SOLID-STATE SCIENCE AND TECHNOLOGY

The Hydrogen Content and Properties of SiO₂ Films Deposited from Tetraethoxysilane at 27 MHz in Various Gas Mixtures

M. G. J. Vepřek-Heijman¹ and D. Boutard

Max Planck Institute for Plasma Physics, Euratom Association, D 8046 Garching, Germany

ABSTRACT

The deposition of SiO₂ films from tetraethoxysilane (TEOS) is investigated in gas mixtures containing either a large amount of oxygen or Ar. It is shown that films with good insulating properties can be deposited from both mixtures. In Ar/TEOS mixtures, films with C content below 1% have been obtained. In the case of oxygen, the rate must be kept low at low temperature to allow for the disappearance of hydrogen (water) from the film, for example, in our system, at 280°C below 12 nm/min. The dependence of the deposition rate on the temperature, dwell time, and partial pressure of TEOS are shown. Variation of the pressure (at constant flow rate and pumping speed) upon ignition of the plasma in the oxygen and Ar system are different, as well as the dependencies of the deposition rate on temperature. Possible reasons are discussed.

The deposition of SiO_2 films from tetraethoxysilane is one of the oldest systems in the field of plasma induced chemical vapor deposition (1, 2). The films obtained in the past were, however, surpassed in quality (low carbon contamination, water content, and chemical stability) by the use of silane and an oxygen-containing reagent as educts (3). Recently the interest in this system revived because of the need for a deposition process at low temperature giving better step coverage than the above-mentioned silane system (4, 5). Also, the handling of the nonexplosive TEOS places less severe safety requirements on the processing environment as compared to silane.

Most studies reported the use of some carrier gas to deliver the TEOS to the reactor. To this mixture, oxygen is usually added to reduce the carbon content of the films (2, 4, 6, 7). Typically, an O₂-to-TEOS ratio of at least four has to be used. Recently, however, Hsieh *et al.* (8) reported that very good SiO₂ films (with low OH content) in a He/TEOS mixture containing very little oxygen (O₂-to-TEOS ratio 0.5) could be obtained. Even in an oxygen-free plasma, the carbon content was not more than 3 atom percent (a/o).

Already Ing and Davern (1) have remarked that the quality of the film decreased with increasing deposition rate. Because the addition of oxygen normally enhances the rate, the dependence of the quality of the films on the OTR (oxygen-to-TEOS ratio) is not unambiguously established.

Another important parameter in plasma chemical vapor deposition (CVD) is the excitation frequency of the electrical field. The deposition from TEOS has been carried out with a large variety of plasma excitation frequencies from the kHz range (9, 10) to microwave, including downstream deposition in the afterglow (2, 6, 11, 12). This variety of deposition conditions results in different degrees of ion bombardment of the growing film and, possibly, also in different intermediates for the deposition. The influence of the excitation frequency on the step coverage was investigated recently (12b). It was concluded that low-energy ions promote directional deposition, at least in an oxygen-lean gas mixture.

Our SiO_2 films are intended to be used on stainless steel foils of 5 μ thickness as an isolation layer in bolometer devices for controlled fusion machines (13). Because of the large difference in thermal expansion between SiO_2 and steel, large compressive stress in the films should be avoided and annealing at very high temperatures cannot be allowed. The main quality indicator is the breakdown field strength. Large compressive stresses in films deposited by plasma CVD result usually from high-energy ion bombardment during processing and from the cooling to room temperature from the high deposition temperature. If the films are to be used on sensitive device structures, excessive ion bombardment has also to be avoided. Highfrequency plasmas provide efficient excitation in the gas phase combined with low-energy ion bombardment which can be beneficial for densification and/or desorption of products from the surface.

For these reasons, we investigate the deposition from a 27.12 MHz plasma. To elucidate the role of the oxygen-to-TEOS ratio, the influence of the deposition rate on the film properties at high OTR and a constant temperature is studied. The H-content and the breakdown voltage were measured. For comparison, we also study film deposition in the Ar/TEOS system.

