

Control of Reactive Deposition Process by Stabilization of the Power Supply Work

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ABSTRACT

Oxide coatings deposition in reactive process from metallic targets has particular importance thanks to cheapness of metallic target in comparison with conductive ceramic targets. Silicon oxide can be deposited only from a metallic target or from dielectric target by RF-sputtering, but deposition speed in case of RF-sputtering is low. Reactive sputtering of metallic targets is characterized by voltage hysteresis and has a characteristic appearance depending on type of metal and value of secondary electronic emission factor for metal and oxide. From voltage-current (VC) characteristics follows, that for silicon oxide (SiO_2) deposition it is necessary to work in power supply constant voltage mode and for titanium oxide (TiO_2) deposition - at constant current mode and at constant oxygen flow. For process stabilization it is necessary to stabilize the second electric parameter by changing oxygen flow in the chamber. We develop an algorithm and a control system of process for this case. As a result in process there is only one variable value of process which can be controlled, this is oxygen flow, at a constant pumping speed, leakage, etc. In this case process will be stabilized. Changes of outgasing flow during process as a result of heating of design elements of a chamber will be considered also. Stabilization of parameters of the power supply creates favorable conditions for reproducibility of properties of a coating. In the work results of process stabilization are presented at SiO_2 and TiO_2 coating deposition on polymeric PET film.

INTRODUCTION

Reactive process of oxide magnetron deposition from a metal target is of interest caused by cheaper material of a target and target manufacturing techniques. However, such deposition process is characterized by a hysteresis. As a result it is required to control process in a transition mode for obtaining high deposition rates and high quality of a coating.

A number of control methods are developed for work in process transition mode: PEM, a partial pressure method, an impedance method. Those methods demand fast control of an oxygen flow in the chamber because of transient (10–30 sec) transition of a mode metal - oxide and on the contrary [1]. Development of process control methods has not lost its urgency.

In reactive process change of oxygen flow changes target conditions that are expressed by its partial coating by oxide. It also involves change of secondary electronic emission from a target during sputtering process. Depending on the nature of metal, the ratio of secondary electronic emission factors from metal γ_M and from same metal oxide γ_O differs: $\gamma_M < \gamma_O$ typical, for example, for Si, $\gamma_M > \gamma_O$ it is typical, for example, for Ti [2]. In [2] it is shown, that VAC (volt-ampere characteristic) for various metals have different character, depending on a ratio of secondary electronic emission factors γ_M and γ_O . The important conclusion is that if $\gamma_M < \gamma_O$ it is necessary to work in constant voltage mode of magnetron power supply, but if $\gamma_M > \gamma_O$ then it is necessary to work in constant current mode of magnetron power supply.

We showed possibility for further development of the control system of reactive process for the purpose of stabilization of process and coating properties.

EXPERIMENTAL

Studying of control of oxide coating reactive deposition processes for silicon and titanium were carried out on roll-to-roll vacuum machine UV80. It is equipped with planar magnetron with a target size 120 x 790 mm. The coating was deposited on PET film. For sputtering pulsed DC power supply Pinnacle Plus, 10 kW (325 - 800 Vdc) has been used. Sputtering was carried out in a constant voltage mode or in constant current mode on frequency 50 kHz. The algorithm and the control unit are developed for process control, allowing to operate with gas flow by Mass-Flo® Controller (MKS Type 1179A) for controllable input of oxygen flow in to the chamber for the stabilization of electric parameters of the process.

Test experiments were carried out at silicon reactive sputtering by dual magnetron with planar targets 120 x 790 mm and power supply Huettinger TIG 30/100 P, 30 kW MF and on rotary magnetron with length of a target 2400 mm and power supply AE® Crystal® 60 Power Supply.

