OXYGEN ACTIVATION EFFECT ON REACTIVE MAGNETRON SYNTHESIS OF ALUMINA COATINGS

J. Walkowicz [1], A.V. Zykov[2], S.V. Dudin [2], S.D. Yakovin [2]

[1]Institute for Sustainable Technologies, Pulaskiego Str. 6/10, 26-600, Radom, Poland;
[2]V.N. Karazin Kharkov National University, Department of Physical Technologies, Kurchatov av. 31, 61108 Kharkov, Ukraine

The investigation results of the DC magnetron sputtering system for synthesis of high-quality oxide coatings are presented. In the system oxygen, activated by an independent Inductively-Coupled Plasma (ICP) source, is introduced into an aluminum sputtering chamber as an alternative to conventional reactive sputtering techniques employing the injection of ground state molecular O_2. Characteristics of deposition behavior are investigated with and without the activation of the reactive species.
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1. INTRODUCTION

Aluminum oxide thin films are widely used in many mechanical, optical and microelectronic applications because of their excellent properties, in terms of chemical inertness, mechanical strength and hardness, transparency, high abrasive and corrosion resistance, as well as insulating and optical properties. At present thin alumina coatings (less then or about 1 micron) are used in production of solar sells, elements for integral optics, diffusion barrier applications and in microelectronics [1].

However, despite the obvious virtues of alumina coatings, process of introduction of such coatings to the industry is in an initial stage, and scope of their use is far from the same of TiN coatings. Direct current planar magnetrons are widely used for deposition of metal coatings [2]. At the same time ion-plasma synthesis of dielectric coatings, in particular the oxides such as TiO_2, Al_2O_3, Ta_2O_5 etc. by DC magnetrons has got some serious difficulties for the simple reason that the films being processed do not conduct current. This leads to the target passivation, intensive arcing, and the instability of discharge (phenomenon of "disappearing anode"), that essentially constricts the "window" of technological parameters and reduces the coatings quality. One of traditional solutions of these problems is the use of high-frequency or pulsed power source. However in this case the deposition rate falls and the equipment cost is essentially increased.

Reactive sputtering from a metallic target is the preferred choice for high rate deposition, but here the controllability issues of target and anode oxidation must be addressed. Because of these issues, the search continues for a solution providing high throughput, reproducible reactive sputtering results for materials like aluminum oxide.

In this paper the results of development of the DC magnetron sputtering system for synthesis of high-quality oxide coatings are presented. The basic idea of the system consists in separation of 2 processes: metal target sputtering by DC magnetron discharge in inert gas and activation and transport of reactive gas by additional plasma source based on RF inductive discharge [3].

2. EXPERIMENTAL SETUP

Film depositions were performed in a Balzers BA-510A high vacuum pumping system with the base pressure about 10^{-5} mbar. A schematic layout of the magnetron and ICP source in the sputtering chamber is shown in Fig. 1. A pure aluminum target of 170 mm diameter mounted on magnetron 1 served as a sputtered aluminum source. Power to the sputter cathode was applied using 10 kW dc power supply 2, produced by ITeE, operated in current or voltage regulation mode. Current regulation was chosen in these experiments simply as a monitor of process stability. By allowing the cathode voltage to drift, the level of poisoning could easily be monitored by tracking the target voltage [4,5].

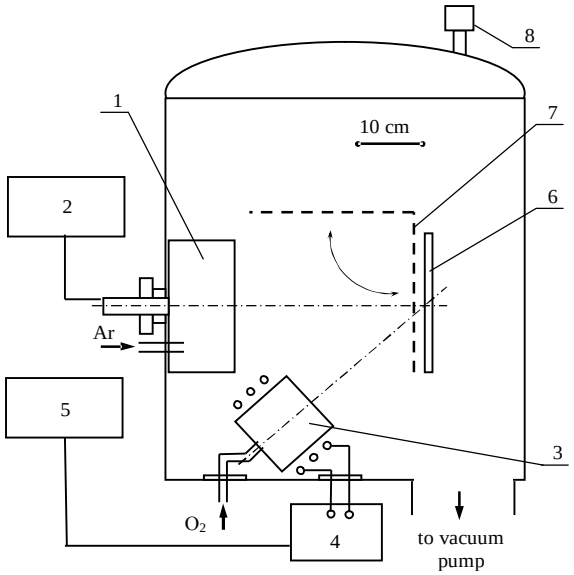

Fig.1. A schematic diagram of the alumina deposition system: 1 - magnetron, 2 - DC power supply, 3 - ICP source, 4 - RF matchbox, 5 - RF power supply, 6 - substrate holder, 7 - substrate shield, 8 - vacuum gauge

Argon used as the sputtering gas in all deposition experiments was fed to the chamber independently of the reactive oxygen – through the dedicated manifold – directly on the magnetron target. Flows for both argon and oxygen were regulated using BETA ERG mass flow controllers operated by two-channel process control unit. Pressure monitoring in the sputter chamber was accomplished using Balzers PKR-250 Pirani/Cold Cathode gauge 8.

