Comparison of Different Concepts for the Stabilization of Reactive Sputtering Processes

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ABSTRACT

For the efficiency of the reactive magnetron sputtering process high deposition rates as well as precise control of stoichiometry are of fundamental importance. However, in most cases it turns out to be a demanding task to fulfill both requirements due to the characteristic instability of the reactive process. The present work compares the performance of different concepts for the stabilization of reactive sputtering processes in the case of different oxides (TiO_2 , ZnO:Al and Nb_2O_5). On the one hand, the well known techniques for process control, e.g. optical emission spectroscopy at fixed sputtering current, λ-probe at fixed oxygen flow, optical emission spectroscopy at fixed current and impedance control are considered. In addition we present recent approaches as 2-emission-line-OES operated at variable sputtering current and nonclassical impedance control. On the other hand, in contrast to the process control techniques, the second approach for process stabilization focuses on the modification of the sputter arrangement for an appropriate separation of the sputter target and the chamber wall / substrate region in the coating plant. The performance of the different concepts will be discussed and supported by simulations. In addition we introduce the concept of the specific deposition rate.

INTRODUCTION

High deposition rates are of essential importance in reactive magnetron sputtering on architectural glass. On the other hand stoichiometric films are required for the application. To meet both the requirements for high deposition rates and highly transparent films, the deposition process has to be stabilized in a specific window between the stable deposition modes at low and high oxygen partial pressure, called metallic mode and oxide mode, respectively.

The metallic mode is characterized by a clean (metallic) target, understoichiometric films and high deposition rates. The oxide mode, on the other hand, is characterized by highly transparent films, but poisoned target and therefore low deposition rates. However, the most striking problem of reactive magnetron sputtering in the transition mode is the abrupt change from the metallic mode into the oxide mode accompanied by a sharp increase of reactive gas partial pressure as well as a decrease of deposition rate. In addition, it is often difficult to hit the restricted process window for optimum layer quality, e.g. for transparent conductive oxides.

The reactive sputter process has widely been investigated in the past [1,2], and even the dynamic behavior [3–6] as well as the simulation of process control [7] has been considered. The goal of this work is to give a survey of different concepts for process stabilization.

EXPERIMENTAL SETUP/BASIS

The experiments and the simulation in this work refer to i) a Balzers/Pfeiffer PLS580 boxcoater equipped with two 500 mm PK500 MF-magnetron sputter cathodes [8,9] and ii) a sputter compartment of an inline coating plant equipped with two 750 mm PK700 MF-magnetron sputter cathodes [10].

An Advanced Energy PE10k 10 kW MF power supply was used. The oxygen and argon gas flow was controlled with MKS mass flow controllers. To monitor the oxygen partial pressure a λ-sensor was used. It was optimized with respect to the response time, yielding values of clearly smaller than 1 s. In addition the data acquisition enables us to acquire optical emission spectra as well as electric discharge parameters (the latter quasi DC and time dependent). These process parameters were fed into a PC based control system. Using a software controller, the sputtering process can be stabilized either by using the sputtering power at a fixed reactive gas flow or by using the reactive gas flow at a fixed sputtering power.

PROCESS STABILIZATION BY MEANS OF STATIC MODIFICATIONS

Increase of pumping speed

In principle, the "overpumping" of the hysteresis is always possible by increasing the pumping speed. From the Berg-Larsson model the required pumping speed can be calculated. The key is the so called characteristic function. It can be calculated following the procedure given in Reference [11] with parameters used in Reference [12], which are appropriate for TiO_2 at a fixed sputtering current of 9 A in our batch coater. The resulting characteristic function is shown in Figure 1. Also shown in this figure is the pumping speed of the vacuum pump, which amounts to 0.8 m^3/s. The hysteresis region is the

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region where the characteristic function exceeds the pumping speed of the vacuum pump. Hence, in the present setup, the hysteresis should vanish above a pumping speed of 4.5 m^3/s. This is shown in the modeled curves in the inset of Figure 1.

