease of samples optical density is observed in the short-wave spectrum region and as a result the shift of absorption band edge into the region of short wave length. In the long-wave spectrum region the increase of optical density is observed for the samples with the thickness of $d>20$ nm and the decrease of optical density for preparations with the thickness of $d<20$ nm. More rapid increase of change effects of optical density occurs at the same initial thickness of MoO_3 layers at thermal treatment temperature rise. As the thickness of MoO_3 layers increases (up to 130 nm) at constant temperature (in the range of 373 ...600 K) and time of thermal treatment the consecutive decrease of change effects of samples optical density is observed in the whole studied spectral range. The limiting values of optical density changes at increasing the thickness of MoO_3 layers grow.

To ascertain the laws of occurring the thermal conversion process of molybdenum oxide films (VI) the kinetic dependences of conversion degree $\alpha=f(\tau)$ (where τ is the time of thermal treatment) at different wave length and thermal treatment temperature were calculated and plotted. To plot kinetic curves in coordinates $\alpha=f(\tau)$ the following approach was applied.

The absorption spectra of MoO_3 layers measured at different times of thermal treatment cross in one (isobathic) point (its position depends on MoO_3 layer thickness, temperature of thermal treatment) in which optical density does not depend on thermal treatment time. On the left and on the right of isobathic point absorption (A) depends on the time of thermal treatment and at certain time of thermal treatment it is formed of absorption connected with the presence of centre 1 (A_{C1}) and centre 2 (A_{C2}):

$$A_{\text{sample}} = A_{C1} + A_{C2}.$$

If we denote by α the degree of centre 1 thermal conversion into centre 2 then at $\lambda=870$ nm of the corresponding spectral region in the range of which centre 2 absorbs and centre 1 does not practically absorb light (Fig. 3) the current optical densities of centre 1 (A_{C1}) and centre 2 (A_{C2}) may be presented in the following form:

$$\begin{aligned} A_{C1} &= A_{C1}^1(1-\alpha), \\ A_{C2} &= A_{C2}^1\alpha, \end{aligned}$$

where A_{C1}^1 , A_{C2}^1 are the limiting optical densities of centre 1 and centre 2 at $\lambda=870$ nm.

As a result we obtain the following expression for the degree of centre 1 thermal conversion into centre 2

$$\begin{aligned} A_{\text{sample}} &= A_{C1}^1(1-\alpha) + A_{C2}^1\alpha, \\ \alpha &= (A_{C1}^1 - A_{\text{sample}}) / (A_{C1}^1 - A_{C2}^1). \end{aligned}$$

Light wave from the radiation source falling by normal on the surface of any system undergoes mirror reflection, diffusion, absorption and transmission [24, 25]. When passing through the boundaries of several media (air – MoO_3 – glass substrate – air) with different refraction coefficients n the specular reflected light wave R is formed of several components:

$$R = R_1 + R_2 + R_3,$$

where R_1 , R_2 , R_3 is the specular reflected light wave off the boundary: air – MoO_3 , MoO_3 – glass substrate, glass substrate – air.

Thus, the complete value of optical density measured in real conditions at spectrophotometer includes (at least) several constituents

$$A = A_{\text{sample}} + A_{\text{refl}} + A_{\text{diff.}}$$

where A_{sample} , $A_{\text{refl.}}$, $A_{\text{diff.}}$ is the value of sample optical density either stipulated by the losses for glass reflection or diffuse scattering of light by sample surface.

It was stated by special investigations that diffuse scattering of MoO_3 films by surface is negligible in comparison with glass reflection (Fig. 2) and as a result $A_{\text{diff.}}$ may be considered to be ≈ 0 . Then

$$A = A_{\text{sample}} + A_{\text{refl.}}$$

After simple transformations the final formula for calculating real (caused by light absorption in substance) value of optical density is

$$A_{\text{sample}} = A + \lg(1-R).$$

It was stated that the degree of MoO_3 layers conversion depends on their initial thickness, temperature and time of thermal treatment. As the time of thermal treatment increases the degree of MoO_3 layers conversion (calculated by optical density change in absorption band of centre 2) rises. Kinetic curves of MoO_3 layers conversion degree at 423 K depending on the initial samples thickness are given as an example in Fig. 4.

