

Measurement of coating thickness using laser heating

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Abstract. The analysis of thermal processes during the measurement of coating thickness with the use of heating with laser radiation is conducted. The obtained curves of the heating process allow determining thickness of the formed coatings.

1. Introduction

At present time to protect products from corrosion in aggressive environments and to enhance their heat resistance various types of coatings on metallic and dielectric surfaces are actively employed. For coatings of high thickness (100...500 μm) high-performance vacuum-arc deposition installations are usually used [1]. Drop phase in the products of sputtering may cause non-uniformity of film thickness. The uneven thickness of the coating reduces the lifespan of the products, because coating during service life is gradually destroyed and in the case of different thickness quickly enough on the product «clean» substrate areas appear, erosion of which will occur at high speed.

2. Analysis

Today there are a number of nondestructive methods of coating thickness control based on sensing using ultrasound or X-ray radiation, as well as measuring basic capacity of individual sections of a coating. Typically thickness of a substrate is several orders of magnitude greater than thickness of a coating and therefore use of X-ray diagnostics will be ineffective [2]. Measurement of thickness by elementary capacity is possible for dielectric coatings and it is not applicable for metal and semiconductor layers. Application of ultrasonic method is limited because of the strong attenuation of ultrasonic waves in thick coating layers, which hinders fixing of a weak reflected signal. In this regard development of nondestructive methods of coating thickness control, capable of exploring a significant surface of a large batch of products and determining status of the applied coatings, is very relevant. One way to solve this problem can be probing the surface by laser radiation with subsequent investigation of the dependence of sample temperature on time.

Lasers based on gas discharge, due to high density of the energy flow and high spatial uniformity, provide unique technological capabilities in this direction. The most widely applied in this case are molecular CO₂ lasers with a wavelength of 10.6 μm .

Let us review conditions of heating for measuring coatings thickness [3]. First of all we will consider the principles of heat conduction in one-dimensional approximation for unrestricted environment. It distributes heat flux in the direction of x axis and through the lateral surface of the object heat is not supplied. If environment settings are changed in the direction x and time t , then the



heat flux density q is dependent on coordinates and time $q(x, t)$. In an infinite environment let us imagine a cylinder and consider an infinitely small section with length dx and cross section area S .

The amount of heat that comes in the direction x through the cross section of the cylinder S during time dt is $q(x)Sdt$. The amount of heat radiated during time dt through the opposite base of the cylinder will be $q(x+dx)Sdt$. Then the total amount of heat passing during time dt through an area of cylinder dx is equal to

$$[q(x) - q(x+dx)]Sdt = (\partial q / \partial x)Sdxdt.$$

On the other hand this heat is determined by expression $cdTdm$, where c is specific heat capacity; $dm = \rho Sdx$ – increment of the cylinder mass; ρ – specific density. Equating these two formulas and making conversion we will receive the following:

$$\rho c \partial T / \partial t = \partial q / \partial x. \quad (1)$$

Let us consider distribution of heat in infinite homogeneous plate with thickness d . On one side of the plate temperature T_1 is supported and on the other side – T_2 ($T_1 > T_2$). The heat flow will be proportional to the temperature difference and inversely proportional to the plate thickness d . Then in this case heat flux can be written in the following form:

$$q = \lambda \Delta T / d, \quad (2)$$

where λ is coefficient of thermal conductivity.

Value of heat transfer by conduction depends on the temperature gradient, i.e. temperature difference at the ends of the cylinder to the distance between them $\Delta T / \Delta x$. It also depends on the cross section area of the cylinder and coefficient of thermal conductivity of the material, which characterizes the ability of a substance to conduct heat, and in general it also depends on temperature, structure, moisture, density and pressure.

Assuming that the plate is infinitely thin and x axis is pointing in the direction of decreasing temperature: $d = dx$; $T_1 = T(x)$; $T_2 = T(x+dx)$, thus

$$\frac{T_2 - T_1}{d} = \frac{T(x+dx) - T(x)}{dx} = \frac{\partial T(x)}{\partial x}.$$

Then formula (2) transforms into the following equation:

$$q = -\lambda \Delta T / d. \quad (3)$$

Relation (3) is derived from the assumption that x axis is directed in the direction of decreasing temperature. This ratio is the Fourier law of heat conduction; the minus sign indicates that heat is propagated in the direction opposite the temperature gradient.