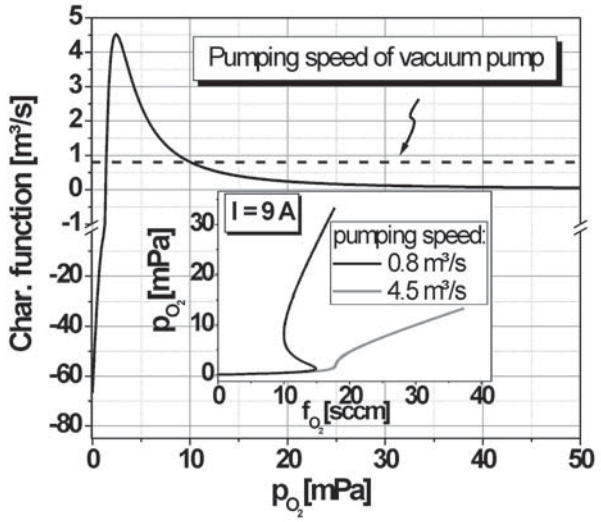

Figure 1: Characteristic function modeled for the TiO_2 process at fixed sputtering current of 9 A. Inset: Vanishing hysteresis at sufficient pumping speed.

However, this huge pumping speed would be difficult to achieve in the given setup. In conclusion, the stabilization of the reactive sputtering process fails in most practical sputter arrangements.

Process stabilization by means of shieldings

Another approach to passive process stabilization focuses on the stabilization by means of shieldings between the target and the substrate. A similar solution has been reported by Reference [13]. The goal is to allow a higher reactive gas partial pressure in front of the substrate than at the target itself. In Figure 2, the setup is shown schematically for a dual magnetron system.

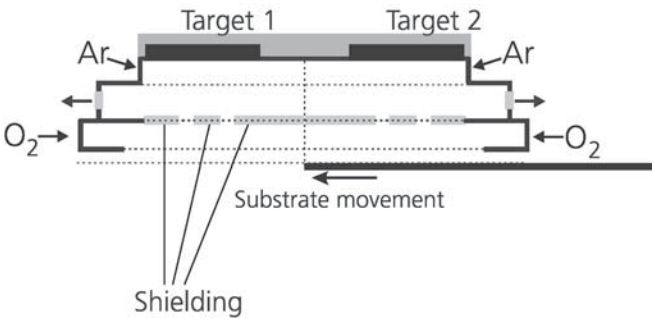

Figure 2: Sputter arrangement with optional shieldings between target and substrate.

For optimum performance, the process might be operated in the stable metallic mode, whereas the films still are stoichiometric due to the gradient in reactive gas partial pressure. Hence no equipment for active process stabilization like OES-control or lambda-control would be required at all.

To model the characteristics of the process, the multichamber Berg / Larsson model was taken into account. This model is described in the work of A. Pflug [14]. It allows one to consider a gradient in the reactive gas partial pressure. Hence, it allows to simulate the reactive gas partial pressure at the inner side of the shielding and at the target region. Figure 3 shows both s-curves. As expected, in the substrate region the reactive gas partial pressure can significantly be increased in comparison to the target region.

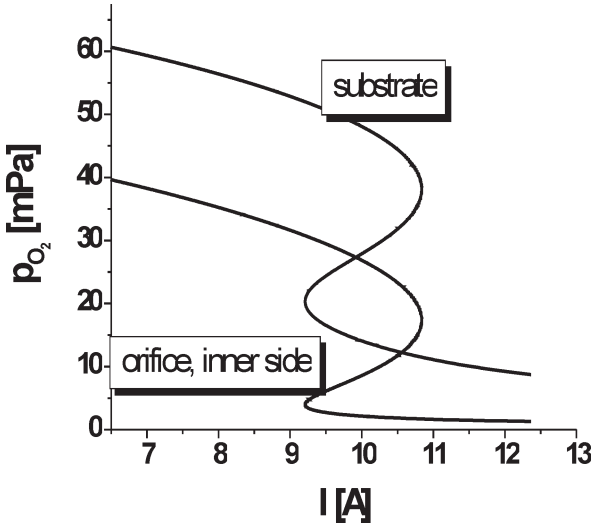

Figure 3: Modeled reactive gas partial pressure in a sputter arrangement corresponding to Figure 2 at the target and the substrate with shieldings.

