A versatile coating tool for reactive in-line sputtering in different pulse modes

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Abstract

Recently, FEP developed a new family of flange mounted magnetron sources together with pulsed powering and process control. This system can be applied either as single magnetrons powered in unipolar pulse mode, e.g. with a pulsed d.c. voltage, or can be applied as a pair of magnetrons powered in bipolar mode, e.g. with an alternating pulsed voltage between both magnetrons that act alternately as the anode and cathode of the discharge. The pulse unit can be switched between the two pulse modes. This allows to investigate directly the influence of the pulse mode and pulse parameters using the same magnetron configuration and the same type of pulse power supply. Experimental results of the reactive deposition of SiO$_2$, Si$_3$N$_4$, and TiO$_2$ demonstrate the specifics of both process modes. There are effects of the pulse mode observed for all these materials that are independent on the target material. Furthermore, we observed effects of the duty cycle, i.e. the ratio of the pulse-on-time to the cycle time of the rectangular current or voltage pulses. The deposition rate and thermal substrate load exhibit a pronounced dependence on the pulse parameters. The values of film properties like refractive index and internal stress will be discussed for the materials SiO$_2$, Si$_3$N$_4$, and TiO$_2$. The flexibility of the pulse sputtering system with free choice of pulse mode allows the universal use either for basic research or in production. The investigations made with this system demonstrate the new possibility to use the pulse parameters to influence film properties. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Reactive sputtering; Pulse sputtering; Magnetron sputter tool; SiO$_2$; Si$_3$N$_4$; TiO$_2$

1. Introduction

The need of coating large substrates with oxides and nitrides at an industrial scale led to the development of high rate reactive sputtering processes. For some materials d.c.-sputtering could successfully be used. But especially for highly insulating films like SiO$_2$ or Al$_2$O$_3$ two main problems of reactive sputtering had to be solved. The covering of the target with reaction products leads to arcing because of charging up of insulating films on the target surface until electric breakdown. The covering of the anode with insulating deposits leads to a drift of the discharge parameters until the break down of the discharge. For a long-term stable process the long-term efficacy of the anode and the arcing problem had to be solved.

Two different solutions using the pulse magnetron sputtering (PMS) technology had been developed in the past.

Solution 1: The bipolar pulsed magnetron sputtering technique uses a bipolar pulse generator, e.g. a sine wave generator. A voltage with alternating polarity is applied between two targets of a Dual Magnetron System. The targets act alternatively as cathode and anode. This ensures a perodical de-charging of the charged deposits on the target surface and avoids arcing. The sputtering of the target keeps the race track electric conductive solving the anode problem without the need of a separate anode. This technique is now
introduced in industry in conjunction with magnetrons mounted inside the chamber and sine wave generators for a variety of applications, especially architectural glass, flat panels and web coating [1–3].

Solution 2: The unipolar pulsed magnetron sputtering technique uses an unipolar pulse generator connected between the target acting as cathode and a so-called ‘hidden anode’. The hidden anode is designed to prevent its coverage with reaction deposits. The pulse powering solves the arcing problem by d.c.-charging of insulating films on the target. This technique is successfully used for instance in conjunction with flange mounted magnetrons for stationary sputtering and square wave pulse generators [4].

Recent investigations using a double ring magnetron for stationary deposition have shown that the pulse mode and the pulse characteristics have an important influence on the behaviour of the sputter process, the charged particle bombardment of the growing film and several film properties [5,6]. These results gave the motivation for the development of a magnetron sputtering system for in-line coating machines that is suited for sputtering in the unipolar as well as in the bipolar pulse mode. In this paper after the description of the system the experimental results obtained using different pulse modes and pulse parameters will be discussed.

2. Rectangular magnetron sputtering system

The RM type rectangular magnetron sputtering system consists of:

- special magnetron sources suited for both unipolar and bipolar pulse sputtering;
- powering that can be switched between unipolar and bipolar pulse mode and allows to adjust pulse parameters; and
- process control system.

2.1. New family of flange mounted magnetron sources type RM

The cross-section in Fig. 1 shows the schematic of the new family of rectangular flange mounted magnetron sputter sources type RM. On the vacuum side of the flange are mounted:

- the target on the backing plate;
- the plasma shield with integrated gas inlet surrounding target and backing plate;
- the hidden anode outside the plasma shield; and
- the outer shield surrounding the discharge space.

The substrate is situated opposite to the target. On the air side of the vacuum flange are mounted:

- the movable magnet system; and
- means for providing the media (water, current and gas).