Taking into account ρ , c and λ equation (1) takes the following form

$$\rho c \frac{\partial T}{\partial t} = \lambda \frac{\partial^2 q}{\partial x^2},$$

if coefficient of thermal diffusivity $a = \lambda / (\rho c)$, then

$$\frac{\partial T}{\partial t} = a \frac{\partial^2 q}{\partial x^2}.$$

Solving problems on propagation of heat in environment is linked to the solution of a parabolic equation. To determine uniqueness of the parabolic equation solution in a finite region D bounded by a surface S , it is necessary to define initial and boundary conditions taking into account:

- geometrical parameters characterizing size and shape of the body, in which process flows;
- physical parameters characterizing physical properties of a body (thermal conductivity, density, heat capacity, power of internal heat sources, etc.).

Initial and boundary conditions are set by physical formulation of the problem. The initial conditions are defined as follows: $T(r, t)_{t=0} = T(r)$. Boundary conditions depending on the temperature regime at the borders may be different.

The heat source affects the surface of the single layer sample creating a constant heat flux, which gradually heats the surface (figure 1a).

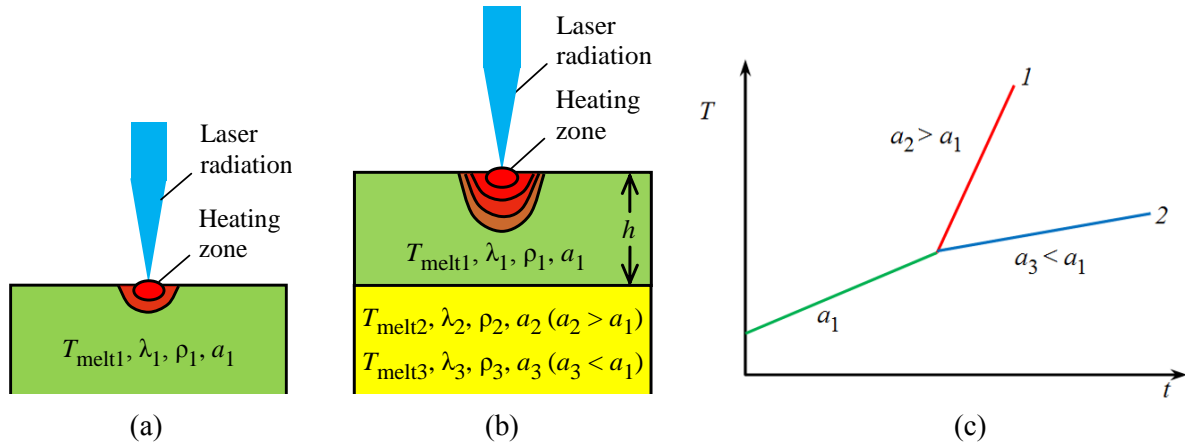


Figure 1. Heating of a sample surface by laser radiation: a – monolithic sample; b – two-layer sample; c – curves of the heating process.

If the heated surface is a two-layer sample with a finite thickness and is in metallic contact with the basis (figure 1b), the heat flows into a material with a thermal wave that travels a distance x in time t . Upon receiving heat from the heat source wave reaches the boundary between two media. Accounting that the thermal contact of a film to the substrate is ideal (adhesion of a film and substrate is ideal) this implies the equality of the temperatures and heat flows at their interface. Power dissipation in a film is uniformly distributed over the cross section, constant along the film thickness and constant throughout the duration of exposure.

If this surface is thermally insulated, then the rate of heat propagation at the boundary is reduced and temperature in the heating zone is raised (figure 1c, line 1). If not, the heat will be dissipated from the coating faster and the temperature rise will be slowed down (figure 1c, line 2). In this case, at the interface of two-layer material with an arbitrary initial distribution, boundary conditions of the fourth kind and heterogeneous asymmetric boundary conditions of the first kind at the external borders, when on the border of the body surface temperature is defined, are considered. For each layer its own equation of thermal conductivity is written:

$$\frac{\partial T_1(x, y, z, t)}{\partial t} = a_1 \Delta T_1(x, y, z, t), \quad \frac{\partial T_2(x, y, z, t)}{\partial t} = a_2 \Delta T_2(x, y, z, t),$$

while the boundary conditions are:

$$-\lambda_1 \frac{\partial T_1(x, y, z, t)}{\partial z} \Big|_{z=h} = -\lambda_2 \frac{\partial T_2(x, y, z, t)}{\partial z} \Big|_{z=h}, \quad T_1(x, y, z, t)_{z=h} = T_2(x, y, z, t)_{z=h},$$

where h is the boundary of a section (coating thickness).