The corresponding grade of chamber wall / substrate oxidization is shown in Figure 4. The grade of oxidization can be increased from circa 0.57 to 0.93 at the transition from the stable metallic mode to the unstable transition mode.

Clearly, the deposition rate will decrease due to the gettering of ejected target material at the inner side of the shieldings. Figure 6 shows the deposition rates corresponding to Figure 5.

With non optimized shielding geometry a decrease of circa a factor of 2 at a nominal shielding area of 75% is achieved. However, very recent calculations, taking into account the Monte Carlo particle simulation, show, that due to an optimization of the shielding geometry a significant enhancement of the deposition rate should be possible.

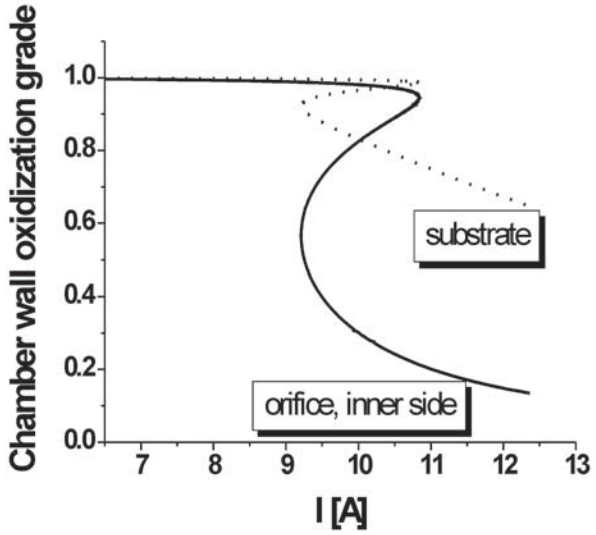

Figure 4: Modeled grade of oxidization at the target and the substrate with shieldings in a sputter arrangement corresponding to Figure 2.

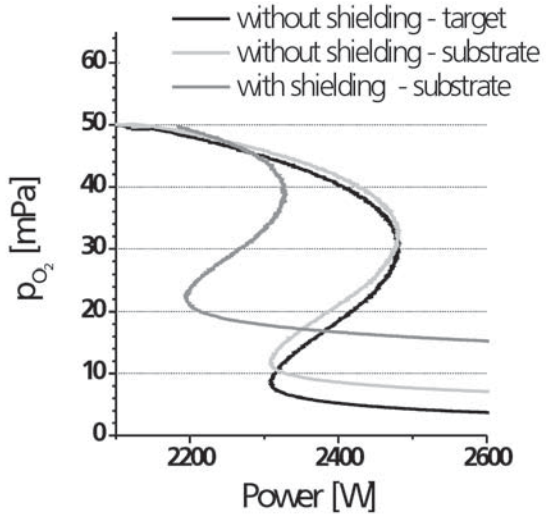

Figure 5: Experimental reactive gas partial pressure curves at the target and the substrate with and without shieldings in a sputter arrangement corresponding to Figure 2.

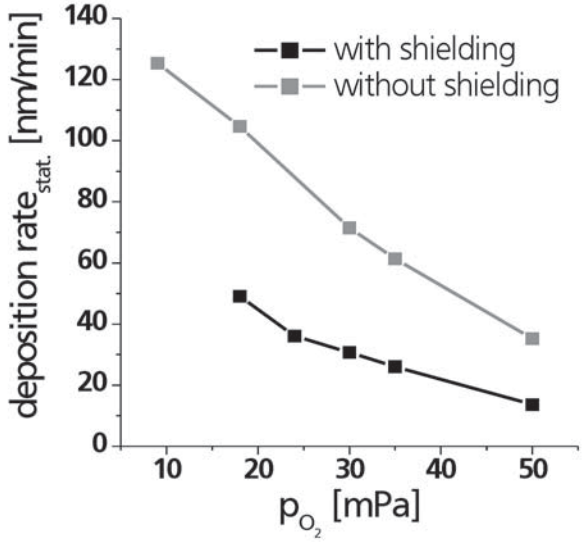

Figure 6: Experimental deposition rates at the substrate with and without shieldings in a sputter arrangement corresponding to Figure 2.