Experimental

Reactor.—The reactor consists of a stainless steel double cross (inner diameter 63 mm). The substrate holder (2 cm diam) is introduced from the bottom end and the powered electrode from the top. The powered electrode is a cylinder, the height of which can be varied. The substrate holder is part of the large grounded area consisting of the upper leg of the double cross into which the powered electrode is placed and the substrate holder from below. A schematic view is shown in Fig. 1.

By retracting the powered electrode also thick samples, like the bolometersubstrate, can be put on the substrate holder while keeping the electrode spacing equal. However, in that case the plasma volume is smaller, and generally higher deposition rates can be obtained.

The substrate holder is a cylinder with a built-in resistance-heater element enabling deposition up to 400°C. A thermocouple is positioned close to the substrate holder surface from the inside. TEOS is introduced from a thermostated bath through heated tubes without carrier gas. The flow can be regulated by changing the bath temperature or by adjusting a needle valve. The bath temperature or by adjusting a needle valve. The bath temperature was 50-60°C. Oxygen or other gases can be introduced through a separate needle valve. The system was pumped by a two-stage rotary pump through a zeolite trap. The base pressure obtained was about 1 µbar. Gas pressure during processing was measured with a capacitance manometer and was kept at about 0.30 mbar. TEOS was introduced into the evacuated chamber and the pressure was regulated by a throttle valve. Thereafter the other gas (O₂

¹ Present address: Institute for Chemistry of Information Recording, Technical University Munich, D-8046 Garching/Munich, Germany.



Fig. 1. Schematic view of the deposition setup.

or Ar) was introduced and its flow regulated to obtain the desired total pressure. The oxygen-to-TEOS ratio (OTR) was always high (10 or more).

The flow rate of TEOS, as well as the total flow rate, were measured by measuring the rate of pressure increase after closing the valve between the chamber and trap. In the case of TEOS this measurement was done starting from the partial pressure of TEOS without oxygen. An estimate of the dwell time of the reactive species in the reactor can be then obtained by means of Eq. [1]. As the reactor is not designed to fulfill plug-flow conditions, this number can only be used qualitatively

$$\tau_{\rm dwell} = pV/q \qquad [1]$$

Here V is taken as the reactor volume, q is the flow in mbarl/s, and p the pressure in mbar.

The RF power was delivered through an L-type matching network which was located directly at the top of the chamber. Forward and reflected power were measured with a Bird Watt meter. The operating frequency was 27.12 MHz. Peak voltages at a power level of 5 W were typically 230 V as measured at the powered electrode with a high voltage probe. The dc selfbias was about 40 V.

Film diagnostics.—Films were deposited on $15 \times 15 \text{ mm}^2$ Si samples. The thickness of the films was measured by the weight increase of the substrate after deposition and by ellipsometry. Elastic recoil detection (ERD), protonenhanced scattering (PES), and infrared-absorption-spectrometry were used to determine their composition. The ERD was performed with 2.6 MeV ⁴He for the determination of the H-profile and total content by detection of the recoiled H. Analysis fluences were typically 2.4 1013 ions/ cm² (for more details see Ref. (14)). The ERD measurements are limited to film thicknesses smaller than $0.5 \ \mu\text{m}$. In that range of thickness they are very sensitive (1013 at/cm2), whereas the infrared spectra give accurate values (for low H-contents) for thicker films. The ERD energy spectrum was converted into a concentration profile using a density of the film of 2.2 g/cm³. The PES measurements for the determination of the total amount of carbon (15) were carried out with 1.735 MeV protons in the geometry as described in Ref. (16). At this energy the scattering cross section on carbon shows a resonance, allowing us to detect very small amounts of C. The detection limit for C was about 0.5%. The PES signal from carbon was calibrated with the aid of a-C:H films on Si (16). No depth profile was obtained.

The breakdown field strength was measured by determining the onset of current flow through a brass needle in contact with the substrate. The index of refraction was measured by ellipsometry.

