Highly Insulating Al_2O_3, SiO_2, and Si_3N_4 Films for Sensor Applications Deposited by Reactive Pulse Magnetron Sputtering

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ABSTRACT

Applications in sensor, automotive and aviation technology require thin films that exhibit electrical insulating properties at room temperature but also at elevated temperatures. One technology for the deposition of such films is reactive pulse magnetron sputtering. Because of the high deposition rate this technology is especially interesting for the deposition of thick insulating films of several microns allowing high insulation voltages up to 800V or deposition onto relatively rough substrates e.g. stainless steel. In this paper the breakdown field strength and resistivity of such sputter deposited Al_2O_3, SiO_2 and Si_3N_4 films are investigated in the temperature range between room temperature and 400°C. All investigated films show excellent insulation properties at room temperature. At high temperatures films remain insulating at slightly reduced brekdown voltage. The combination of different film materials allows fulfilling the requirements not only on insulating but also on thermo mechanical properties. One example of industrial application is the deposition of electrical insulation films onto the membranes of pressure sensors using cluster type sputter equipment.

INTRODUCTION

Thin layers having high insulation resistance are required for many applications in electronics, sensor technology and photovoltaic technology. These applications include gate oxide layers in microelectronics (which are only a few nanometers thick), insulating layers in sensor applications (where dielectric strengths up to 1000V can be required) and insulating layers in photovoltaic technology (where the requirements on dielectric strength are lower but where large areas must often have a breakdown free layer).

In addition to having high breakdown field strength, high insulation resistivity and high area yield, other properties are often required. These are for example resistance to high temperature, resistance to aggressive media, a satisfactory mechanical load limit, effective permeation barriers, good adaptation of the coefficients of expansion to the substrate, dielectric strength (also in contact with electrolytes) and resistance of the layer in downstream processing steps such as laser trimming or wet-chemical etching processes. The coating costs must also be in reasonable proportion to the value of the end-product, meaning that there is often a demand for high deposition rates.

Chemical vapor deposition processes (CVD and PECVD) are the most established processes for manufacturing such insulation layers (e.g. [1]). The advantages of these processes are the high deposition rates and good insulation properties that are achieved. However, some of these layers have shortcomings regarding their mechanical properties, temperature resistance and permeation barrier properties. RF magnetron sputtering on the other hand allows very effective insulation layers to be deposited, albeit at relatively low deposition rates [2,3].

This article describes the use of reactive pulse magnetron sputtering in the stationary mode for manufacturing insulating layers [4-6]. Due to the high deposition rates that can be achieved, this process is especially suitable for producing thick layers where very high dielectric strength is required. For example, such layers are already used in pressure sensors. In order to demonstrate the insulation properties at high temperature, investigations at a 400°C sample temperature are presented. Layers that are insulating at this temperature could find applications in sensor, automotive and aviation technology.

EXPERIMENTAL SETUP

Film deposition was carried out by stationary pulse magnetron sputtering (PMS). Two different sputter plants were used: a laboratory sputtering system at the Fraunhofer-Institut für Elektronenstrahl- und Plasmatechnik (FEP) and a cluster type sputtering system (ClusterSystem CS 400 S, Von Ardenne Anlagentechnik) at Siegert TFT (Figure 1). In both cases, the Double Ring Magnetron DRM 400 (FEP) was used as the sputter source. This type of magnetron combines two concentric discharges on two separate targets in one magnetron source [7]. Film thickness uniformity of up to ±1% across an 8” wafer (200 mm) is achieved by superposition of the film thickness distributions of these two discharges.

52nd Annual Technical Conference Proceedings, Santa Clara, CA, May 9–14, 2009 ISSN 0737-5921

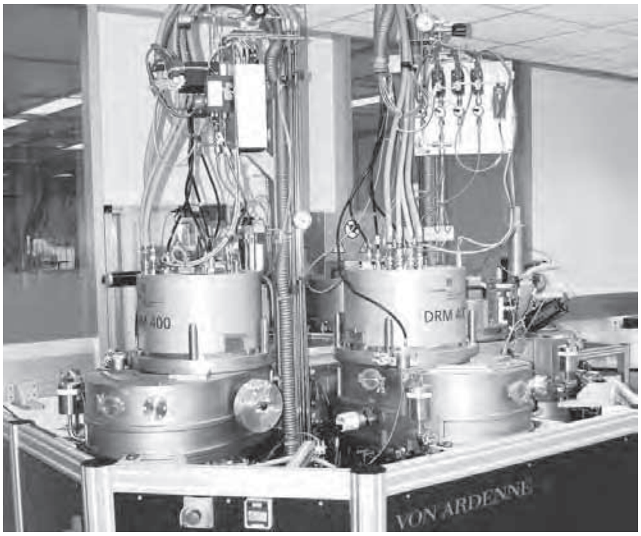

Figure 1: Deposition plant ClusterSystem CS 400 S with DRM 400 magnetrons.