ACTIVE PROCESS STABILIZATION

Survey of different concepts for process control

The most important features of a process control are the expense and the speed at uncompromising precision. In addition, for large area applications the need of a spatial resolution has to be taken into account. Last but not least, for process development the ability of modeling the process is considered to be an important tool for the improvement of the deposition process. Besides, the process control has to be suited for a variety of different materials.

In general, to provide short time constants, the regulation of the process power or sputtering current, respectively, is preferred rather than regulation the reactive gas flow. The time constant of the pressure response for changes in the reactive gas flow is given by the ratio of the pumping speed (including target and chamber wall "pumps") and the chamber volume. In contrast, the sputtering power or sputtering current allows to change the target coverage virtually immediately.

Table 1 shows the most important combinations of control and controlled variables for the stabilization of the reactive sputtering process.

Table 1: Survey of different concepts for process control. Graduation: "+" = good, "0" = medium, "-" = bad, "\" = impossible.

CONTROL VARIABLE → / CONTROLLED VARIABLE ↓	CURRENT OR POWER (CONSTANT FLOW)				REACTIVE GAS FLOW (CONSTANT CURRENT)				
	EXPENSE	SPEED	SPATIAL RES.	MODELING	EXPENSE	SPEED	SPATIAL RES.	MODELING	MATERIALS
U-TARGET	+	+	\	Berg-Larsson + secondary electr.	+/0	0	\	Berg-Larsson + secondary electr.	ZnO, SnO_2, MgO, AlN, Al_2O_3,SiO_2,
OES 1 LINE	impossible				0	0	+	qualitative Larsson	all
OES 2 LINES	0	+	+	qualitative Berg-Larsson	-	0	+	qualitative Berg-Larsson	all
P_{O2}-λ-PROBE	0	0	+	Larsson, Multi-chamber Larsson	0	-	+	Larsson, Multi-chamber Larsson	All Oxides
IMPEDANCE HIGHER HARMONICS	- / +	+	\	At present no model	- / +	0	\	At present no model	TiO_2, Nb_2O_5, ...? Only AC processes

The most universal and flexible control is the 2-line OES at a fixed reactive gas flow. This control is suited for virtually any material and can easily be extended to allow for spatial resolution. In addition, the 2-line OES can easily be upgraded to allow for the stabilization of multigas or multitarget processes. As shown in Figure 7, the single emission line OES can not be adopted for the case of constant reactive gas flow. This figure shows the Zn*-emission line versus reactive gas partial pressure monitored on a s-curve stabilized via λ-control. Due to the non-constant sputtering current, the emission line shows a non-monotonic dependence on the reactive gas partial pressure. As indicated by the intensity of the Ar*, the electron density is strongly varied, although the Ar partial pressure is kept constant. To apply the OES-control in the case of constant reactive gas flow, one has to consider the ratios of 2 emission lines, (e.g. the ratio of Zn* and Ar*, or the ratio of Zn* and O*, which show both a monotonic dependence on the reactive gas partial pressure).

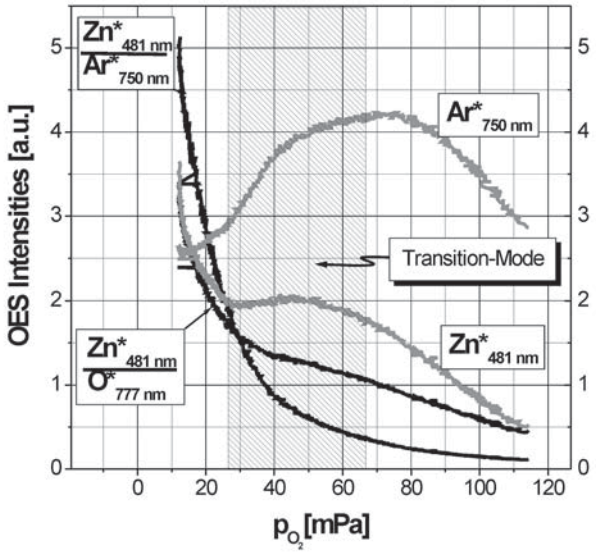

Figure 7: Intensities and ratios of the selected optical Zn*, Ar* and O* emission lines versus reactive gas partial pressure in the reactive ZnO process.