Results

Oxygen-TEOS mixtures.—The deposition rate decreases with increasing temperature, as does the H-content. This is shown in Fig. 2 for OTR about 10. If expressed as an Ar-



Fig. 2. The dependence of the deposition rate in O2-TEOS on reciprocal temperature (•). The hydrogen content as measured by ERD is also shown (+).

rhenius plot, the effective activation energy in the temperature range from about 400 to 250°C amounts to -0.23 eV (-5.3 kcal/mol). Below 250°C a smaller value of about (-0.03 eV, -0.69 kcal/mol) is found, in agreement with previously published data (5, 10). This negative activation energy is indicative for a mechanism in which there is an exothermic equilibrium reaction before the ratedetermining step, for example, the adsorption of reactive species at the surface, which decreases with increasing temperature.

The deposition rate increases with increasing partical TEOS pressure and decreases with increasing dwell time (Fig. 3). Chin and van de Ven (5) have shown that at high OTR the addition of nitrogen has the same effect on the deposition rate as the addition of more oxygen, namely, dilution. Thus, we did not discriminate between experiments with different large OTR (in the range 10-20). At higher partial TEOS pressures the deposition rate increases with power; however, the decrease with increasing dwell time is steeper and shifted to lower dwell times, indicating rapid depletion. The deposition becomes inhomogeneous (in the flow direction), whereas it is homogeneous within few percent otherwise. By decreasing the size of the upper electrode and thus the plasma volume, higher rates could be obtained, showing the same trends.

The total pressure upon ignition of the plasma shows a fast increase followed by a decrease which can bring the pressure level to even under the starting point. The curve depends on the dwell time of the reagents. Examples are shown in Fig. 4a.



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Fig. 3. The dependence of the deposition rate in O₂-TEOS mixtures at 280°C on the dwell time for two partial pressures of TEOS. Total pressure: 0.28 mbar.



Fig. 4. The dependence of the total pressure on the time after ignition and extinction of the plasma. 10 W $p_{\text{TEOS}} = 0.025 \text{ mB}$, $p_{\text{total}} = 0.3 \text{ mB}$. (a) O_{2r} (b) Ar.

Deposition from Ar-TEOS mixtures.—The deposition without oxygen showed a completely different temperature behavior. Figure 5 shows this dependence. The rate in Ar is much lower than in oxygen and increases slightly with increasing temperature. The setup with a smaller plasma volume was used in this case. The rates in oxygen measured with the same TEOS partial pressure and power show the same trend as in Fig. 2 (although they were higher due to a smaller plasma volume used). An activation energy of about 0.08 eV (1.84 kcal/mol) was estimated for the deposition with Ar. Thermal decomposition of TEOS above 550°C shows an activation energy of about 2 eV (18). At the temperatures used the thermal decomposition is slow and can be neglected.

The pressure development upon ignition is shown in Fig. 4b. Now only an initial pressure rise is observed, as can be expected for the fragmentation of TEOS into smaller components.

Film analysis.—As seen in Fig. 2, the H-content closely follows the deposition rate as a function of temperature in the oxygen/TEOS system. We investigated also the relation between the deposition rate and the H-content at a constant deposition temperature.

An important finding is the increase of the H-content, measured immediately after deposition, if the deposition rate is increased by changing the dwell time and/or the partial TEOS pressure at constant temperature. Figure 6 shows this dependence for different temperatures in the oxygen/TEOS system. After the deposition, most films take up some water as can be followed in the IR spectra at 930 cm⁻¹ (Si-OH) and around 3300-3400 cm⁻¹ [H₂O or hydrogen-bonded OH (11)]. The films with lowest H-content (2 a/o) show no detectable absorption after deposition, 930 and 3660 cm⁻¹. After 4 months in air, about 3 a/o H mainly due to H₂O absorption were found in the films by ERD. The Si—O—Si stretching (1070 cm⁻¹) and bending



Fig. 5. Dependence of the deposition rate in Ar-TEOS on reciprocal temperature.



Fig. 6. The dependence of the hydrogen content on the deposition rate at constant temperature in the oxygen/TEOS system.

