REVIEW ARTICLES

HEAT AND MASS TRANSFER IN VACUUM METALLIZATION OF POLYMERS

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Applying metal coatings to the surface of polymers makes it possible to obtain materials that combine the useful properties of the ingredients. The use of such materials was, in many respects, decisive for the achievements of optics, electrical and radio engineering, chemical technology, and a number of other branches of industry. Moreover, in the near future an even broader utilization of vacuum technology in metallizing polymer materials may be possible, but this depends on the technical equipment, on the devising and introduction of continuously operating automatic vacuum metallizing machines into the technological process, and on success being achieved in the field of studying the mechanism of the formation of metal films on the surface of polymer bodies.

In its fairly simple technological execution, vacuum metallization of polymers involves the successive processes of formation of an adsorption phase, and also the nucleation and growth of a condensed phase whose mechanism is complex and insufficiently researched, even for relatively simple two-layer systems. In the metallization of polymers, the processes of adsorption and condensation are substantially affected by peculiarities of the state of the surface manifesting themselves primarily in the specifics of the molecular and supermolecular structure of the material of the substrate, its low surface energy, and good rheological properties.

A considerable number of recently published works had as their object the detailed study of certain aspects of the process of interaction of metal atoms with the surface of a polymer, especially of mass and heat transfer in adsorption and condensation. Two principal trends of the practical application of the results of the investigations thereby emerged: improvement of the technology of applying vacuum coatings by optimizing the existing and devising new methods of surface activation, and study of the properties of the surface of polymers by the method of recording the effects taking place upon interaction of the surface with a flux of metal atoms.

Such effects, most widely used in research practice, are the selective deposition of metal atoms on sections with electrophysical and structural inhomogeneity (vacuum decorating of the surface) and nonsteady reevaporation of metal atoms from the polymer surface at the stage of formation of an insular film. On the basis of established bonds of the structure, of the physicochemical state of the surface, and of the kinetics or intensity of the process of transfer of metal atoms, the correlations are determined which may be used for devising methods of evaluating the surface properties.

Kinetics of the processes of mass transfer in adsorption and condensation of metal atoms on polymer surfaces. The special features of the kinetics of metallization of polymer materials are most pronounced at the initial stage of deposition at which the interphase bonds between the metal and the polymer are formed. If the incident flux of metal atoms has constant density, then the condensation number at the initial stage changes in a complex manner with time [1-3]. At a certain, fairly low filling of the surface, a considerable part of the atoms (for a number of systems up to 60%) is reevaporated, after which the condensation number increases and becomes stabilized. With practically equal nature of the kinetics of condensation for different polymers and metals, the minimum condensation number and duration of nonsteady condensation depend chiefly on the temperature of the substrate and on the nature of the interacting substances.

It was established that when the density of the incident flux of atoms increases, the values of K and τ_0 decrease both for metallic [4, 5] and for high-molecular substrates [6]. For the investigated regimes of metal deposition on polymer surfaces, the relation $J\int_0^{\tau_0} K dt =$

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const is fulfilled, i.e., the condensation number becomes stabilized at the instant when an equal number of atoms is being deposited on the surface of the substrate. Investigation of the state of the condensed phase by electron microscope showed that this stage corresponds to the formation of the insular structure characterized by fairly large distances between nuclei [2], which is possible only when the growth of the dispersed film is of diffusional nature on account of the high mobility of the metal atoms on the surface of the polymer. These results enable us to examine the process of condensation at the initial stage as a diffusion problem, and to use the analytical expressions obtained in its solution for describing the kinetics of the growth of the metal phase. The necessity of taking into account the reevaporation and its nonsteady state at the initial stage of deposition causes a number of difficulties in formulating and solving the diffusion problem. For a homogeneous and isotropic surface of the substrate in the approximation R = const (taking into account the mobility of the metal adatoms only), the authors of [7, 8] obtained analytical expressions describing the change of concentration of atoms on the surface and of the size of the isolated nucleus of metal phase with time. Also determined was the size of the zone of capture on which the condensation number linearly depends [9]. A more general problem, the diffusional growth of the system of nuclei upon nonsteady reevaporation of metal atoms, was examined in [10, 11].

Under certain simpler conditions (e.g., with $\tau \gg \tau_a$ or $\tau \ll \tau_a$), the known relations for the concentration of adatoms and the capture zones [7-9, 12, 13] follow from the obtained expressions. On the basis of the results of investigations by electron microscope and the obtained analytical expressions, values of τ_a, D, and x were obtained for a number of metal—polymer systems that are indispensible for calculating the technological regimes of applying the metal phase with specified dimensions of the nuclei.