THE FURTHER DEVELOPMENT OF A CONTROL SYSTEM FOR SILICON OXIDE DEPOSITION PROCESS

Except controllable oxygen flow in the chamber, there are other sources of uncontrollable change of partial pressure in the chamber: oxygen desorption from inside chamber devices (especially after magnetron switch on and heating of devices inside chamber); change of pumping speed; temperature changes indoors, that causes change of temperature of the chamber, i.e. uncontrollable processes which can influence processes of adsorption and desorption. As a rule, in order to decrease uncontrollable oxygen flow in the chamber long preliminary pumping and magnetron annealing are done. Uncontrollable oxygen flow in process can be accounted, if electric parameters of the process make related with an oxygen flow.

On Figure 1 well known voltage hysteresis during silicon oxide reactive sputtering and a control mode by voltage with constant power is shown. Family of VAC at constant oxygen flows, is shown on Figure 2. In traditional scheme control of oxygen flow for voltage stabilization at constant discharge power allows to work in a transition mode. However it requires fast control because the transitive mode is unstable [1, 4].

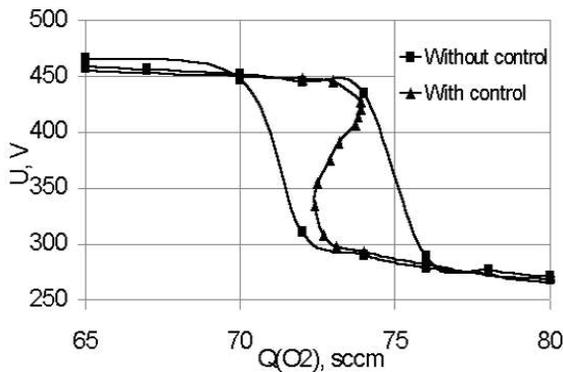


Figure 1: Voltage hysteresis at silicon reactive sputtering in UV80.

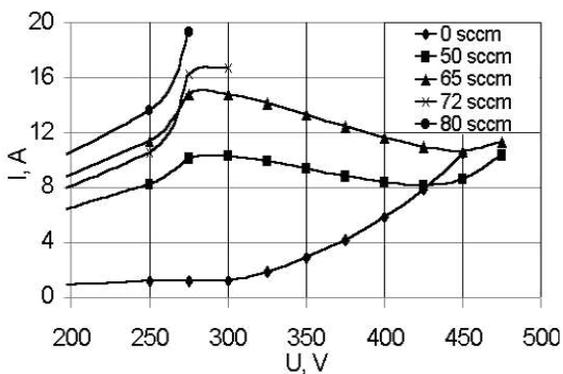


Figure 2: Family of VAC for Si reactive sputtering in UV80.

Considering voltage hysteresis at silicon oxide sputtering and VAC, and considering, that for silicon $\gamma_M < \gamma_O$, [2, 3] for process of SiO_2 deposition it is necessary that magnetron works in constant voltage mode of power supply. In this case discharge current monotonously changes with change of oxygen flow and has no hysteresis, Figure 3. At elimination of a hysteresis there is no necessity for high-speed feedback and fast control of an oxygen flow that reduces requirements of speed of a control system.

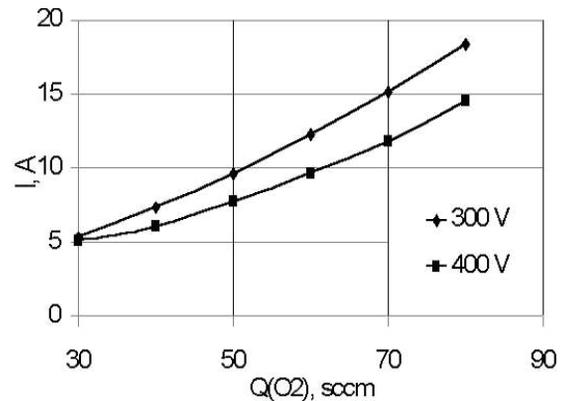


Figure 3: Magnetron discharge current vs oxygen flow in the chamber at constant voltage.

For discharge current stabilization it is necessary to control oxygen flow in the chamber. The offered control scheme for deposition process of SiO_2 looks so:

- the setted discharge voltage, $U = \text{const.}$, stabilizes the power supply,
- stabilization of a discharge current, $I = \text{const.}$, it is reached by an operational control of oxygen flow $Q(\text{O}_2)$.