Oxygen for the reactive sputtering was delivered through the ICP source 3 for all deposition processes. A ceramic tube 100 mm in diameter and 120 mm in length was used as ICP source chamber. The source was located 10 cm closer to samples than the magnetron such that the ICP source did not block the path of sputtered aluminum (see Fig. 1). The substrates were mounted at shielded substrate holder 6 that allows to deposit up to 12 samples per pumping cycle.

3. TECHNOLOGY OF ALUMINA SYNTHESIS USING THE ICP ENHANCED REACTIVE MAGNETRON SPUTTERING SYSTEM

The key novelty of the present system comparing to known designs is the operation pressure range $(0.7\text{-}3.5)\cdot10^{-3}$ mbar where motion of particles may be treated as free fall. It allows increasing the distance magnetron – substrate holder up to 30 - 40 cm, significant increase in the deposition area with acceptable deposition rate.

In Fig. 2 magnetron voltage-current characteristics are presented at various oxygen flows. Measurements were done in the following sequence. At high discharge current (8 A) oxygen feeding was carried out up to the necessary flow rate, and then the magnetron current was reduced down to the passivated mode transition. The dependence of minimum DC power of the magnetron operation in metallic mode has been in such a way measured versus oxygen flow rate (Fig. 3). Apparently from the figure it is practically linear with the slope of about 10-12 sccm/kW.

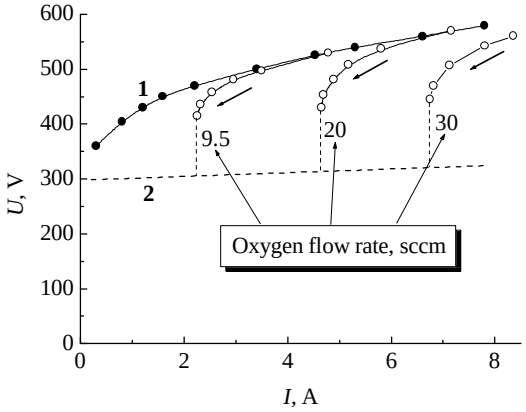

Fig. 2. Current-voltage characteristics of the magnetron:1 – Ar at 0.8 mTorr, 2 – O_2 at 0.8 mTorr

To achieve a stable environment for aluminum oxide deposition and to provide control margin to the reactive deposition process, the effective oxygen partial pressure at the substrate was modified using the preactivation of the reactive gas using the independent RF ICP source. The idea of this technique is to deliver gas of improved reactivity to the substrate, further-increasing the effective oxygen partial pressure in the deposition zone and thereby increasing the operating margin by maintaining the target in a more stable range on the hysteresis curve. This idea was successfully verified in our experiments.

The standard sequence of the deposition process stages used in the present experiments is described below, the choice of technological regime for stoichiometric

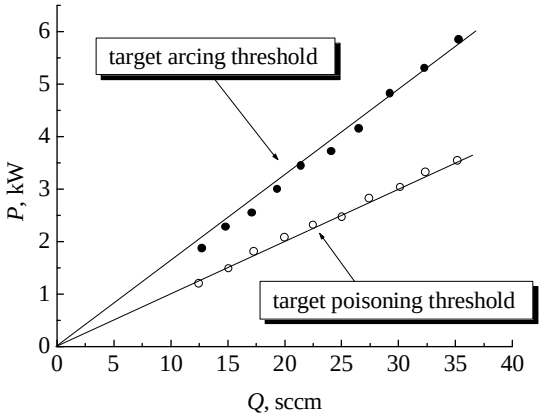

Fig. 3. Dependence of minimum magnetron DC power P for metallic mode operation on oxygen flow rate Q

alumina deposition was done with the help of diagrams given in Figs. 2, 3.

Question of principle at deposition of stoichiometric Al_2O_3 films when using the directed source of activated oxygen is the relative positioning of the magnetron, the plasma source and the substrate. It has been established, that alongside with main technological process parameters (magnetron and RF discharge power, oxygen flow rate, argon pressure), the geometrical parameters (distances and angles of the magnetron and the plasma source in relation to the substrate holder) determine 3-dimensional Al_2O_3 stoichiometrical region in the chamber space.

In the Fig. 4 a typical 2D stoichiometrical diagram of the films synthesized in the present system is shown in the plane passing trough the axis of the magnetron and ICP source.

