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Estimation of the effective reflection coefficient from the titanium surface for the CO_2 laser radiation

K. Rożniakowski, J. Zimnicki, A. Drobnik

Institute of Physics, Technical University of Łódź, ul. Wólczańska 219, 93–005 Łódź, Poland.

B. Wendler

Institute of Materials Engineering, Technical University of Łódź, ul. Stefanowskiego 1, 90–924 Łódź, Poland.

In the present paper, an estimation method for the effective reflection coefficient R from the surface of a thin titanium layer deposited on the substrate of carbon steel using the technique of dc magnetron sputtering, is presented. The estimation of the reflection coefficient consists in comparing the structural changes in the titanium due to both conventional and laser annealing.

1. Introduction

One of the more prospective methods of thermal treatment of metals and alloys employs the laser radiation. The fundamental role in these processes is played by the reflection coefficient R of the electromagnetic radiation defined as $R = q/Q$, where Q and q are the power densities of the incident and reflected beams, respectively. The magnitudes Q and q are often found by using very complex calorimetric methods [1] or by exploiting the phase and structural changes in the material occurring due to the process of the laser beam interaction [2]–[5].

In the present paper, the estimation of the reflection coefficient R is based on structural changes in the titanium layer (obtained by the method of dc magnetron sputtering) evoked by a continuous wave CO_2 laser operation. Analogous changes were observed in the titanium layers during conventional annealing [6], [7]. The comparison of the results of both methods allows us to estimate the effective reflection coefficient R.

2. Experimental

In the experiment, use was made of the titanium layers of 4 μm in thickness obtained by the method of dc magnetron sputtering of a pure titanium disc. The layers were deposited on one of the front planes of the rolled samples of the carbon steel.

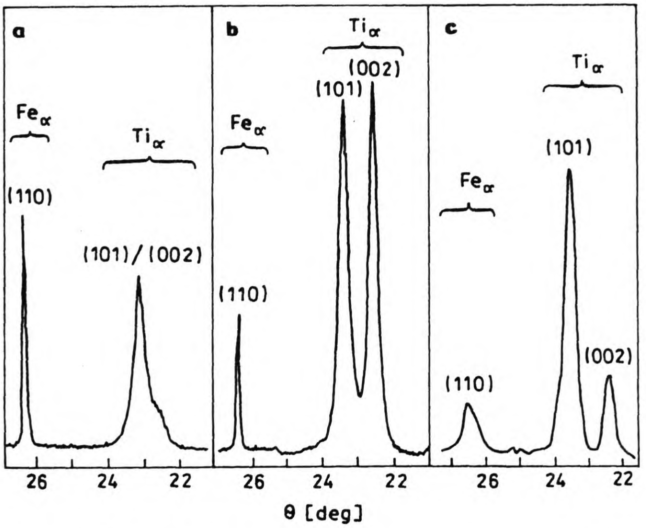
After having deposited titanium the structure of the Ti layer was examined by using a HZG-3 X-ray device (of continuous recording). The obtained diffractograms were compared with those of the layers subjected to conventional annealing in the temperature range 350–400° C, as well as with diffractograms of the layers treated (annealed) with the aid of a continuous wave CO_2 laser beam (we used Tryumph laser, 6.5 kW, $\lambda = 10.6$ μm) travelling along the layer.

A number of trials for different speeds and widths of the laser beam have been performed in order to establish the best consistence of the obtained diffractograms for both conventional and laser methods. The presented example refers to the case of the beam width $d \simeq 3.5$ mm, while the travelling speed was $v \simeq 0.27$ m/s. Thus, the exposure time of the layer was about $\tau = d/v \simeq 0.013$ s, as it is commonly accepted. In each trial the sample surface was swept only once.

The width of the analysing X-ray beam was equal to that of the laser beam.