Voltage U gets out starting with necessity to work in a transition mode and is specified starting with demanded properties of a coating. In these conditions all electrical discharge characteristics will be stabilized. The considered scheme of control supposes other possibility. Instead of discharge current stabilization by oxygen flow it is possible to stabilize discharge power, which is proportional to a current at constant voltage.

In control mode with constant discharge current when discharge current decreases the control system should increase oxygen flow in the chamber and on the contrary, Figure 3. As consequence, if the uncontrollable oxygen flow increases in the chamber the control system will react by reduction of a controllable oxygen flow for stabilization of a discharge current, Figure 4.

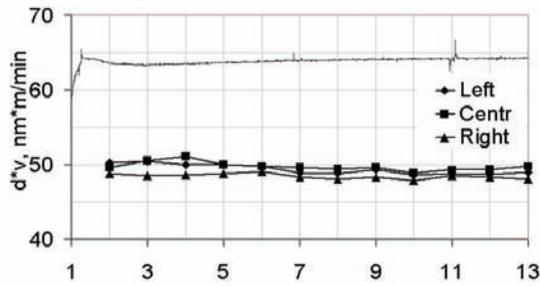


Figure 4: Change of oxygen flow in the chamber and dynamic deposition rate for SiO_2 in the deposition process beginning.

Thus, electric parameters of process are stabilized indefinitely long; process has one variable (adjustable) value - an oxygen flow in the chamber. Despite change of oxygen flow in the chamber, coating dynamic deposition rate does not vary. As a result, the control system considers uncontrollable oxygen in the chamber that allows to receive already in the process beginning reproduced results.

For the purpose of magnetron work stabilization the algorithm is developed and the equipment for automatic control of reactive gas flow in the chamber by means of Mass Flow Controllers (MFC) is created. In this case equipment for oxygen flow control is inexpensive, as gauges is used regular measuring equipment of the power supply and gas MFC. At a correct choice of an operating mode of the power supply there is no necessity for fast control as in this case the hysteresis is absent.

Results for process beginning and for long process are presented on Figure 5. In long process the oxygen flow in the chamber changes not only due to processes in the chamber, but as due to change of conditions in the environment.

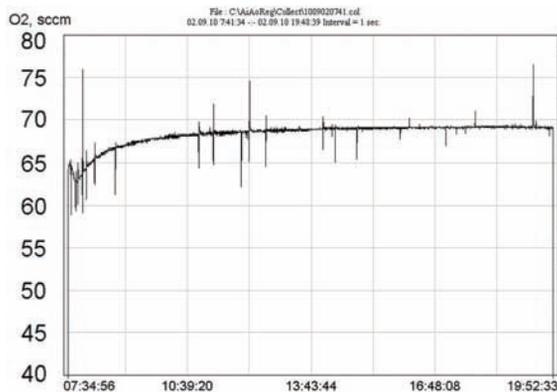


Figure 5: Change of oxygen flow in long process.

Similar results have been received on other types of magnetrons and power supplies. In reactive process with target Si (Al 10 %) For silicon oxide deposition received dynamic deposition rate was close to $80 \text{ nm} \times \text{m} / \text{minutes}$. Restriction of deposition rate is connected with possibilities of heat removal from a target, therefore it was necessary to limit sputtering power.

SOME RESULTS ON TITANIUM OXIDE DEPOSITION

The deposition mode for titanium oxide has differences in comparison with silicon oxide because factor of secondary electronic emission from the titanium is higher than from titanium oxide, therefore orientation of a hysteresis loop is a mirror in relation to silicon, and VAC is symmetric for Ti to similar characteristics for Si, Figures 1, 2, 6, and 7. Hence, the scheme of reactive process control for titanium sputtering should differ from the scheme for silicon.