The features of this type of source are:

- Because of the flange mounted principle there is only a minor number of parts inside the vacuum chamber and no water–vacuum sealing. Furthermore, the concept is versatile concerning the arrangement of two or more targets because the distance and angle between the targets can be adapted to the sputter task by arranging the magnetrons as required. If necessary also single flange mounting of two targets is possible.
- The erosion optimised movable magnet system leads to nearly no redeposition areas and to a remarkably reduced tendency towards flaking and arcing. The ability of moving the magnet system according to the target erosion allows to keep the magnetic field strength and therefore the electric discharge parameters constant throughout the target life time. This is a good basis to get constant film properties during target erosion. The magnet movement and the erosion optimised magnet array lead to low target costs per coated area due to high utilisation of the sputter particle stream and of the target.
- The integrated separate gas inlets for argon and reactive gas are optimised for fast process control.
- Integrated process sensors like pressure sensor (Baratron), mass flow meter and optical emission detector (OED, to measure the intensity of the plasma emission at one or more wavelengths) allow direct and fast access to essential process parameters.
- Integrated process actuators like piezo valves and mass flow controllers ensure fast control of the gases.

The above mentioned features are the basis for the long-term stability of the reactive sputter process.
2.2. Unipolar and bipolar powering

The configuration of the unipolar pulse powering is shown in Fig. 2. The cathode and the anode of the magnetron source are connected to the poles of the pulse unit UBS-C2. This pulse unit transforms the d.c.-power of the d.c.-power supply to unipolar square wave pulses. It delivers current pulses. The pulse unit UBS-C2 developed by Fraunhofer-Institut für Elektronenstrahl- und Plasmatechnik (FEP) contains two independent channels. Therefore together with two magnetrons and two d.c.-power supplies two different processes like SiO₂ and TiO₂ deposition can be operated.

The bipolar pulse powering can be carried out using the same hardware configuration and the bipolar pulse mode of the pulse unit UBS-C2 (Fig. 3). The anode is not connected to the power lines. Optionally the outer shield can be modified to get a common discharge space.

For the experiments discussed below all parts of the magnetron remained in the system and only the powering is switched between the different pulse modes.

The features of the powering with pulse unit UBS-C2 are:

- The pulse mode unipolar or bipolar can be chosen according to the process needs.
- The duty cycle, i.e. the ratio of pulse-on time to the total cycle time, is variable between 15 and 85%.
- The powering via two independent channels allows easy to guarantee symmetric power levels of both discharges that is important in the bipolar mode or — if required — defined asymmetric discharges.
- Furthermore, the separate sputtering or the co-sputtering of two different materials is possible.
- The current source behaviour of the UBS-C2 ensures minimum energy input in the case of micro-arcing independent on other measures regarding arc-handling.

2.3. Coating system

Fig. 4 shows the schematic of the coating system. Additionally to the already described components — magnetron sources and powering — a process control system is necessary.

The process management computer (PMC) gets the measured values from the process like:

- the electrical discharge parameters and target lifetime from the power supplies;
- the pressure from the integrated Baratron pressure sensor;
- the gas flow values for argon and reactive gas from the integrated mass flow meters or mass flow controllers;
- the position of the magnet system from the integrated position sensor; and
- arc detection values from the UBS-C2.
The PMC controls:
- the pulse mode (unipolar or bipolar);
- the electrical discharge parameters of each of the discharges;
- the argon gas inlet to ensure constant process pressure;
- the flow of the reactive gases to stabilize the discharge in the reactive working point via the integrated piezo valves or to ensure a defined reactive gas flow in the case of reactive gas mixtures;
- the position of the magnet systems to compensate the target erosion;
- the communication with the host computer of the sputter plant;
- the communication to FEP by remote control via telephone line to allow maintenance, service, process support.

The communication between the PMC and the different units is implemented via Profibus with an optical fibre loop. This ensures a very stable operation of the whole system despite the electromagnetic noise coming from the pulsed plasma process. The system operates self-sufficiently. The automatic run of the complete deposition procedure including pre-sputtering, adjustment at the reactive working point and defined substrate coating will be carried out via recipes. Process sequences contain among others setting of pressure, electrical discharge parameters, optical emission values and gas mixture including time dependence of the parameters.

3. Pulse mode and pulse characteristics as technological parameters

The following experiments were carried out in an in-line sputtering equipment using magnetrons with a target length of 400 mm and a target width of 130 mm. For all experiments the target to substrate distance was 73 mm, the target power density on the two targets was 9.1 W/cm² each (SiO₂ and Si₃N₄) and 18.2 W/cm² (TiO₂) each, the pressure was set to 1 Pa and the pulse frequency was 50 kHz. No changes were made concerning the hardware components used except switching the pulse mode and changing the duty cycle.

3.1. Deposition rate

The deposition rate was determined by profilometer measurement of a layer step on a glass substrate using a Tencor P2 profilometer. The maximum deposition rate to achieve fully transparent films was determined by a series of deposition runs at constant power level varying the reactive working point between metallic and reactive mode within small steps. The criterion for the right working point was an absorption value at 550 nm below 2 × 10⁻¹⁴ (SiO₂), 1 × 10⁻¹⁴ (Si₃N₄) and < 5 × 10⁻¹⁴ (TiO₂), respectively.