During reactive sputtering, insulating deposits on plasma shields and on the target edges may charge up and result in unwanted arcing. Therefore, pulse powering was applied in order to regularly discharge the surfaces of insulating deposits and hence ensure the long term stability of the process, even at high powers up to 10 kW. For this the pulse unit UBS-C2 (FEP) was used. This converts the dc power of two dc power supplies into current pulses for each target.

Most deposition experiments were carried out by unipolar and bipolar pulse sputtering in the mid-frequency range (50 kHz) from metallic Al or Si targets. In addition, some experiments were carried out by RF sputtering (13.56 MHz) from an SiO_2 target (Figure 2c). For RF powering, the Advanced Energy RFX 3000 was used.

In the unipolar mode (Figure 2a), pulse powering having negative polarity is applied between each of the targets and the common anode. This anode is not sputtered. In the bipolar mode (Figure 2b), pulse powering with alternating polarity is applied between the two targets that act alternately as cathode and anode of the discharge. Due to the higher power at the outer target compared to the inner target, the bipolar powering is asymmetric.

In unipolar as well as in bipolar pulse mode closed loop reactive gas control for oxygen was applied in order to stabilize the reactive working point of the discharge in the so-called transition mode. This allows the highest possible deposition rate at a given power level to be achieved because stoichiometric films can be deposited by sputtering from a near-metallic target at high sputter yield. The SiO_2, Si_3N_4 and Al_2O_3 films investigated in this paper were deposited at 7 kW power with deposition rates of approximately 150 nm/min for SiO_2 and Al_2O_3 and 80 nm/min for Si_3N_4.

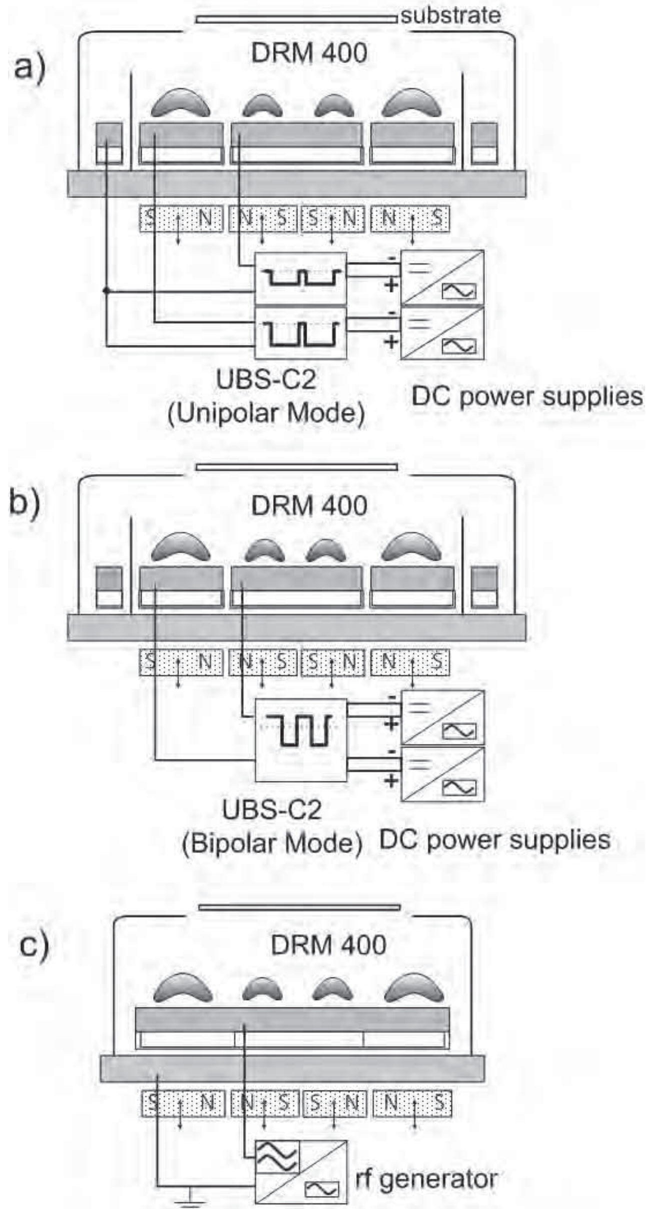

Figure 2: DRM 400 in unipolar pulse (a), bipolar pulse (b) and RF (c) operation.