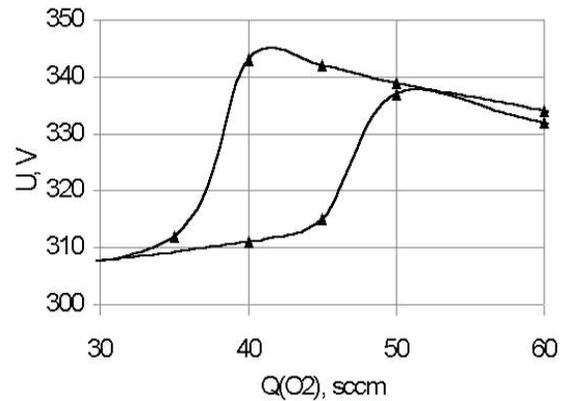


Figure 6: A voltage hysteresis at reactive sputtering of the titanium in UV80.

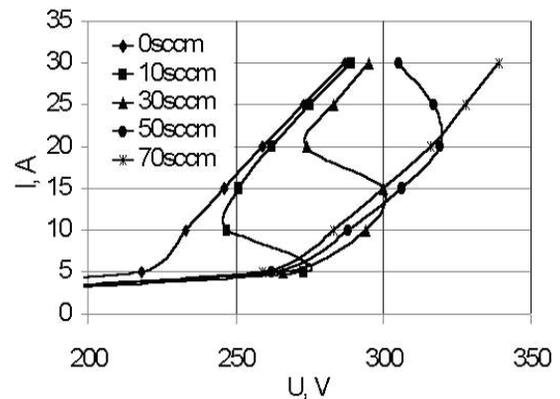


Figure 7: Family of VAC for Ti reactive sputtering in UV80.

From the aforesaid follows, that for titanium the scheme of process control should be such:

- the set discharge current, $I = \text{const.}$, stabilizes the power supply,
- stabilization of discharge voltage, $U = \text{const.}$, it is reached by control of oxygen flow $Q(O_2)$.

Discharge current I gets chosen depending of the necessity to work in a transition mode and is specified starting with demanded properties of a coating.

For the reactive deposition of titanium oxide little changes of voltage is typical in transition between modes metal/oxide. Nevertheless, the specified scheme of the process control allows to receive reproduced properties of titanium oxide coating at short processes and in long process, Figure 8.

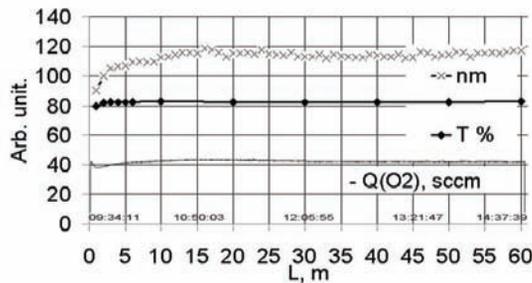


Figure 8: Changes of oxygen flow in the chamber and properties of TiO_2 coating in process.

CONCLUSIONS

The method of stabilization of power supply work with one variable (an oxygen flow) is universal in sense of different types of magnetrons and types of power supplies.

Process control mode, using a constant voltage mode of a power supply for Si with stabilization of a discharge current (power) and a constant current mode for Ti with stabilization of discharge voltage, provides long time stability at least for coating thickness in the process.

REFERENCES

1. D.C.Carter, W.D.Sproul, D.J.Christie. Effective control for reactive sputtering processes. VT&C 2006, April, 60-67.
2. E.Berlin, S.Dvinin, L.Sejdman, Vacuum technology and the equipment for deposition and etching of thin films. Moscow, 2007.
3. H. Ohsaki, Y. Tachibana, J. Shimizu, T. Oyama. High-rate deposition of SiO_2 by modulated DC reactive sputtering in the transition mode without a feedback system. Thin solid films 281-282(1996) 213-217.
4. L. Lou, G. MacDonough, H. Walde, D. Carter, G. Roche, R. Scholl. Closed loop controlled reactive dual magnetron sputtering. Advanced Energy Industries, Inc.