Fig. 5 shows the deposition rate of non-reactive sputtered Si films and the maximum rate of reactive sputtered transparent SiO₂ films using two targets in dependence on the pulse mode and the duty cycle, i.e. the ratio of the pulse-on time to the cycle time.

The deposition rate in the unipolar pulse mode decreases linearly with the duty cycle in the investigated range between 50 and 80%. The deposition rate is reduced by 26% at duty cycle 50% compared to duty cycle 80%, i.e. a factor of 1.6 in the power density during the pulse-on time. Because of the constant time averaged power fed into the discharge, the power density during the pulse-on time increases linearly with decreasing pulse-on time. Also in d.c. sputtering the deposition rate per kW is reduced with increasing power density, however, for our system only by 13% for

![Fig. 5. Deposition rate of SiO₂ films in dependence on the pulse mode and on the duty cycle; process parameters: pressure 1 Pa; target to substrate distance 73 mm; two targets with target power density 9.1 W/cm² each; frequency 50 kHz. ■, SiO₂ (unipolar pulse mode); □, SiO₂ (bipolar pulse mode); ▲, Si (unipolar pulse mode); △, Si (bipolar pulse mode).](image-url)
an increase in power density by a factor of 1.6. Therefore the decrease in deposition rate partly has to be ascribed to another effect. A shorter duty cycle leads to a larger amplitude of recombination and generation of charged particles resulting in more pronounced heating of the bulk plasma. We therefore attribute the decrease in the deposition rate to energy losses in the plasma that rise with shorter pulse-on time.

The influence of the pulse mode is different for Si and SiO. The pulse-on time in the bipolar mode is 47% of the cycle time. For comparison the corresponding deposition rate in the unipolar mode was extrapolated from the straight line. The deposition rate of Si in the unipolar mode is 102% of the bipolar value, i.e. almost the same. In contrast the deposition rate of unipolar pulse sputtered SiO amounts to 120% of bipolar pulse sputtered films. Because of the result of Si discussed above, we assume that in the bipolar pulse mode the oxide coverage of the target to achieve transparent films needs to be higher compared to the unipolar mode. The higher target coverage would lead to a lower sputter rate because of the lower sputter rate of silicon dioxide compared to silicon.

Of practical interest is the significantly higher deposition rate in the unipolar mode at the same power level. The rate of SiO in the unipolar mode at 80% duty cycle amounts to 120% of bipolar pulse sputtered films. Because of the result of Si discussed above, we assume that in the bipolar pulse mode the oxide coverage of the target to achieve transparent films needs to be higher compared to the unipolar mode. The higher target coverage would lead to a lower sputter rate because of the lower sputter rate of silicon dioxide compared to silicon.

3.2. Thermal substrate load

The thermal substrate load during deposition was calculated from the temperature rise during sputtering of a glass substrate measuring 50 × 50 × 3 mm positioned thermally insulated on the substrate carrier. The temperature was measured using temperature measurement strips (ibv TM-strips of van Belle GmbH). The power loss in the UBS-C2 was determined to be 5 ± 1% of the used d.c.-power for all experiments — that means constant.

The accuracy of the absolute values may be limited by this method to approximately ±15%. The ratio of the thermal load of the unipolar and the bipolar pulse mode is more accurate (approx. ±5%) because we used the same conditions for both modes.

The influence of the duty cycle was investigated for SiO using static substrate deposition (no substrate movement) and 50 and 80% duty cycle, respectively. The values were 0.3 ± 0.01 W/cm² in both cases, indicating only minor influence of the duty cycle on the thermal substrate load.

Two different experiments were carried out coating the substrate with single pass only. In the unipolar mode the duty cycle was set to 80%.

To determine the mean power density acting on the substrate within the first experiment the temperature rise at the same carrier speed and at the same power level of the discharge was measured.

The second experiment was done to determine the thermal load in the two pulse modes for the same film thickness on the substrate. Because of the lower deposition rate at the same power level in the bipolar pulse mode, we used a lower carrier speed.

The materials investigated are: SiO, SiN₄ and TiO₂.