Some of the Al_2O_3 films were deposited in the fully reactive mode without closed loop control. In this mode, constant argon and oxygen flows of 40 sccm each were supplied. The target is completely covered with insulating deposits and the deposition rate is only 10 nm/min.

In the RF deposition mode, constant argon and oxygen flows of respectively 40 and 20 sccm were used. The deposition rate of SiO_2 was 15 nm/min at 3 kW RF power.

The sputtering pressure was 0.5 Pa for pulse deposition and 1.0 Pa for RF deposition. The target to substrate distance was always 90 mm.

The targets and anode of the magnetron are water cooled. The substrates were positioned on a substrate carrier which itself is placed on a substrate platform. This platform is water-cooled. The substrate temperature during coating rises from room temperature to 150-200°C. Usually the substrates were at floating potential. During some of the SiO_2 deposition experiments, an additional RF substrate bias was applied. This was achieved by applying an RF power (13.56 MHz) of 300W to the metal plate resulting in a power density of 0.4 W/cm^2. The measured bias voltage was -170V in the unipolar pulse mode and -30V in the bipolar pulse mode. The considerably lower bias voltage at the same RF power in the bipolar mode is caused by the significantly higher plasma density and ion current at the substrate [8].

FILM CHARACTERIZATION

On the one hand, single films of SiO_2, Al_2O_3 and Si_3N_4 with a thickness of 1 µm were deposited onto Czochalsky type p doped silicon wafers. After deposition the substrate was coated with an Al pad structure using magnetron sputtering with a shadow mask at the substrate. The electrode area of the pads was 12.6 mm^2. The dc-resistivity and breakdown voltage at room temperature were measured using a Fischer-Elektronik Tera-Ohmmeter TO-3 (current resolution: 0.1 pA; voltage: 0-1500V). The dc values of resistivity were measured at 400V for the SiO_2 and Al_2O_3 films and at 100V for the Si_3N_4 films. The breakdown field strength was determined at the voltage where a threshold current density of 0.1 µA/cm^2 was exceeded.

On the other hand, SiO_2 and Al_2O_3 single layers as well as a SiO_2-Si_3N_4-Al_2O_3 layer stack were deposited on steel membranes of pressure sensors. The working principle of these pressure sensors is based on the fact that the resistance of a resistive structure deposited onto the insulating films changes upon deformation of the steel membrane. The overall layer thickness of the insulating films was 7 µm. For characterization of the insulating film an Al pad was sputtered on top of the insulating film. In this case, the electrode area was 8.0 mm^2. The SiO_2-Si_3N_4-Al_2O_3 layer stack already finds application in a pressure sensor for low temperature applications. The insulating stack not only fulfills the requirements on insulation but also regarding the thermo-mechanical properties and diffusion barrier properties (Figure 3).

Figure 3: Pressure sensors based on metal membranes with sputtered insulating layers and structured resistive and contact layers (Siegert TFT).

To evaluate the high temperature stability of the insulating layers, the yield of the pads was evaluated applying a criterion of threshold current density (13 µA/cm^2) at sample temperatures of room temperature (RT) and 400°C. The measurement voltage was 250, 500 and 800V. For example, a yield of 90% at 400°C sample temperature and 800V measurement voltage means that the leakage current density of 90% of the pads was below 13 µA/cm^2 at 400 °C and 800V measurement voltage. At least 40 pads were measured for each measurement parameter set.

RESULTS AND DISCUSSION

SiO_2 films on silicon wafers

Using the measurement data in Table 1 the influence of the powering (unipolar pulse, bipolar pulse or RF) and an additional RF substrate bias on the electrical properties of SiO_2 will be discussed. Furthermore, the deposition rate and the energy input per unit film thickness are given in Table 1.

Values of resistivity and breakdown field strength range from $3.2\cdot10^{15}$ to $1.3\cdot10^{17}$ Ω·cm and from 4.3 to 8.1 MV/cm respectively. Films deposited in the bipolar pulse mode have higher values of breakdown field strength and resistivity compared to films deposited in the unipolar pulse mode. Films deposited by pulse sputtering with additional RF substrate bias also have higher values of breakdown field strength and resistivity than films deposited without bias, in both unipolar and bipolar pulse modes. Films deposited by RF sputtering have a comparable resistivity but have a higher breakdown field strength than the films deposited by pulse sputtering.

Table 1: Deposition rate, resistivity, breakdown voltage and energy bombardment of SiO_2 films deposited onto silicon wafers in unipolar pulse, bipolar pulse and RF modes with and without additional RF substrate bias; reactive working point of unipolar and bipolar pulse depositions in the transition mode.