3. Experimental results

As an example, the results of the X-ray analysis are shown in the Figure (figure **a** shows the diffractogram of the layer taken immediately after deposition of the latter). It may be seen that the annealing of the layer at a temperature of 400° C causes the reflexes (002) and (101) of the deposited titanium layer to move in the direction of their position for pure Ti (figure **b**). An analogous phenomenon is observed after exposing the layer to a laser beam of power density equal to about $Q \simeq 26 \times 10^7$ W/m^2 (figure **c**).



Diffractograms of titanium layers deposited on the carbon steel substrate using the dc magnetron sputtering method immediately after deposition (**a**), conventional annealing at a temperature of 400° C (**b**) and after irradiating with a CO_2 laser of power density $Q \simeq 26 \times 10^7$ W/m^2 and speed of beam sweeping $v = 0.27$ m/s (**c**). Radiation CoK_α, continuous recording

The structure of the titanium layer immediately after deposition differed slightly from that of the bulk material. The overlapping of the (002) and (101) reflexes (figure **a**) on the X-ray diffractograms used to be attributed (in the former work [2]) to the stress and strain of elementary cells of the titanium lattice due to considerable energy of the Ti atoms and ions. The later examinations showed that there is a significant quantity of dopants such as Fe (about 3 weight percent), Cr (about 1 weight percent) and Ni (about 0.5 weight percent) in the titanium layer [7] coming from the magnetron cathode housing, the latter being produced of stainless steel, which suffered from sputtering together with the titanium cathode. The overlapping of the (101) and (002) reflexes should be connected with this fact. The quantity of the dopants built in the crystal lattice of titanium significantly exceeds the solubility limits of the doping elements in titanium matrix. Thus, the lattice is in the metastable state. The conventional annealing of those layers at a temperature of about 400° C results in splitting of both the reflexes, which, in turn, is connected with the reversion of the titanium layer structure to that of the bulk material. This fact is attributed to the diffusive processes of the dopants travelling to the grain boundaries and the other defects of the crystaline structure. These processes, as it is well known, are of activation nature and thus depend strongly on the temperature.

It is obvious that in both cases considered (*i.e.*, of conventional and laser annealing) the defective structure of the deposited titanium layer transfers to the structure of more stable pure titanium at the same temperature. By estimating in such a way the layer temperature the reflection coefficient may be determined.

4. Estimation of the effective reflection coefficient R

It is well known that the electromagnetic radiation of the CO_2 laser applied in our experiment is absorbed in a very thin near-surface metal layer. Thus, it may be assumed that the whole energy of the laser beam is absorbed within the titanium layer of thickness $l \simeq 10^{-7}-10^{-8}$ m [8]. In the case of such a thin layer and suitably large laser beam diameter, it may be assumed that $d \gg l$, where d is the diameter of the irradiated area. The heat source appearing in the titanium layer due to the laser beam operation may be considered to be of surface character. In the case considered and under the assumptions that the distribution of the laser radiation power is of rectangle form, while the irradiated region is semi-infinitive, the temperature increment ΔT in the titanium layer may be estimated on the basis of the expression [9], [10]

$$\Delta T(x,t) = \frac{2Q(1-R)\sqrt{a_1 t}}{K_1} \times \left\{ \mathrm{ierfc}\left(\frac{x}{2\sqrt{a_1 t}}\right) + \sum_{n=1}^{\infty} (-m)^n \left[\mathrm{ierfc}\left(\frac{2nb+x}{2\sqrt{a_1 t}}\right) + \mathrm{ierfc}\left(\frac{2nb-x}{2\sqrt{a_1 t}}\right) \right] \right\} \tag{1}$$

where coefficient $m = \left(\frac{K_2}{\sqrt{a_2}} - \frac{K_1}{\sqrt{a_1}}\right) \Big/ \left(\frac{K_2}{\sqrt{a_2}} + \frac{K_1}{\sqrt{a_1}}\right)$ is determined by the thermal parameters of both the titanium layers (K_1, a_1) and the steel substrate (K_2, a_2); K – thermal conductivity coefficient, $a = K/c\rho$ – thermal diffusivity, c – specific heat, ρ – density, b – layer thickness, x – depth measured from the upper surface of the layer, Q — power density of the laser radiation, R — reflection coefficient, $\mathrm{ierfc}(z) = \pi^{-1/2}\exp(-z^2) - z\,\mathrm{erfc}(z)$, $\mathrm{erfc}(z) = 1 - \mathrm{erf}(z)$, $\mathrm{erf}(z)$ — error function.