The power density at the substrate during SiO₂ deposition (Table 1) is by a factor of 1.8 higher in the bipolar mode compared to the unipolar mode. Because of the lower deposition rate the thermal load onto the substrate for the deposition of a film with the same thickness is in the bipolar mode by a factor of 2.75 higher than the unipolar value. This means that the

<table>
<thead>
<tr>
<th>Material</th>
<th>Pulse mode</th>
<th>Target power density (W/cm²)</th>
<th>Power density (W/cm²)</th>
<th>Single pass dynamic deposition</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Film thickness (nm)</td>
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<td>SiO₂</td>
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<td>0.14</td>
<td>600</td>
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<tr>
<td></td>
<td>Bipolar</td>
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</tr>
<tr>
<td>SiN₄</td>
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<td>0.096</td>
<td>360</td>
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<tr>
<td></td>
<td>Bipolar</td>
<td>9.1</td>
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<tr>
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<td>Bipolar</td>
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<td>100</td>
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</tbody>
</table>

*Process parameters: pressure 1 Pa; target to substrate distance 73 mm; two targets with target power density 9.1 W/cm² each; frequency 50 kHz.*
temperature rise of the substrate is also by a factor of 2.75 higher in the bipolar mode.

We observed similar results for Si$_3$N$_4$ (2.1 higher power density and 2.6 higher temperature rise for same film thickness) and TiO$_2$ (1.5 higher power density and 2.0 higher temperature rise for same film thickness).

Jäger et al. [7] observed for a bipolar pulse powered system with sine wave generator a factor of ten in the energy of plasma ions compared to d.c.-sputtering of two targets.

At FEP more detailed investigations regarding the influence of the pulse mode and pulse parameters were done by Bartzsch et al. [6] using another dual cathode arrangement — the double ring magnetron. The results of these investigations show clearly that the magnetically shielded anode in the bipolar pulse mode leads to a strongly increased plasma density and electron temperature in the substrate region. This again results in an increased energetic bombardment of the substrate and accounts for the difference between the unipolar and bipolar pulse mode. There are additional effects of the pulsing depending on the duty cycle like a rise of the substrate self bias voltage and ion current density resulting in a higher power density at smaller duty cycles.

As a consequence of these results, the unipolar pulse mode is recommended for the coating of thermal sensitive substrates. The bipolar mode is favourable when higher substrate temperatures and higher particle bombardment of the growing film are required. According to Bartzsch et al. [6] values of the ion current density can be achieved that are comparable to d.c. closed field unbalanced magnetron systems. The ion current densities for the system described here is subject to further investigations.

3.3. Optical data

The optical refractive index and absorption constant were determined using a Perkin-Elmer UV/VIS/NIR spectrometer Lambda 19 on 400-nm-thick films on glass and an ellipsometer Sentech SE 850 on films of 100-nm film thickness on silicon wafers coated in the pass mode.

Values of the refractive index and absorption coefficients of SiO$_2$, Si$_3$N$_4$ and TiO$_2$ are listed in Table 2. We observed no significant difference of these optical data between the two pulse modes.

### 3.4. Hardness

The hardness was determined on films with a thickness of 2–4 μm on glass substrates or silicon wafers by means of a Leco Microhardness tester M400-PC3/Vickers Hardness.

The hardness of Si$_3$N$_4$ in the bipolar pulse mode (1704 HV 0.01) was higher compared to that value of the unipolar pulse mode (1623 HV 0.01). The same behaviour had also been observed for Al$_2$O$_3$ deposited in the stationary deposition mode exhibiting approximately 100 HV 0.01 higher hardness values in the bipolar pulse mode (verified at different argon pressures 0.5 and 1 Pa [5,6]).

We assume that the higher particle bombardment during bipolar pulse deposition leads to harder films.

### 3.5. Internal stress

The internal stress was calculated from the bending of a 150-μm-thick glass strip coated with a 100-nm-thick Si$_3$N$_4$ film. The bending was determined by profilometer measurement.

The internal stress of films deposited in the bipolar pulse mode (−610 MPa) was much more compressive than in the unipolar pulse mode (−150 MPa).

Further investigations also on other materials are necessary to be able to draw conclusions regarding a correlation between the pulse mode and the internal stress.

### 4. Summary

Within this paper a versatile tool for reactive in-line
sputtering could be presented that allows to use different pulse modes and duty cycles.

Some of the process characteristics and film properties for different materials like SiO$_2$, Si$_3$N$_4$ and TiO$_2$ exhibit a correlation to the pulse mode. The deposition rate is up to 40% higher in the unipolar pulse mode depending on the sputtered material and the reactive gas and on the duty cycle. The thermal substrate load is up to a factor of 3 higher in the bipolar pulse mode than in the unipolar pulse mode. The hardness of the films deposited in the bipolar pulse mode is higher than in the unipolar pulse mode.

Other parameters are more or less independent like the optical data.

The choice of the pulse mode influences the deposition parameters at the substrate like plasma density, ion current density and ion energy leading to differences regarding thermal substrate load and regarding some of the film properties.

Our investigations lead to the conclusion that the pulse parameters like pulse mode and duty cycle are technological parameters and can be used to optimise the deposition process. This gives new degrees of freedom to meet the required combination of film properties.

Acknowledgements

The authors would like to thank Dr. O. Zyowitzki for all measurements of film properties and Dr. U. Hartung for creating the process control software.

References