Powering/RF Substrate Bias	Deposition Rate [nm/min]	DC-Resistivity [Ω·cm]	Breakdown Field Strength [MV/cm]	Energy Input per Unit Film Thickness [J/(cm²·nm)]
Unipolar Pulse	231	$3.2 \cdot 10^{15}$	4.3	0.04
Bipolar Pulse	156	$6.3 \cdot 10^{16}$	5.6	0.27
Unipolar Pulse/RF Substrate bias	151	$1.3 \cdot 10^{17}$	5.9	0.21
Bipolar Pulse/RF Substrate Bias	159	$5.2 \cdot 10^{16}$	6.2	0.42
RF Sputtering	15	$1.0 \cdot 10^{17}$	8.1	0.68

We assume that the observed differences in the electrical properties are mainly related to the energy input per unit film thickness (Table 1). To estimate this value, the thermal substrate load was determined by measuring the temperature rise of the substrate during deposition using temperature labels. The resulting load (in W/cm^2) was divided by the deposition rate. The given value for the energy input therefore represents the sum of all the contributions to the energy input onto the substrate such as the heat of condensation and reaction, plasma radiation and bombardment with energetic particles from the plasma. In the bipolar pulse mode the energy input is considerably higher than in the unipolar pulse mode. This effect has been explained in detail elsewhere [8]. It is mainly due to the extension of regions with high plasma density and electron temperature towards the substrate in the bipolar pulse mode. When an RF substrate bias is applied, the energetic bombardment of the film is also increased. The high value of energy input in RF sputtered films is caused on the one hand by the higher plasma density of RF discharges and on the other hand by the considerably smaller deposition rate in RF sputtering.

Comparison of the results in Table 1 shows that the increase in energetic substrate bombardment both by using the bipolar pulse mode and by applying an additional substrate bias leads to higher values of breakdown field strength and resistivity. This result indicates that changing the pulse mode from unipolar to bipolar has a similar effect on these film properties to applying an RF substrate bias. The very high value of energy input in RF sputtering is associated with the highest measured value of breakdown field strength.

Si_3N_4 films on silicon wafers

For the deposition of Si_3N_4 films, the bipolar pulse mode was chosen. Table 2 shows the deposition rate and the electrical properties of these layers deposited on silicon wafers. Both the resistivity and breakdown field strength are significantly lower than for the SiO_2 films.

Table 2: Deposition rate, resistivity and breakdown voltage of Si_3N_4 films deposited onto silicon substrates; reactive working point in the transition mode.

Powering	Deposition Rate [nm/min]	DC-Resistivity [Ω·cm]	Breakdown Field Strength [MV/cm]
Bipolar Pulse	79	$5.2 \cdot 10^{13}$	2.4

The main reason for using Si_3N_4 layers in high insulating layer stacks is because of their high diffusion barrier properties against oxygen and water vapor. Only secondary come their electrical insulating properties. The electrical properties of the Si_3N_4 films can be favorably combined with SiO_2 layers. This is especially true because SiO_2 and Si_3N_4 films can be deposited in one deposition chamber, by simply switching the reactive gas between oxygen and nitrogen [9].

Al_2O_3 films on silicon wafers

The data in Table 3 show the influence of the pulse mode and reactive working point on the electrical properties of Al_2O_3. Films deposited in the unipolar and bipolar pulse modes have similar properties, with slightly higher breakdown field strength in the unipolar mode and a higher resistivity in the bipolar pulse mode. Films deposited with unipolar pulse powering at constant reactive gas flow in the fully reactive mode rather than in the transition mode also have good insulating properties. Al_2O_3 films deposited in the bipolar pulse mode with fully reactive working point at a high energy input have very poor insulating properties.

It is known from the literature that strong energetic bombardment causes removal of oxygen atoms, and especially so in Al_2O_3 films [10]. We assume that this slight oxygen deficiency in the Al_2O_3 films causes the poor electrical properties. In

Table 3: Deposition rate, resistivity, breakdown voltage and energy bombardment of Al_2O_3 films deposited onto silicon wafers in the unipolar pulse and bipolar pulse modes; reactive working point in the transition mode (closed loop reactive gas control) or in the fully reactive mode (constant reactive gas flow).

