Time saving technique for studying thermal processes in magnetron sputtering of titanium target

V A Pavlov, V I Shapovalov, D S Shestakov, A V Rudakov and A E Shabalin
Saint Petersburg Electrotechnical University "LETI", St. Petersburg, Russia
E-mail: vishapovalov@mail.ru

Abstract. The kinetics of thermal processes on a substrate during magnetron sputtering of a titanium target has been studied. A technique is proposed for reducing the time of the substrate heating temperature measurement, which is useful in performing extensive experiments in multifactorial problems. The selection criterion for the duration of the observation interval can be the relative error in extrapolating the stationary temperature of the substrate. The technique is universal in nature and allows to reduce the time of a single experiment by a factor of 2 – 3 with an error of no more than 5%.

1. Introduction
When a metal film is deposited by magnetron sputtering during 8–10 min, the substrate temperature can increase up to 100–200 °C [1]. The kinetics of thermal processes on a substrate has been studied by many authors. This problem remains to be interesting until now [2–7].

In order to reveal the influence of several independent variables on the kinetics of substrate heating, it is necessary to perform a large number of experiments. Therefore, the time required for a single experiment is of particular importance. Reaching a steady state takes time and leads to unnecessary loss of time, which can be avoided. The goal of this work is to develop a correct technique for reducing the time of measuring the kinetics of thermal processes on a substrate. The technique was developed on the basis of experimental results obtained during sputtering a titanium target.

2. Experimental details
The experiments were carried out in a system with a 7.8·10⁻² m³ vacuum chamber, in which the residual pressure was less than 10⁻² mTorr. A 130 mm diameter planar dc magnetron with a cold titanium target was installed in the chamber. Sputtering was carried out in argon at a pressure of 3 mTorr and a current of 2, 4, and 6 A. In order to study the kinetics of thermal processes on substrates, a thermocouple sensor with a sensitive element in the form of a copper disk of 1 cm² in area and 1 mm thick was used. The sensor is described in detail in [5]. Each measurement included heating the sensor during magnetron operation and cooling it down after turning off the source.

3. Results and discussion
Figure 1 shows the typical results of measuring the kinetic curves. The sections reflecting the processes of substrate heating and cooling describe exponentials with 0.998 confidence:

\[
T_h(I,t) = T_0 + T_x(I) \left[ 1 - \exp \left( -\frac{t}{\tau_h(I)} \right) \right],
\]

(1)
\[ T_c(t) = T_s(I) \exp \left( -\frac{t}{\tau_c} \right). \]  

In (1) and (2) the following notations are used: \( T_0 \) - constant component; \( T_s(I) \) - stationary temperature; \( \tau_h(I) \) - heating time constant; \( \tau_c \) - cooling time constant. In expression (2), which describes heat outflow from the sensitive element of the thermal sensor, only the initial value of \( T_s(I) \) depends on the discharge current. To simplify calculations, the value of \( T_0 \) in (1) is taken to be zero. In this regard, choosing between kelvins and degrees Celsius for measuring temperature is an arbitrary matter.

Next, we analyze the experimental results aimed at studying the possibility of reducing the substrate heating time:

1) we present a complete experiment on heating at a current of 4 A as several shortened experiments, performed at shorter time intervals \([0, t_i]\) \((i = 1, 2, 3)\). The choice of the \( t_i \) values is arbitrary, therefore, we will focus on \( t_i = 250, 500 \) and 1000 s, assuming that three values are quite enough to establish the main regularities in subsequent calculations. If necessary, the number of reduced intervals can be increased in the future. Figure 2 shows the kinetic curves for these three intervals formed from the complete kinetic curve in Figure 1 measured over a time interval of 1400 s;

2) we define an analytical description (model) of all experiments approximating them using the exponent (1);

3) we perform extrapolation according to the model built on each interval to the region of the steady state of the process;

4) let us estimate the errors arising from extrapolation to the steady state region comparing the model dependences of the shortened and complete experiments. The estimate of the relative error \( \delta_T(t_i) \) in the prediction of the stationary temperature is defined in the form

\[ \delta_T(t_i) = \frac{|T_{s0}(t_i) - T_{s\infty}|}{T_{s\infty}}, \]

where \( t_i \) is the time of the shortened experiment; \( T_{s0}(t_i) \) and \( T_{s\infty} \) are the stationary temperatures obtained according to model (1) for the observation interval of time \( t_i \) and the full experiment, respectively. If the heating time constant is also estimated from the reduced observation interval, then the relative error of this procedure, by analogy with (3), is defined as

\[ \delta_{\tau}(t_i) = \frac{|\tau(t_i) - \tau_f|}{\tau_f}, \]

where \( \tau(t_i) \) and \( \tau_f \) are the time constants in model (1) for the observation interval of time \( t_i \) and the full experiment, respectively.
Table 1 presents the parameters of model (1) calculated by the least squares method for all observation intervals. Table 1 shows that as the duration of the observation decreases, the value of the predicted stationary temperature decreases. The heating time constant also changes.

| i  | $t_i$, s | $T_{\infty}$, $^\circ$C | $\tau_{th}$, s |
|----|----------|----------------|--------------|
| 1  | 250      | 148            | 323          |
| 2  | 500      | 162            | 361          |
| 3  | 1000     | 165            | 371          |
| full | 1400   | 168            | 405          |

These changes are shown by dots in figure 2, which are described by exponents with a confidence of 0.99:

$$T_{\infty}(t) \approx 167 - 27\exp\left(-\frac{t_i}{313}\right),$$  \hspace{1cm} (5)

$$\tau_{th}(t) \approx 276 + 62\exp\left(-\frac{t_i}{1876}\right),$$  \hspace{1cm} (6)

shown in figure 3 by solid lines.

Figure 3. Dependences on the observation time for the substrate stationary temperature (a) and the time constant (b). Points are the experiment; solid lines – approximation.

Figure 4. Influence of the duration of the observation interval on the relative error in predicting the stationary temperature (a) and the dependence of the duration of the observation interval on the relative error $\delta_{\tau}$ in predicting the stationary temperature (b). Points are the experiment; solid lines – approximation.
In figure 4, a, the dots show the experimental dependences plotted using expression (3). The approximation was carried out using function (5):

$$\delta T(t) \approx 0.163 \exp\left(\frac{-t}{313}\right).$$  \hfill (7)

Note that the time constants in expressions (5) and (7) are the same. Expression (7) can be used to select the observation time of the substrate heating kinetics for a given error. For this, it is more convenient to transform (7) into the function $t = f(\delta T)$, which has the form

$$t \approx 126 + 1665 \exp\left(\frac{-\delta T}{0.0267}\right)$$  \hfill (8)

and is shown in figure 4, b. Figure 4, b shows that a relative error of less than 0.05 is given by observation of substrate heating for 400 s and more. Considering the fact that the total observation interval in this work was about 1400 s, we may assume that using the proposed technique for a single experiment more than threefold time savings can be obtained.

4. Conclusions

The proposed technique for reducing the duration of the experiment is universal. For its application in each practical case, it is necessary to perform one long experiment, in which the process of heating the substrate reaches a steady state, and perform a series of proposed calculations.

References

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