We want to point out that when the density of the nuclei is fairly high and their size fluctuates, mass transfer is greatly affected by processes of disintegration of small atomic clusters and by their diffusion. Experimental investigations carried out directly in an electron microscope to study the growth of copper crystals on a carbon film, which by its chemical composition is similar to polymers, showed that, around the largest islets, a fairly large zone free of nuclei forms [14, 15]. Surface migration of clusters, which manifests itself upon deposition on substrates with low surface energy at high temperatures, also exerts a substantial effect on the distribution of nuclei and on the kinetics of their growth [16]. Calculation of mass transfer through the diffusion field of adatoms, taking interaction of nuclei into account, is extremely complicated, and the existing literature in this field provides, for the time being, only a qualitative notion.

When only the effective thickness of a coating has to be calculated, the phenomenological model of reevaporation [17] is of a certain interest. On the basis of the present notions of the theory of adsorption and condensation, and of the experimental dependence of the electronic work function on the degree of filling of the surface, expressions were obtained describing the kinetics of change of the condensation number. The satisfactory coincidence of the experimental and theoretical dependences indicates that this approach is justified.

A fairly promising aspect seems to be the use of the theory of similarity [18-21] for calculating mass transfer in the metallization of polymers. The system of criteria must in that case be chosen with a view to the nonsteady reevaporation, the charge state of the reevaporated particles, and the temperature field which changes in the process of deposition. Therefore, as complement to the heat and mass transfer Nusselt and Stanton numbers [18, 19], we must include in the examination also their electrochemical analogs [21] which take the existence of moving charged particles and electric fields into account. The advantage of introducing this kind of criterion consists in the fact that, for any molecular transfer process, the Nusselt numbers are equal to the gradients of the corresponding field in dimensionless coordinates. Consequently, the electrodynamic Nusselt number, similarly to the heat and mass exchange number, is a magnitude depending on the geometrical parameters of the system, and it is equal to the density gradient of the charged particles near the surface. The notion of the kinetic and temperature dependences of deposition in the form of criterial equations and their solution make it possible to determine more accurately the thermophysical aspects of the process of interaction of metal atoms with the surface of the polymer. At the same time it should be pointed out that there are practically no works in the literature that deal with this aspect, and that the subject has not been developed to the stage of calculating actual systems.

Effect of the Physicochemical State of the Polymer Surface on Processes of Mass Transfer. Since polymers are in most cases amorphous or partially crystalline bodies, they are characterized by a large number of morphological forms and by composite structures [22]. Because of the specific structure of the macromolecules, defects in the regular disposition of kinetic units in crystalline polymers are inevitable, and they do not occur only on the crystal boundaries. In addition to that, the macromolecules of many polymers have dipole moments due to the shift of electron density upon combination of polar groups of type $-OH$, $-H$, $=O$, whose distribution on the surface is determined by the supermolecular structure. The processes of adsorption and condensation of metal atoms on the surface of polymers therefore have a number of special features.

The most substantial changes of practically all physicochemical properties of polymer materials are encountered when these materials are heated or cooled. However, studying the temperature dependences of mass transfer in metallization is made difficult by the possible heating of the polymer under the effect of heat radiation of the source of vapors, liberated heat of condensation, and a number of other energy factors [23-25]. Investigations of the effect of the temperature on the kinetics of deposition, with heating taken into account, showed that the temperature dependence is of a complex nature [26]. In the case of metallization of nonpolar polymers at the temperatures of transition from the vitreous to the high-elastic state and from the latter to the viscous-flow state, the condensation number is fairly small ("breakdown" of condensation occurs). On the basis of data of mass-spectrometric analysis of reevaporated and desorbed atomic and molecular fluxes [3], and also on the basis of calculation of the change in entropy near the temperatures of relaxational and phase transitions [2], it was established that the segmental mobility of the macromolecules has a substantial effect on mass transfer. This result fully coincides with the results of investigations of the kinetics of deposition in the mechanical glass transition of nonpolar polymers [3, 26].

In vacuum metallization of polar polymers the temperature dependence of the condensation number is determined by the thermal activation of the reevaporation processes, and when $T \leq T_{me}$, this dependence is monotonic. The established effect of the polarity of polymers on mass transfer during their metallization is due to the different nature of the centers of nucleation of the metallic phase [2, 3, 27] manifesting itself.