Powering/Reactive Working Point	Deposition Rate [nm/min]	DC-Resistivity [Ω·cm]	Breakdown Field Strength [MV/cm]	Energy Input per Unit Film Thickness [J/(cm²·nm)]
Unipolar Pulse/Transition Mode	146	$2.3 \cdot 10^{16}$	6.2	0.06
Bipolar Pulse/Transition Mode	65	$2.0 \cdot 10^{16}$	5.1	0.65
Unipolar Pulse/Fully Reactive Mode	12	$2.2 \cdot 10^{16}$	5.7	0.7
Bipolar Pulse/Fully Reactive Mode	10		<0.1	4.2

contrast, energetic substrate bombardment causes a densification of the SiO_2 films and hence improves the electrical properties.

Figure 4 shows the SEM micrograph of an Al_2O_3 film deposited in unipolar pulse /transition mode. The film shows a very dense, glass-like structure.

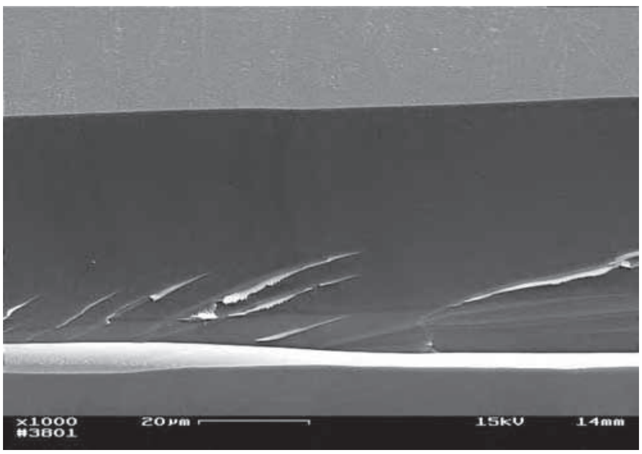

Figure 4: SEM micrograph of an Al_2O_3 film deposited in the unipolar pulse mode, film thickness 40 µm.

Insulating layer stacks in high temperature pressure sensors

Figure 5 shows the yield of high insulating layers on steel membranes for sample temperatures of room temperature (RT) and 400°C. For a measurement voltage of 250V, the yield of high insulating pads is >95% independent on the sample temperature. For higher measurement voltage, the yield only slightly decreases after increasing the sample temperature from RT to 400°C.

Surprisingly, pure SiO_2 and Al_2O_3 layers show slightly better insulating properties than the SiO_2-Si_3N_4-Al_2O_3 layer stack. We attribute this effect to electronic defects that are present at the layer interfaces. However, the SiO_2-Si_3N_4-Al_2O_3 layer stack also fulfills the thermo-mechanical and diffusion barrier requirements. Thus, it is better suited for application in pressure sensors than single layers having the same total thickness.

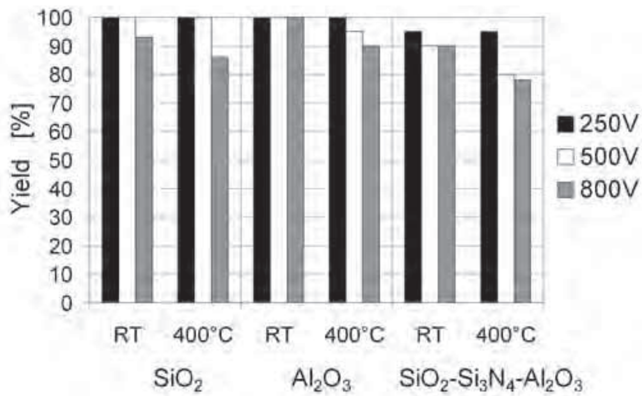

Figure 5: Yield of high insulating pads on steel membranes for measurement temperatures of RT and 400°C and for measurement voltages of 250, 500 and 800V (criterion of threshold current density for breakdown: 13 µA/cm²).

SUMMARY

The electrical properties of SiO_2, Si_3N_4 and Al_2O_3 films deposited by reactive pulse magnetron sputtering have been investigated. The properties of the SiO_2 and Al_2O_3 layers are close to the reference SiO_2 films deposited by RF sputtering. The investigations show that films with high breakdown field strength and low leakage current can be deposited with a deposition rate one order of magnitude higher than the conventional techniques.

Thicker films deposited by reactive pulse sputtering show breakdown voltages above 800V and can be used, for example, as protective layers and for sensor applications. These layers are already finding application in low temperature pressure sensors. The high temperature stability of the insulating layers could be proven up to a sample temperature of 400°C. They may therefore have potential applications in automotive and aviation technology where stability at elevated temperatures is also required. One example application is direct pressure measurement in the combustion chamber of vehicle engines.

ACKNOWLEDGEMENTS

This work was partially funded by the Federal Ministry of Economics and Technology, grant number KF0271302KDA3 and by the Federal Ministry of Education and Research, grant number 13N9285.

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