The fastest and most simple process stabilization is the impedance control at fixed reactive gas flow. Almost any power supply provides a constant voltage mode, so that an external control loop is not required. The disadvantage of impedance control is that the measured quantity is an integral quantity connected with the state of the sputtering target, which can not be extended to allow for a spatial resolution. Hence, impedance control is limited to systems with smaller cathodes.

The best compromise of expense and speed is the λ-control at fixed reactive gas flow. The response time of an optimized λ-sensor setup is well below 100 ms, which enables a robust control in combination with a fast control of the target poisoning via the discharge power. A spatial resolution can easily be achieved by an upgrade to several λ-sensors. Another important advantage of the reactive gas partial pressure control is the "easy" modeling.

Unfortunately, there is no cheap and robust sensor similar to a λ-sensor available for nitrogen. The use of mass spectrometers is also reported in the literature [15]. However, the maintenance of mass spectrometers is much more demanding than in the case of λ-sensor.

A drawback of the simple impedance control is clearly the inability for the stabilization of certain processes (e.g. the reactive TiO_2 or the Nb_2O_5 process). In both cases the dependence of the target voltage or plasma impedance, respectively, on the reactive gas partial pressure and the process state is indefinite. However, recently a dependence of the higher harmonics of the target voltage has been observed in the case of the mid frequency process (operated at 40 kHz) [16]. This is shown in Figure 8.

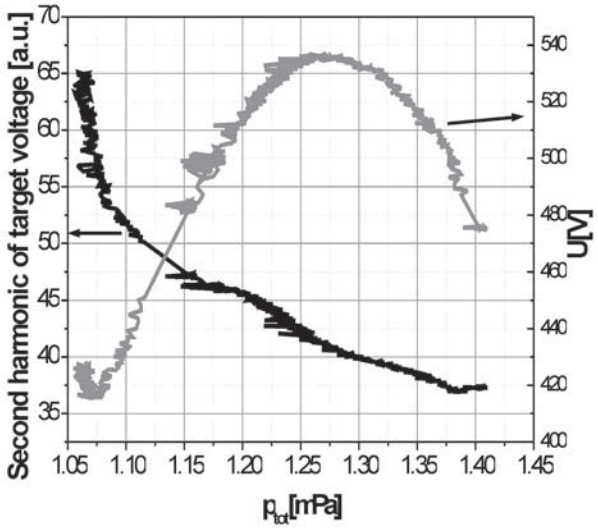

Figure 8: Non monotonic dependence of the target voltage and monotonic dependence of the second harmonic of the target voltage on the reactive gas partial pressure for the Nb_2O_5 mid frequency MF process.

As in the case of classical impedance control this control method is fast in combination with the regulation of the process power at a fixed reactive gas flow. The expense given in the Table 1 is still high due to the used lock-in amplifier or digital oscilloscope in combination with software FFT. However, using simple electronic filters the expense might be drastically decreased. As in the case of the classical impedance control, the nonclassical impedance control is not suited for spatial resolution.

SPECIFIC DEPOSITION RATE

All of the different methods for the process control mentioned above are not suited to ensure transparent films. As example the following Figure 9 may be considered. This figure shows the deposition rate in the reactive TiO_2 process for two different reactive gas partial pressures. Also indicated is the transition from transparent to absorbing layers. Clearly, the reactive gas partial pressure is indefinite with respect to the problem "absorbing or transparent". For example, increasing the reactive gas flow at 3 mPa oxygen partial pressure from 7.5 sccm to 15 sccm will switch the films from transparent to absorbing.