At sufficiently high surface temperatures ($T \geq T_{me}$), when high mobility of the macromolecules is realized and processes of their thermal destruction occur, the state of the condensed phase is determined to a large extent by mass transfer into the bulk of the polymer [28, 29]. With such regimes of metallization it is possible that the metal atoms interact chemically with the polymer and that synthesis of new metalloorganic compounds is effected, which is of considerable practical interest. It was also discovered that when the intensity of the incident flux of metal atoms is sufficiently low, a surface film does not form because of the diffusion of the metal atoms into the bulk. With a number of systems the atomic state of the metal is unstable, and within the bulk, processes of cluster formation with distinct texture occur. The mechanism of the formation of texture can be ascribed to the peculiarities of crystallization in the presence of adsorption-active media whose orienting effect was established by Valynskii et al. [30].

The processes of mass transfer in metallization are greatly affected by oxidation. For instance, with increasing concentration of carbonyl groups in the surface layers of polyethylene, the condensation number increases noticeably, and the systems that form are characterized by fairly high adhesive strength [2, 31]. The formation of an electret structure in the surface layers also helps reduce the intensity of reevaporated fluxes of metal atoms and increase the adhesion of combinations [32]; this makes it possible to use this technological method as an effective way of surface activation.

Of particular interest is the study of the peculiarities of the deposition of metal atoms on the surface of structurally inhomogeneous polymers. It was shown [33-35] that the regions with amorphous structure in nonpolar polymers have better adsorption properties than the regions of the crystalline phase. In the metallization of polar polymers the change in the supermolecular structure has no noticeable effect on the surface distribution of the nuclei. The selective nature of nucleation of the metallic phase, established upon deposition of nonpolar polymers of spherulitic crystal formations [33], may be used for studying their fine structure.

The molecular and supermolecular organization of the surface layers of polymers manifests itself chiefly in the nature of the surface diffusion of metal atoms which determines the morphological traits of the growing particles of the metallic phase, and in the surface distribution of the nuclei. On the example of the study of the peculiarities of metallization of a fairly large number of oriented polymers it was established that elliptical metal particles form on the surface, and from the size of the axes of these particles the diffusion anisotropy of the processes of surface mass transfer [36] and the anisotropy of heat conduction of the surface can be evaluated, which is of practical interest. On the basis of the results of the investigation, an effective method was suggested for the vacuum decoration of polymers with the object of studying their structure [37].

It is known that thin polymer films and their layers, applied to the surface of solids (boundary layers), have a number of properties that differ from the properties of the material in solid specimens [38-42]. Yet the application of thin polymer layers is widely used in preparing articles for metallization. It is therefore of particular interest to study the special features of mass transfer upon interaction of a flux of metal atoms with polymer boundary layers.

It was shown in [39-41] that thin layers of various substances, including polymers, are able to provide structural information on the substrate to which they were applied. A remote effect of the substrate can be discerned when the layer is several microns thick. The kinetics of deposition of atoms on thin layers also has a number of special features. It was established that the material of the substrate to which a polymer layer was applied also affects the nature of the temperature dependence of the condensation number [42]. For instance, when lead atoms are deposited on the surface of a polymer layer applied to aluminum, and on the surface of aluminum without such a layer, the nature of the temperature dependences of the condensation number is the same, but in deposition on the surface of a massive polymer the dependence has a completely different form. The conditions were determined under which the above-mentioned effect of transfer of the adsorption activity of the substrate manifests itself [6, 42].

Also established was the activating influence of metal on the adsorption properties of the surface layers of a polymer when adhesion contact forms between them and when it is subsequently broken up [32]. It should be pointed out that more detailed substantiation is required for the suggested mechanism of contact activation of polymers and of the anomalous effects in the boundary layers consisting in the formation of an electret structure in the polymers which is dynamically connected with the metal. Further research along these lines will not only provide results that are important to practice, it is also of great interest to the study of the physics of interphase and surface phenomena.

Special Features of the Accommodation of Metal Atoms on the Surface of Polymers. The existence of intense reevaporated fluxes of metal atoms at fairly low temperatures at the initial stage of deposition indicates incomplete accommodation of the atoms.