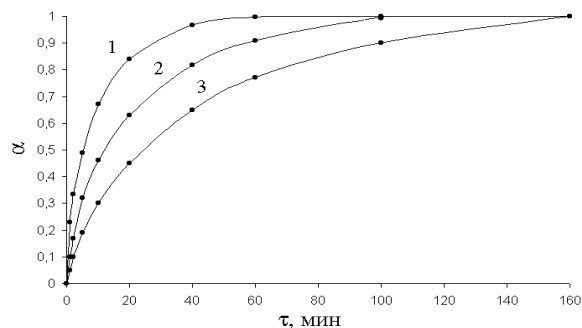


Fig. 4. Dependence of conversion degree on films thickness of molybdenum oxide (VI) at 423 K: 1) 28, 2) 59, 3) 89 nm

As the thickness of MoO_3 layers decreases (at constant time of thermal treatment) the degree of conversion in all studied temperature range increases. The increasing of thermal treatment temperature (at constant thickness of MoO_3 films) results in rise of thermal conversion rate (Fig. 5).

It was stated by the authors of [8] that the absorption band with the maximum at $\lambda=350$ nm for MoO_3 monocystals is connected with stoichiometric lack of oxygen and stipulated by oxygen vacancies with one captured electron $[(V_o)^{++}e]$ (the analogue of F -centre). This centre is obviously formed in the process of preparing MoO_3 layer of different thicknesses. Depth of occurrence of this $[(V_o)^{++}e]$ -centre amountes to $E_F^1=3,54$ eV. We suppose that the decrease of absorption maximum at $\lambda=350$ nm as well as formation of absorption maximum

at $\lambda=870$ nm at thermal treatment of MoO_3 layers are the interconnected processes and they are the result of centre $[(V_a)^{++}e]$ transformation.

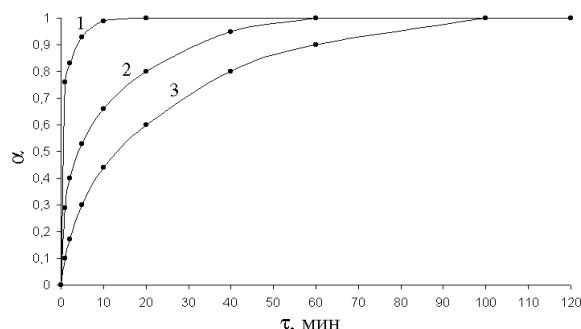
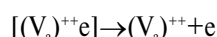


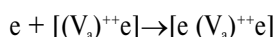
Fig. 5. Dependence of conversion degree of molybdenum oxide (VI) films with the thickness of 50...60 nm on temperature of treatment 1) 573 K, 2) 523 K, 3) 473 K

It is known [23] that at stimulation of solid body electron subsystem the electron transition in k space from valence band to the conduction band, from valence band on the acceptor level, from the donor level to the conduction band, from the low filled up level to the upper empty one may occur.

To secure electron transition from the filled up low level to the upper empty level at thermal stimulation of solid body electron subsystem and ensure enough rate of this process it is necessary for the average phonon energy (kT) to correspond to the value of surmounted energy barrier. The $[(V_a)^{++}e]$ -centre may be transformed by means of electron transfer from the level of centre occurrence level to the bottom of conduction band



(energy E_F^1 is required for this process) or by means of electron transfer from the top of valence band to the centre level



(energy $E = E_{\text{egw}} - E_F^1$ is required for this process, where E_{egw} is the thermal energy gap width of MoO_3 , $E_{\text{egw}} = 3,54$ eV is smaller by 0,2...0,3 eV than optical energy gap width [8]). Let us estimate the possibility of

carrying out the stated processes in real conditions of the experiment. Phonons are not homoenergetic. Their distribution over the energies follows Boltzmann equation [23]. According to this equation there is always the probability of the fact that the phonon with energy equal to $E_F^1 = 3,28$ eV or $E = 0,26$ eV exists at temperatures 373...600 K. (To ensure thermally activated transitions energy consumptions amount to 0,2...0,3 eV in optical [8]). The equation for the rate of thermal stimulation process for the electron with F -centre level to the bottom of conduction band or thermal electron stimulation from the top of valence band to the levels of F -centre may be presented in the following form

$$W = \nu N \exp(-\Delta E/k_0 T),$$

where ν is the frequency factor (for phonons in the order of dimension amounts to $10^{13}...10^{14}$), N is the concentration of $[(V_a)^+e]$ -centres, ΔE is the value of surmounted barrier ($E_F^1 = 3,28$ eV, $E = 0,26$ eV), k_0 is Boltzmann constant ($8,57 \cdot 10^{-5}$ eV/T), T is the temperature (600 K).