Let us consider the analysis of thermal processes in the heated heat-conductive coating on the insulating substrate. Assuming substrate has thermal diffusivity so small compared to the coating that it can be neglected, at short times of heating the depth of coating heating $x = \sqrt{at}$ will be much less than the thickness of the coating. Thus, at the beginning of a process surface heating can be considered as heating of the monolithic sample with unlimited thickness. If heat is produced by a laser beam having a Gaussian distribution with radius r the following formula to determine the temperature in a center of a beam can be used [4]:

$$T_{11}(0, 0, t) = T_1 = \frac{\alpha I_s r}{\lambda \sqrt{\pi}} \operatorname{arctg} \left[\left(\frac{4at}{r^2} \right)^{1/2} \right], \quad (4)$$

where $T_{11}(0, 0, t)$ is a surface temperature at the center of a beam ($z = 0$; $r = 0$); α – absorption coefficient of the sample; I_s – density of incident power. As the depth of the coating heating becomes comparable with the thickness a different mathematical expression for the temperature is needed:

$$T_{12}(0, 0, t) = T_2 = \frac{\alpha I_s r^2}{4\lambda h} \ln \left[1 + \frac{4at}{r^2} \right], \quad (5)$$

Transition from the expression (4) to the expression (5) is smooth in nature (figure 2). For its determination it is more appropriate to consider not function $T = f(t)$ but the change of temperature increase speed.

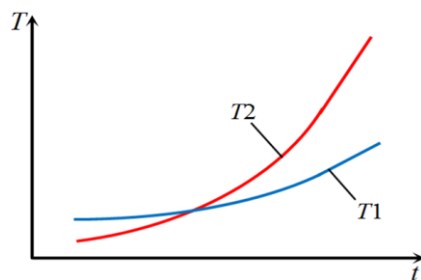


Figure 2. Time dependences of the laser heating.

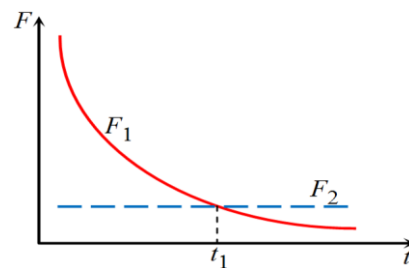


Figure 3. Function of the heating rate.

Let us differentiate equations (4) and (5) by time

$$\frac{dT_{11}}{dt} = \frac{\alpha I_s \sqrt{a}}{\lambda \sqrt{\pi}} \frac{1}{1 + (4at/r^2)} \frac{1}{\sqrt{t}}, \quad \frac{dT_{12}}{dt} = \frac{\alpha I_s a}{\lambda h} \frac{1}{1 + (4at/r^2)}. \quad (6)$$

Based on (6) auxiliary function F_i can be made:

$$F_i = \frac{\lambda}{\alpha I_s \sqrt{a}} \left[1 + \frac{4at}{r^2} \right] \frac{dT_{1i}}{dt}, \quad (7)$$

where $i = 1, 2$. It is obvious that $F_1 = (\pi t)^{-1/2}$ and $F_2 = a^{1/2}/h = \text{const}$. Hence there is a relatively simple method of finding h .

Simultaneously with the beginning of heating of the coating low-inertia radiation sensor detects the value of the continuously rising surface temperature, then using a PC in real time is taken the derivative dT/dt and the value of the auxiliary function F_i is calculated using (7). When the value of F_1 becomes equal to F_2 (time t_1 in figure 3) h is defined as $h = a^{1/2}/F_2$. In accordance with the physics of phenomena with increasing h the value of F_2 decreases and the time after which the heat flow reaches the insulating substrate grows. Naturally, after this time the rate of temperature rise becomes larger than it would be in the case of a monolithic sample.

Minimum thickness of the coating which can be evaluated by this technique is determined by the parameters of a coating and infrared receiver, as it has a certain response time t_{\min} : $h_{\min} = (a \pi t_{\min})^{1/2}$.

Let us consider coating of chromium carbide CrC ($\rho = 6.68 \cdot 10^3 \text{ kg/m}^3$; $c = 0.54 \text{ J/(g}\cdot\text{K)}$; $\lambda = 19.1 \text{ W/(m}\cdot\text{K)}$; $a = 5.24 \cdot 10^{-6} \text{ m}^2/\text{s}$) deposited on the insulating ceramics. Then, taking t_{\min} of the infrared receiver equal to $5 \cdot 10^{-4} \text{ s}$ calculated h_{\min} will be $90 \text{ }\mu\text{m}$.