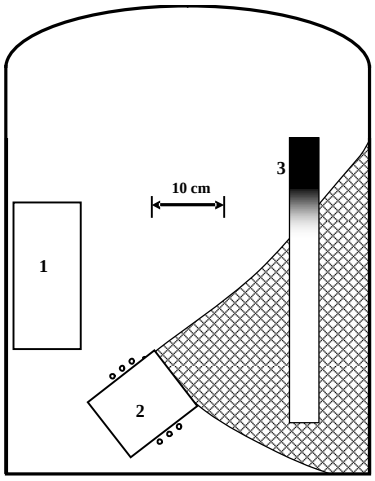

Fig.4. 1 - magnetron, 2 - ICP source, 3 - substrate holder with film transparency distribution shown. Stoichiometrical region is hatched

3.1. TECHNOLOGICAL BASELINE

1. The substrates (glass, clear an TiN coated high-speed steel SW7M) cleaned in ultrasonic bath with standard technology, were mounted on a shielded substrate holder allowing multiple sample deposition per pumping cycle. Processing started after pumping to the base pressure 10^{-5} mbar or less.

2. The substrates were cleaned and activated in Ar ICP discharge at pressure (1 - 3)·10^{-3} mbar and RF power 500 W during 5-15 min. DC bias (0, -100, -500, -1000 V) was applied to the substrates, and substrate current was measured.
3. After the cleaning stage the substrate bias was switched off, the ICP discharge remained on, shield was closed, and the magnetron was ignited at constant argon pressure. After the oxidized magnetron target surface recovery to metallic mode and stabilization of the magnetron discharge parameters within 3-5 minutes, oxygen was filled through ICP source. Oxygen flow rate was set according to the diagram in Fig. 3. After that fine tuning of the ICP source matchbox was performed if needed.
4. The shield was opened, and deposition time counting was started. During the deposition process the RF matchbox was tuned if reflected power exceed 10% of the forward power. The magnetron power supply operated in the current regulation mode, so no adjustment was necessary.
5. After the deposition time termination the shield was closed, oxygen flow was shut down, the magnetron power supply and the RF generator were switched off, after that delivery of argon was stopped and the chamber was opened.
Process of aluminum film deposition was performed in the same sequence, but without oxygen feeding.

4. CONCLUSIONS

To produce a fully oxidized aluminum oxide film and reliably avoid target poisoning in a dc magnetron reactive sputtering process one of two approaches must be taken: 1) to employ sophisticated feedback and control loops in the gas delivery system to maintain the oxygen partial pressure at a precisely specified level or 2) to use directed source of activated oxygen such that the oxygen partial pressure at the target is measurably less than that required to cause poisoning but the partial pressure and reactivity at the substrate is adequate to produce the films of the desired properties. The technique, explored in this study, was shown effective in producing high quality aluminum oxide films in a dc reactive sputtering environment without the issue of target poisoning. Using these enhancements, films deposited in the flat portion of the target voltage hysteresis curve displayed properties comparable, and in some cases, superior to those deposited near the knee of the curve without the aid of the mentioned enhancements.

The complex application of these solutions in the sputtering system has allowed to expand the range of stable system operation: working pressure – (2-10)·10^{-4}Torr, magnetron discharge power – (1–8) kW, power of chemically active particles source – up to 1 kW, coating deposition rate (for example Al_2O_3) – up to 8 microns/hour, and to improve essentially the coatings quality.

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ВЛИЯНИЕ АКТИВАЦИИ КИСЛОРОДА НА ПРОЦЕСС РЕАКТИВНОГО МАГНЕТРОННОГО СИНТЕЗА ПОКРЫТИЙ ОКСИДА АЛЮМИНИЯ

Я. Валькович, А.В. Зыков, С.В. Дудин, С.Д. Яковин

Представлены результаты исследований магнетронной распылительной системы, используемой для синтеза высококачественных оксидных покрытий. В рабочую камеру кислород напускается уже предварительно активированный при помощи источника на базе высокочастотного индукционного разряда, что является альтернативой обычному реактивному магнетронному нанесению, когда в рабочую камеру напускается молекулярный кислород O_2. Исследованы характеристики процесса нанесения, как с предварительной активацией реактивного газа, так и без.

ВПЛИВ АКТИВАЦІЇ КИСНЮ НА ПРОЦЕС РЕАКТИВНОГО МАГНЕТРОННОГО СИНТЕЗУ ПОКРИТТІВ З ОКИСУ АЛЮМІНІЮ

Я. Валькович, О.В. Зиков, С.В. Дудін, С.Д. Яковін

Представлено результати досліджень магнетронної розпилювальної системи, що застосовується для синтезу високоякісних окисних покриттів. В робочу камеру кисень напускається вже попередньо активованим за допомогою високочастотного індукційного джерела, що є альтернативою звичайному реактивному магнетронному нанесенню, коли до робочої камери подається молекулярний кисень O_2. Досліджено характеристики процесу нанесення, як з попередньою активацією реактивного газу, так і без неї.