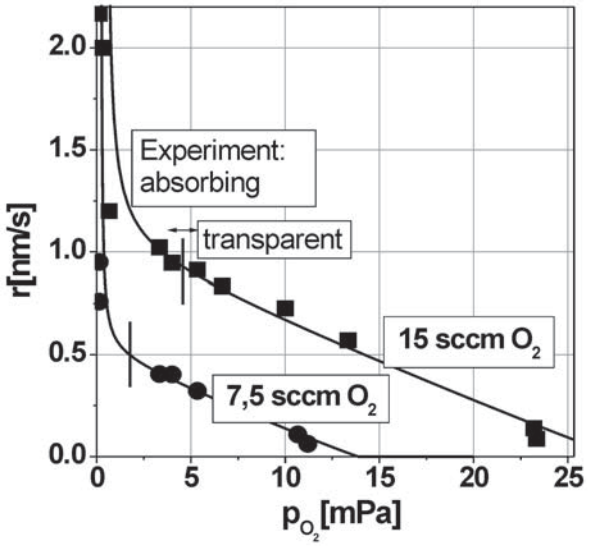

Figure 9: Deposition rate at 15 sccm O_2 and 7.5 sccm O_2 versus reactive gas partial pressure in the reactive TiO_2 process.

Hence, we propose to use the specific deposition rate, which means deposition rate normalized on the sputtering power or sputtering current, respectively. As shown in Figure 10, the transition from absorbing to transparent films occurs at almost the same specific deposition rate, although the deposition rate itself differs by a factor of 2. The same holds in the case of Nb_2O_5, which is not shown here. Thus, limiting the specific deposition rate to a maximum value can ensure transparent films even at different absolute deposition rates.

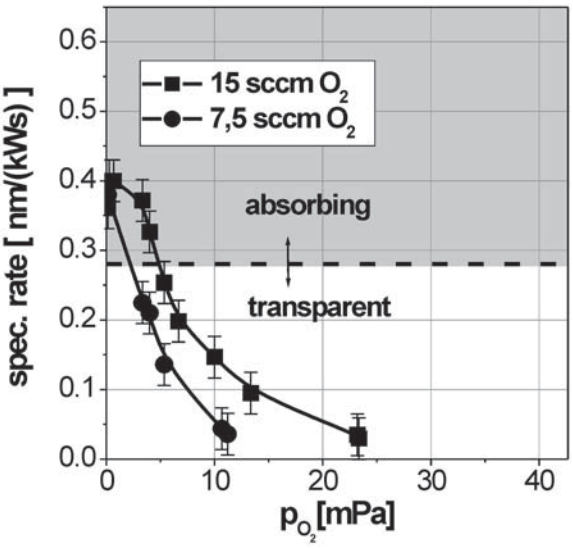

Figure 10: Specific deposition rate at 15 sccm O_2 and 7.5 sccm O_2 versus reactive gas partial pressure in the reactive TiO_2 process.

CONCLUSION

In the present work, different concepts for the stabilization of the reactive sputtering process were considered. In the case of stabilization by means of static modifications, the simulation shows that an overpumping of the hysteresis is virtually impossible for realistic process conditions. Also the use of shielding was modeled in agreement with the experiment. Further improvement of the shielding geometry is required to increase the deposition rate. In the case of active process stabilization we presented a survey of most important control methods. The most flexible is the 2-line OES operated at a fixed reactive gas flow. The best compromise of expense and speed is the combination of λ-sensor and regulating the process power. The most simple control is the impedance control operated at a fixed reactive gas flow. To apply the impedance control also to the well known candidates TiO_2 and Nb_2O_5 , which are inapplicable for classical impedance control, we extended the impedance control to the analysis of higher harmonics in the mid frequency process. However now, the nonclassical impedance control is limited to lab-scale. Finally, to ensure transparent films at different absolute deposition rates, we propose introduce the specific deposition rate as a constraint and to limit the specific deposition rate to a maximum value.

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