Control of Transparent Conductive and Antireflection Coating Deposition Processes

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ABSTRACT

This paper considers an approach to control methods used in longtime reactive deposition processes on roll-to-roll vacuum machines.

Stability of gained coatings parameters was ensured by using double-loop control system, applying emission spectra, and both optical and electrical characteristics.

The paper also considers usage of theoretically calculated spectral characteristics (like visible light reflection and optical transmission) for optimization of the deposition processes of multilayer anti-reflection coatings. The method and hardware were tested on the laboratory vacuum machine and utilized in industrial roll-to-roll vacuum machine for deposition of four-layer antireflection coating.

INTRODUCTION

At deposition of anti-reflection coatings, increased requirements for keeping the set thickness of layers of a coating are shown, and for obtaining of productivity process dynamic control in transition mode of reactive sputtering is necessary. Some schemes of dynamic control of reactive process of magnetron deposition of oxide coatings in a transition mode are known.

We consider the widespread method of control with use of optical emission spectroscopy (OES).

Intensity of radiation of a spectral line of metal I_{met} is measured in the given method in process, and it is kept at the set level I_{set} by the control of oxygen flow Q_{O2} in the chamber at the stabilized sputtering power (current) [1, 2].

Owing to wearing of a target at the same sputtering power, the quantity of the metal sputtered from a target is reduced. In OES method for keeping of same intensity I_{set} the control system will decrease oxygen flow, which will result in enrichment of a coating by metal.

OES method allows us to carry out process in a transition mode, but it does not provide stability of properties of a coating at long operation of a target, and only problems of work in a transitive mode are solved. It was suggested to enter the second contour of control of process which, using measurement of physical properties of a coating, allowed to correct value of intensity [3]. For example, at coating deposition on roll materials, measurement of optical transmission and reflection can be considered as a secondary signal. If transmission or reflection starts to change, it is necessary to make correction of an initial contour to keep the coating's properties at a desirable level.

Despite the simplicity of the approach, it is not always possible to achieve. It is frequently difficult to measure properties of a coating during process, coating stoichiometry, except for that measurement of coating properties, as a rule, lag behind on time the moment of coating deposition, except that in process sputtering, the rate of a target varies during long duration.

EXTENDED OES METHOD FOR REACTIVE PROCESSES CONTROL

Coating deposition by reactive magnetron sputtering on a substrate results in two flows: metal flow Q_{met} and reactive gas flow Q^*_{gas}. As a result of these flows interaction combination (in our case, oxide or nitride) is formed.

Dynamic control of coating deposition process is reduced to two processes:

1. Setting of balance between a reactive gas flow Q^*_{gas} and sputtered metal flow on substrate Q_{met}. Criterion for balance setting is coating physical properties.
2. Keeping of balance between reactive gas flow Q^*_{gas} and sputtered metal flow Q_{met} on a substrate in time.

$$\frac{Q^*_{gas}}{Q_{met}} = const$$

Equation 1

Flows of metal Q_{met} and reactive gas Q^*_{gas} on a substrate cannot be measured directly in process, and usually are measured indirectly, for example, by intensity of radiation of metal spectral line I_0 which is proportional to metal flow in reactive process, and partial pressure of reactive gas p_{gas} which is proportional to reactive gas flow on a substrate is measured. Therefore the condition of balance keeping (Equation 1) can be presented so

$$\frac{p_{gas}}{I_o} = const.$$

Equation 2

Initial characteristics of reactive process are magnetron power P and reactive gas flow Q_{gas} in the chamber (do not confuse with a flow of reactive gas on substrate Q^*_{gas}), which in combination determines intensity of radiation I_0 and partial pressure of reactive gas p_{gas}.

The analysis of relationship (Equation 2) shows that change of intensity of radiation of metal (that is equivalent to change of metal flow) will result in change of partial pressure of reactive gas. The same will take place at change of partial pressure of reactive gas. The ratio will remain the same, but it can cause change of physical properties of a coating, at the very least, thickness of a coating. Therefore, it is necessary not only to keep balance between flow of reactive gas Q^*_{gas} and flow of sputtered metal Q_{met} on a substrate, but also one of values Q^*_{gas} or Q_{met}.

In Figure 1, known dependence of reactive gas flow on intensity in controlled reactive process is shown. In controlled process at the given sputtering power, two parameters (I_0, Q_{gas}) determine properties of coating. At change of sputtering power point I_{met} describing intensity of radiation at sputtering in metallic mode will move, a curve will be deformed, and as a result, we will receive a new pair on intensity/flow for performance of the requirement on properties of a coating.