Maximum thickness of the coating can be determined from the type of $F_1 = f(t)$ function. As can be seen from figure 3 with increasing time the steepness of this function falls that will hinder the fixation of the moment when F_1 becomes constant. If we assume that the minimum observed relative change of the function $\Delta F_1 / F_1 = \gamma$, then the maximum measurement time is defined as $t_{\max} = \Delta t / 2\gamma$.

Since $\Delta t = t_{\min}$ then $h_{\max} = (a\pi t_{\max})^{1/2} = (a\pi t_{\min} / 2\gamma)^{1/2}$. For the above example, assuming that $\gamma = 1\%$ the maximum measured thickness of a coating will be equal to $640\ \mu\text{m}$.

Required laser power can be evaluated from the expression (4):

$$I_s = \frac{T_{\max} \lambda \sqrt{\pi}}{\alpha r \arctg\left(\sqrt{4at / r^2}\right)}.$$

On the basis of this method was developed an experimental device for measurement of coatings thickness which consists of two main parts: test sample heating block and measurement unit. For the heating unit CO_2 laser with electro-magnetic modulator is used. For directing and focusing of the beam a system of mirrors and lenses is applied. The measuring device is a pyrometer, which is based on fixing temperature of an object by its own infrared radiation in the spectral range $2.0 \dots 3.1\ \mu\text{m}$. This pyrometer is adapted to operate in a wide temperature range with low inertia to continuously record the temperature. It includes a long-focus lens, allowing directing it to the treated area of a sample.

With the help of this complex experimental dependences of heating for samples of chromium carbide deposited on a ceramic substrate (figure 4a) and steel substrate (figure 4b) were obtained.

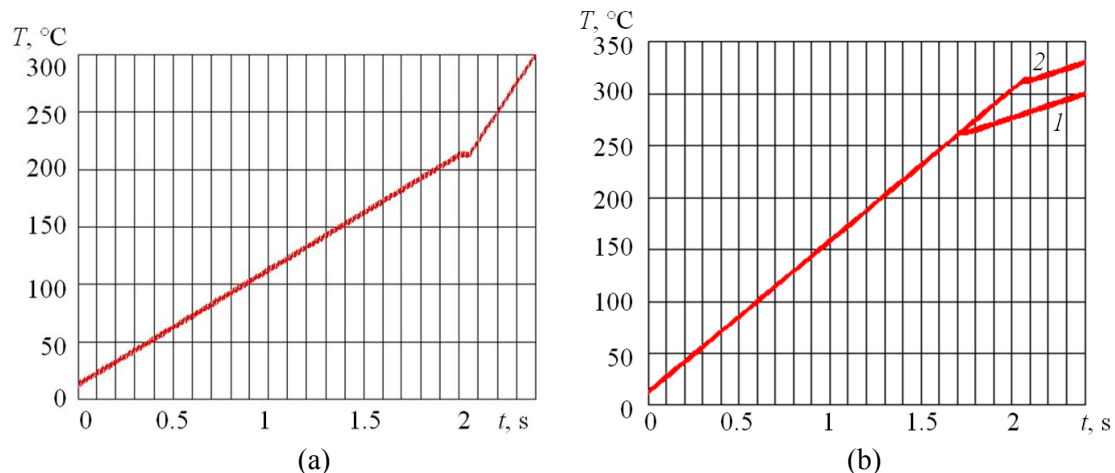


Figure 4. Curves of the heating process allowing determination of the coatings thickness:
a – chromium carbide on ceramic substrate; b – chromium carbide on steel substrate.

It is seen that while the thermal wave has not yet reached the boundary between two media, the temperature increases linearly. When thermal wave reaches boundary between materials a change in the course of schedule is observed. After that if the surface is insulated the rate of heat propagation at the boundary is slowed and temperature in the heating zone rises (figure 4a); otherwise temperature will be dissipated from the coating faster and the temperature rise will be slowed down (figure 4b). Also figure 4b shows curves for different coating thickness: 1 – $140\ \mu\text{m}$; 2 – $160\ \mu\text{m}$.

3. Conclusion

Considered method of determining coatings thickness extends the capabilities of the process control and diagnostics when receiving films and coatings of inorganic substances. When using a laser source with the controlled nature of radiation, this method allows determination of thickness of relatively thick coatings ($100 \dots 500\ \mu\text{m}$) with high accuracy.

4. References

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