The results of calculations show that the difference between temperature of the layer surface and the temperature at the depth b is small in this case (about 10 K) and, consequently, we may assume $x = 0$ and $t = \tau$ in formula (1) and then

$$\Delta T(0,\tau) = \frac{2Q(1-R)\sqrt{a_1\tau}}{K_1}\left\{\frac{1}{\sqrt{\pi}} + \sum_{n=1}^{\infty}(-m)^n \mathrm{ierfc}\left(\frac{nb}{\sqrt{a_1\tau}}\right)\right\}. \tag{2}$$

Relation (2) has been used to estimate the effective reflection coefficient R. Substituting $Q \simeq 26\times 10^7$ W/m^2, $\tau = d/v \simeq 0.013$ s, $\Delta T(0,\tau) = 380$ K (data from experiment) and assuming $K_1 = 21$ W/mK, $c_1 = 550$ J/kgK, $\rho_1 = 4500$ kg/m^3 for titanium, and $K_2 = 34$ W/mK, $c_2 = 461$ J/kgK, $\rho_2 = 7850$ kg/m^3 for steel [11], [12], we obtain the reflection coefficient $R \simeq 0.87$ which is well consistent with the calculation results reported in [13]. The average values of the thermal parameters for titanium and steel for the temperature range 0–400° C were accepted for calculations.

5. Summary

From the invstigations presented, it follows that the reversion process of the structure of titanium deposited in the process of dc magnetron sputtering to the structure of pure titanium occurs at a definite temperature. This holds for both conventional and laser annealing. The comparison of the results referring to the two experiments allows us to estimate the reflection coefficient for the titanium layer annealed with the CO_2 laser.

While comparing the results of both methods the different annealing times of the layer must be taken into account which lead to the same effect of splitting of the (002) and (001) reflexes of titanium. The trials carried out indicate that the conventional annealing for about 1 min. results in the splitting of reflexes. Further reduction of the conventional annealing time is impossible becuase of the thermal inertia deciding about the heating up of the whole sample to a suitable temperature. In the laser method, the same effect is achieved in much shorter time τ corresponding to the annealing time of the layer element. As it was pointed out in [14], the short-range diffusion is possible in the solid body (of order of a few interatomic distances) which is connected with the delivery of high energy in a short time (of order of 10^{-3} s) with the aid of laser beam. The diffusive processes may occur quicker in the case of laser beam operation due to considerable reduction of grain size in the layer (as a result of speedy heating and cooling [14]). Consequently, there occurs some shortening of the diffusion path of the dopants to the grain boundaries as well as to other defects of structure.

Independently, the examinations of the influence of the dopants of other sort (like Cu) introduced to the Ti lattice were carried out. The Cu dopant was introduced to the Ti layer when replacing the magnetron cathode housing formerly made of stainless steel by that made of copper sheet. Consequently, Ti layers doped with 2 weight percent of Cu were obtained. The annealing at a temperature of about 400° C resulted in reversion of the layer structure to that of the solid material, which was confirmed by X-ray examination.

Such a method of temperature determination may be applied also to other thin metal films deposited either with the help of the dc magnetron sputtering method or by using other methods. The necessary condition of its applicability is the appearance of metastable structure in the produced layer.

The kind of substrate plays no essential role in the structural transitions of the layer; for example, identical transitions in the metastable titanium layers at the same temperatures were obtained in the case of conventional annealing on the substrates of pure Ti. A decisive factor for the structural transitions in the layer is the suitable temperature (independently of the kind of substrate).

The presented way of estimating the temperature changes and consequently the estimation of the reflection coefficient R may be applied together with the known calorimetric methods. It may be presumed that in some cases the accuracy of the method described may appear to be higher.

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