If we take concentration of $[(V_a)^{++}e]$ -centers by 10^{16} cm^{-3} (and consider that all anionic vacancies in MoO_3 are occupied by one electron in each one) then in ideal case (when all electrons reach the place intended for them and do not participate in other processes) the values for the rate of thermal stimulation process of the electron from the levels of $[(V_a)^{++}e]$ -centre to the bottom of conduction band or thermal electron stimulation from the top of valence band to the levels of $[(V_a)^{++}e]$ -centre amount to $W_1 \approx 2 \cdot 10^1 \text{ cm}^{-3} \cdot \text{s}^{-1}$ and $W_2 \approx 6 \cdot 10^{26} \text{ cm}^{-3} \cdot \text{s}^{-1}$ correspondingly. It follows from this that at thermal electrons stimulation $\approx 10^1$ electrons transfer from levels of $[(V_a)^{++}e]$ -centre to the conduction band in cm^3 MoO_3 per one second, that is a vanishingly small amount. The rate of the process of electron thermal stimulation from the top of valence band to the levels of $[(V_a)^{++}e]$ -centre is rather high for supporting further conversion of MoO_3 layer. Probably, the wide absorption band with the maximum at $\lambda=870$ nm is connected with the formation of $[e(V_a)^{++}e]$ -centers.

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INFLUENCE OF HOT HYDROGEN ON WATER BOILING

A.P. Astankova, A.P. Ilyin, A.Yu. Godymchuk

TPU Research Institute of High Voltages, Tomsk
E-mail: astankova@rambler.ru

The influence of dispersion and aluminium powder content in aqueous suspension on aluminium oxidation with liquid water has been investigated. It is stated that after heating aqueous suspension of electroblasting aluminium nanopowder to 64...66 °C aluminium oxidation process is characterised by the subsequent induction period and possible suspension self-heating with heat and hydrogen evolution. It is shown that at aqueous suspension self-heating the boiling temperature does not exceed 94 °C. The mechanism of water boiling is described.

Introduction

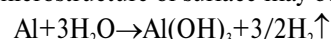
At present the considerable experimental results showing that nanoparticles and nanopowders are the new state of substance characterized by properties of solid and at the same time liquid and macromolecules are cumulated [1]. Due to its structure-energy state they present the number of unusual properties [2].

It is known that each substance in liquid state boils at certain temperature and external pressure. Boiling is the process of liquid evaporation not only from open surface but also in its depth where vapor bubbles start forming. Usually there is dissolved or absorbed air in liquid or in vessel walls where it is placed. Small formed gas bubble is filled up with saturated vapor of surround liquid. Vapor elasticity in it is determined by liquid temperature. If liquid temperature is so that saturated vapor pressure in a bubble is lower than external pressure over liquid the bubble does not grow. Both hydrostatic pressure of liquid column over it and external pressure under which there is liquid prevent this.

The external pressure defines bubble equilibrium state. If external pressure is increased the bubble will shrink. If external pressure is decreased bubble volume will grow. Let external pressure does not change but

temperature increases. When liquid temperature reaches the value at which the elasticity of its saturated vapor equals to external pressure the vapor pressure inside the bubble will also equal to the external one. Further temperature increase results in vapor pressure exceeding the external one inside the bubble, the latter starts growing, emerging and bursts exhausting when reaches the surface. Liquid starts evaporating not only from the surface but also from bubbles surface inside liquid: liquid is boiling. Thus, for liquid boiling it is necessary to bring its temperature to the magnitude at which the elasticity of its saturated vapors equals to the external pressure, or rather a bit higher [3].

At aluminum nanopowder (ANP) interaction with water suspension boiling is observed. Thus, molecular hydrogen and aluminum oxides-hydroxides with the developed microstructure of surface may be obtained:



The interaction of Al with water is exothermic. Its standard enthalpy is –459,1 kJ/mole. The immediate result of heat release is water temperature rising. At certain temperature the aluminum oxidation reaction with spontaneous heating of suspension and subsequent rising the environmental (water) temperature to a boil is possible.