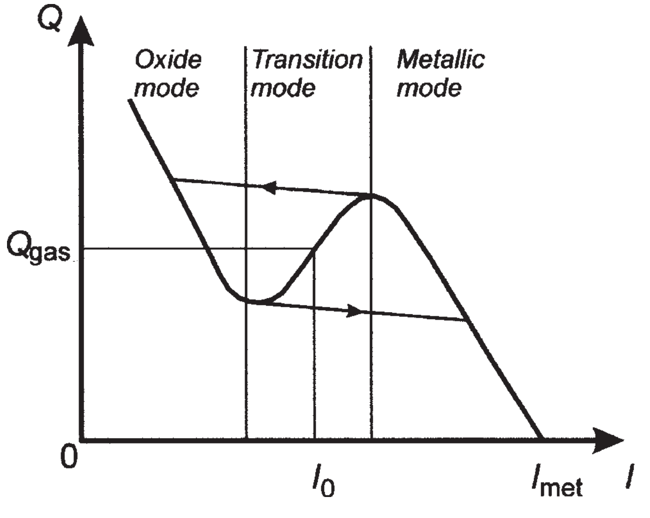

Figure 1: Dependence of reactive gas flow on intensity in controlled reactive process.

In a metal sputtering mode, intensity of radiation is proportional to sputtering power (line OA in Figure 2). In reactive process at the given sputtering power P_1 process control system with the help of OES uniquely determines necessary flow of gas Q_{gas} for maintenance of the set intensity of radiation of metal I_{set} (point B). Only one pair of values of intensity and a flow of reactive gas will allow it to receive a coating with the set properties at the given sputtering power. Thus, the pair rating values of intensity and a corresponding flow of reactive gas in the chamber for reception of a coating with the set properties is established at the given sputtering power. Value of intensity of radiation and a flow of reactive gas for reception of the set properties of a covering should be determined in preliminary experiments.

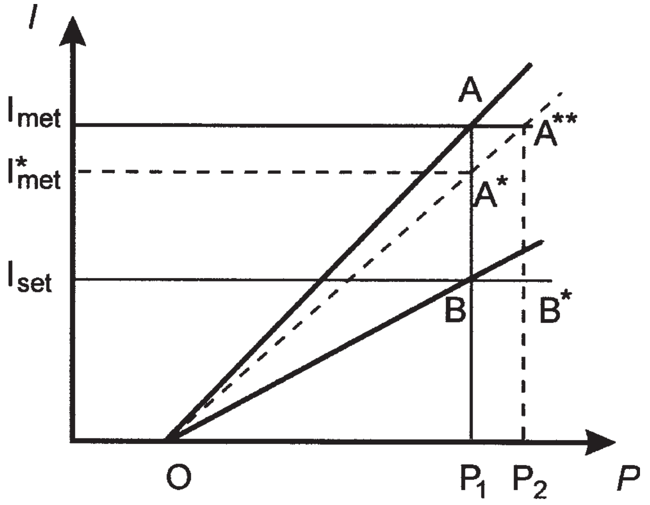

Figure 2: The scheme of extended OES method for control of reactive process.

If sputtering rate of metal is reduced in process (line OA** in Figure 2), point A passes in A^*, it means that intensity of radiation decreases up to I^*_{met} in metal sputtering mode. Thus, the control system reduces a flow of reactive gas Q_{gas} for maintenance of the same intensity of radiation I_{set}, and the balance between a flow of reactive gas and a flow of the sputtered metal on a substrate will be disturbed, resulting in a coating surplus of metal. Generally, for practice, the appearance of value I_{met} is not so important, but it is necessary to know a pair of parameters of process (I_{set}, Q_{gas}) which provides properties of a coating.

By keeping all parameters of the process constant sputtering rate of a target in time is monotonously reduced. It is the consequence of such processes that there is a change of a magnetic field owing wearing of a target, processes of oxidation on a surface of a target, growth nigel, etc. Thus, monotonous reduction of reactive gas flow in reactive process with other things being constant will show that sputtering rate of metal has decreased. In long-time processes for keeping balance between a flow of reactive gas Q^*_{gas} and a flow of the sputtered metal on a substrate, over time it is necessary to keep metal sputtering rate constant.

At decrease of sputtering rate of a target, it is necessary to increase sputtering power to P_2 so that at the given intensity of radiation I_{set} it receives former value of a reactive gas flow, i.e. in addition to control of a gas flow, control of sputtering power is necessary. In metal sputtering mode, we will receive another dependence of intensity on power, straight line OA^{**}; but a kind of curve $Q = f(I)$ (Figure 1), will not change. Accuracy of keeping reactive gas flow in the chamber can be set proceeding from requirements to properties of coatings; the maximum deviation of a reactive gas flow from nominal should be such that properties of a covering have not left for allowable limits.

The additional feedback on power allows us to take into account the reduction of sputtering rate and to exclude its influence on properties of a coating.

The suggested way eliminates the expensive equipment for coating properties measurement operatively control process of coating deposition during a long period of time.