(810 cm⁻¹) agree in relative and absolute strength with results found in the literature. Generally, the H-content from H_2O and OH calculated from the IR absorption according to Pliskin (17) agree within 10% with the values from ERD. Some ERD profiles of films deposited in oxygen are shown in Fig. 7. No carbon could be detected by PES in films deposited in oxygen/TEOS mixtures.

The stoichiometry of the films as deduced from the IR spectra and from the refractive index (very near to 1.45) was Si/O = 0.5. For the films deposited in Ar, the carbon content as measured by PES was deposition temperature dependent. For a film deposited at 200°C it amounts to 8%, whereas at 280 and 380°C the amount is only 0.9%, although the deposition rate was higher. These PES spectra are shown in Fig. 8. The IR spectra of the films with around 1% C were very similar to those of films deposited with oxygen. Only a very weak absorption around 880 cm⁻¹ (possibly SIC) can be seen. No indications for large deviations (oxygen deficiency) from the ideal stoichiometry were found.

Breakdown field strength.—The breakdown field strengths of the deposited films are shown in Fig. 9 as a function of hydrogen content. The films deposited in Ar only are marked separately. Only films with thickness greater than 200 nm are shown in this figure. For films with a low hydrogen content the breakdown strength of thermally grown films is closely approached. The breakdown field strength of films deposited in Ar only fall on the same curve as the ones with oxygen. This dependence of the electrical properties mainly on the H/OH content has been noticed before (19).

Index of refraction, other substrates.—In both systems, we could not find deviations from the ideal stoichiometry by our analysis methods. Because the films take up some water and because of their slightly smaller index of refraction, we conclude that the films are slightly less dense than thermally grown films. The films deposited with only Ar



Fig. 7. Hydrogen content profiles (oxygen/TEOS) measured by ERD. The surface is at 0 Å. The dip is not statistically significant.



Fig. 8. Results (raw data) of the PES experiments: (a) Ar 250 nm thick, 200°C; (b) Ar 200 nm thick, 380°C. The number of detected scattered protons is shown as a function of energy (channel). Depending on the collision partner, a characteristic amount of energy is lost, the more, the lighter the collision partner.

are less dense than the films with oxygen. Immediately after deposition their index of refraction is about 1.44 ± 0.01 , whereas that of the films with oxygen is about 1.45 ± 0.01 . Upon water uptake the index of refraction increases slightly.

The films could be deposited on stainless steel without problems. No cracks were observed in more than 1 μ thick films. On bolometer foils the films adhere well; even on a foil of 5 μ m thickness a 1 μ m film could be deposited without causing noticeable bending or rolling of the foil, indicating low stress.

Discussion

The film analysis shows that good films in the sense of the desired insulating properties can be deposited from both Ar/TEOS and oxygen/TEOS mixtures. Very low Ccontent has been obtained in both oxygen/TEOS and in the oxygen-free Ar/TEOS system. Low H contents at relatively low deposition temperatures has been achieved also in the oxygen/TEOS system. However, the deposition characteristics are very different for both mixtures.

The different temperature dependence of the deposition rate in O_2 and in Ar, as shown in Fig. 2 and 5, reflects a difference in the rate-limiting step. Although the deposition from TEOS is an old process, the reaction steps are still subject to investigation. In recent times more fundamental studies, using various techniques such as ¹⁸O tracers



Fig. 9. Breakdown field strength dependence on hydrogen content. Only films thicker than 200 nm are shown.

(8, 10), adsorption/desorption (20), and the effects of ion bombardment (12b) have been published. It is generally agreed that the film forming process consists of more than one step after the adsorption of an activated TEOS*-like molecule (formed from TEOS by reaction with ozone, excited He, O, or directly in the plasma) at the surface (T_s) . In the presence of oxygen this primary adsorbed species reacts with oxygen (radicals) and then diffuses around to form the film by a so-called condensation reaction: $T'_s + T'_s$ $\rightarrow SiO_2 +$ products. Until now, only speculations are possible about the exact nature of these species.