By the method of partial ionization of the atomic flux with simultaneous control of its density, Rogachev and Krasovskii [43] measured directly the mean kinetic energy of metal atoms reevaporated from the surface of polymers with different structure and chemical composition. The authors ascertained the kinetic peculiarities of the energy exchange and established the correlation of its parameters with the state of the condensed phase determined by mass transfer. We want to point out that the most general aspects of interrelation and interaction of processes of energy and mass transfer were discussed in considerable detail in [44-50], where it was demonstrated, in particular, that in a system consisting of several components (rarefied gas, a stream of spray-coated material, etc.) substance is transferred on account of a temperature gradient; this complicates the analysis of the metallizing process even more since it causes an additional separation of the components in space. In addition to that, it must be taken into account that processes of transfer in rarefied gas, compared with transfer in a continuum, lead to the appearance of so-called temperature jumps, i.e., a difference between the temperature of the wall and of the gas next to it [50]. This effect is usually taken into account by the introduction of some extrapolated lengths that increase the existing gap between the heat exchanging surfaces. The existence of a mass flux in dependence on its intensity complicates the general pattern, and it seriously affects the magnitude of the temperature jumps next to the walls [46] and the temperature distribution in the gap. At the same time the shape of the surface itself (a curvature) substantially affects the processes of transfer because the effective degree of rarefaction [51] changes. Increased curvature of the surfaces leads to increased effective degree of rarefaction, and mass transfer, just like heat transfer, leads to reduced rarefaction [46]. When these effects are not taken into account, it may lead to serious errors in the interpretation of the results of experimental investigations.

It should be pointed out that in the calculation of processes of energy transfer, which is complicated by mass transfer, it is also important to determine the nature of the distribution of the reevaporated molecules according to speed. Because of the nonsteady nature of adsorption and condensation, there is always a concentration gradient when polymers are being metallized, and, under these conditions, the distribution function according to speeds always differs from Maxwell distribution [45]. On the other hand, the form of the distribution function cannot be determined on the basis of the solution of Boltzmann's integrodifferential equation [48, 50] because there are no sufficiently complete experimental investigations of the kinetics of the process of interaction of the metal atoms with the surface of the polymer. Therefore, in the calculation of the temperature (or velocity) jump, occurring near the surface in case of incomplete accommodation [48, 49], there arises an indeterminacy when the accommodation coefficient [47, 48] or some other magnitudes, e.g., extrapolated lengths [46, 49], are used as criterion of energy exchange.

In the experimental investigation of the physicochemical aspects of energy exchange it was established that a correlation exists between the energy of the reevaporated atoms and the physicochemical properties of the material of the substrate [6, 52]. Polymers on whose surface the energy exchange is most intensified have lower electrical volume and surface resistance and higher permittivity. The correlation of W with such physical surface parameters as work function and surface energy [6] was studied. On the basis of the results of the investigations, a method of calculating these magnitudes with the aid of correlation equations was suggested, and they were determined for a number of polymers [43, 52]; this is very important because there is very little information of this kind available. Comparison of the results of measurement of the parameters of mass transfer (K_0, τ_0) and of energy exchange (W) makes it possible to make a relative evaluation of the energy distribution of the electron states [52].

On the whole, the investigations showed that the method of spectroscopy of reevaporated atoms is promising and, in some cases (e.g., in the study of temperature transitions [53]), it is the only relatively accurate and sufficiently efficient method of investigating the surface properties of polymers. Further research on these lines, in particular the study of the temperature dependences W(T) and of the influence of activation on energy exchange, will make it possible to broaden the potential of the method and to obtain additional information on the physicochemistry of the surface. In experiments, the use of the method causes practically no difficulties at all.

The complex investigations of the transfer processes in metallization and of the operational properties of the newly formed systems made it possible to establish a correlation between the parameters of evaporation of metal atoms and the adhesive strength of the metal—polymer bond [2, 32]. For different surface activation regimes it was demonstrated that when metal coatings are applied with a higher condensation number, they are adhesionally more strongly joined to the polymer substrate. The results prove that there is an influence of interphase interactions on the formation of the adhesive contact, and these results may be used for nondestructive quality control of adhesion in the metallization of polymers [54].

Thus the processes of mass and energy transfer in vacuum metallization have a number of special features, determined chiefly by the specifics of the physicochemical state of the surface. The established regularities of the influence of surface activation on the kinetics of the transfer processes make it possible, on the one hand, to obtain additional information on the state of the surface and, on the other hand, to optimize the technological regimes of metallization, and to predict the operational properties of the newly formed metal—polymer systems.

NOTATION

D, surface diffusion coefficient; J, flux density of the metal atoms incident on the substrate; K, condensation number; K_0, minimum condensation number; R, size of nucleus; τ, time; τ_0, duration of nonsteady reevaporation; τ_a, lifetime of atoms in the adsorbed state; T, temperature of the polymer; T_{me}, melting point; W, mean kinetic energy of reevaporated metal atoms; x, diffusion path length of adatoms.

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