EXPERIMENT

Application of Method at Anti-reflection Coating Deposition

The suggested method has well proved to be at reactive processes deposition of complex coatings (oxides, nitrides, etc.) for creation of single layer and multilayer structures, including anti-reflection coatings. In these cases, such target characteristics of deposited coatings as light transmission, reflection, and sheet resistance are usually supervised.

Figure 3 shows experimental data received at reactive sputtering of titanium in an atmosphere of argon and oxygen with use of the above described control process.

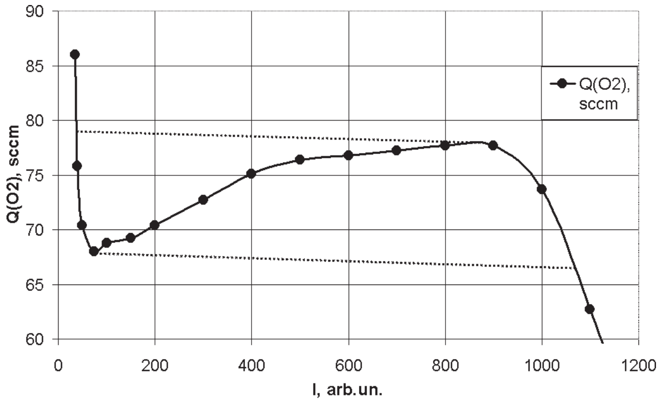

Figure 3: An experimental curve of interdependence of intensity of emission line and oxygen flow in controlled reactive process of titanium sputtering.

For work of control system in transition mode, the speed of measuring system is rather essential. This speed is determined substantially by speed of OES data updating. In our case, good results have been received at the measuring speed of OES data more than three measurements per second.

The suggested scheme of process control has allowed us to work stably in a transition mode on a roll-to-roll machine for oxide coating deposition on PET film during a long period of time.

For independent quality monitoring of a coating deposited at the given control process, measurements of optical characteristics of deposited materials were used. The received spectral characteristics of reflection coefficient and light reflection coefficient were compared with preliminary calculations. Application of such approach for the monitoring of characteristics of a coating has allowed us to receive high accuracy multilayered structures with the preset optical properties.

Application of Method at Titanium Nitride Deposition on a Metal Foil

More difficult are processes in which it is necessary to keep such parameters as structure stoichiometry. The situation is complicated because of the use of metal substrates which completely eliminate an opportunity of resistance and light transmission monitoring. Such process is deposition of titanium nitride on a metal foil.

Figure 4 shows time diagrams of titanium emission line intensity, magnetron power and reactive gas (nitrogen) flow at deposition of titanium nitride stoichiometric coating.

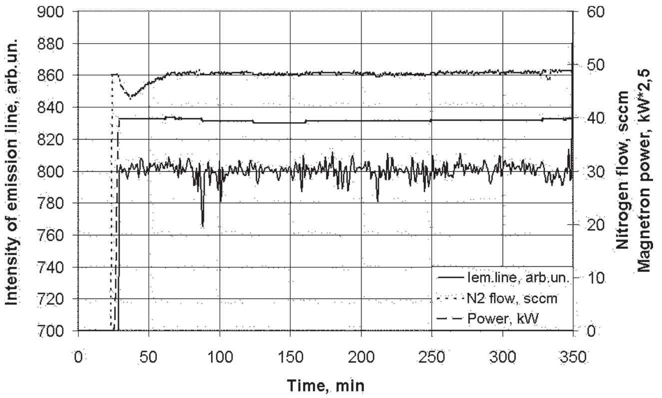

Figure 4: Time diagram of titanium nitride coating deposition process.

From the figure, it is visible that there is a keeping of constant value of emission line intensity by control of a reactive gas flow. If change of a gas flow is higher than preset value, there is a correction of power for restoration of a preset value of a gas flow with keeping of a preset value of intensity of emission line.

During deposition of titanium nitride on aluminum foil for maintenance of pair parameters (intensity/flow of nitrogen in the chamber) during seven hours, power has been increased on 3.3%, the nitrogen flow was kept with accuracy ± 2%. It has allowed receiving during the stoichiometric coating of titanium nitride.

CONCLUSION

Addition of OES method by a feedback power flow of reactive gas allows for removal of influence of sputtering rate change at change of target sputtering conditions. In suggested methods, control is made on two parameters of process: to a reactive gas flow and sputtering power at preset emission line intensity. Two control loops in extended OES method provide:

- Stable course of process of reactive deposition of a coating in a transition mode by keeping of intensity of radiation of a spectral line of metal by control of reactive gas flow in the chamber.
- Stability of properties of a coating in time by control of sputtering power for maintenance of reactive gas flow at the set intensity of radiation of a spectral line of metal.

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