Selamoglu et al. (11) states that this T'_s probably looks like Si-OH, based on the high Si-OH content of films deposited at low temperature in the TEOS-oxygen system. The rate-limiting step should be this condensation reaction which depends only on the surface concentration of T'_{s} . The dependence of the T'_{s} concentration on the temperature (desorption) provides the negative activation energy for the deposition rate. Pavelescu and Kleps (21) suggested that the negative activation energy is associated with a reaction of oxygen at the surface, forming T'_{s} . They attribute the negative activation energy to the adsorption desorption of oxygen or oxygen radicals. On the other hand, Pai and Chang (12a) speculate that the more negative the activation energy, the more TEOS-like the precursor. More TEOS-like precursors could have longer diffusion times at the surface, providing better step coverage. They suggest that in oxygen-lean mixtures, the film is formed by direct ion impact on T_s , thus giving directional deposition. SiO-like species formed in the gas phase or by flowing TEOS and O₂ both directly through the glow discharge, should give bad step coverage. In the experiment, Pai and Chang found a positive activation energy, which was said to be typical for gas-phase reactions. However, in general a thermally activated surface reaction can also account for a positive activation energy, like we found in the TEOS-Ar system.

Our data on the oxygen-TEOS system agree with the mechanism proposed in Ref. (8) and (10). The dependence of the H-content on the deposition rate (Fig. 6) supports that the elimination of OH takes place in the film forming reactions at the surface.

The different development of the pressure after ignition, shown in Fig. 5, can now be understood. Whereas the Ar/ TEOS mixture shows the expected pressure rise due to fragmentation and product formation, the pressure in the oxygen/TEOS (plus products) mixture can fall to a level even below the initial value of the oxygen pressure alone, depending on the dwell time. The reaction at the surface also consumes oxygen (molecules or radicals). Note that the walls of the reaction chamber are at about 60°C so that deposition there (in the oxygen case) is always faster than on the substrate holder. Fast disappearance of TEOS and oxygen radicals from the reaction mixture also explains the strong dwell time dependence of the deposition rate as seen from Fig. 3. The faster depletion at higher powers, giving rise to even lower deposition rates, can provide better films without being caused by ion-bombardment effects.

In the case of deposition with Ar only we propose that elimination reactions of TEOS* on the surface resulting in the formation of alcohols and C_2H_4 , see e.g., Ref. (21), or a reaction with a surface Si-OH (20) leads to SiO₂ deposition. These reactions are expected to display a positive activation energy. They can be enhanced by ion bombardment at lower temperatures in the case of plasma CVD, giving rise to directional deposition. The reaction products, such as alcohols and C_2H_4 , have to desorb. Therefore, the C content increases fast at lower deposition temperatures.

In the case of oxygen/TEOS mixtures, carbon, which might be incorporated into the film, is readily removed as CO and CO₂. We expect that at intermediate oxygen admixtures, the temperature dependence will change from the one with positive activation energy to the case with negative activation energy, depending on the amount of directly transformed T_s (possibly dependent on the ion bombardment) and reacting (with oxygen or oxygen radicals) T_s .

Conclusion

In this paper we have shown that in an oxygen/TEOS mixture good quality SiO₂ films can be obtained even at low temperatures if the deposition rate is kept low. Moreover we also obtained good films with a low C content of <1% without oxygen addition to the gaseous reactants. The deposition rate in oxygen/TEOS and Ar/TEOS mixtures shows a negative and positive effective activation energy, respectively, thus indicating different rate-limiting steps. The measured breakdown field increases with decreasing hydrogen content.

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Interface Characteristics of Metal-Oxide-Semiconductor Capacitors with Ultrathin Oxides

K. S. Chari¹ and S. Kar*

Department of Electrical Engineering and Advanced Center for Materials Science, Indian Institute of Technology, Kanpur-208016, India

ABSTRACT

MOS structures were fabricated on single-crystal as well as polycrystalline silicon. Oxidation was carried out in dry oxygen at 1000 and 1100°C. No annealing was performed either after oxidation or after metallization. The oxide thickness ranged between 35 and 490 Å. Admittance-voltage-frequency measurements were made on a large number of nonleaky structures to obtain the interface charge density at flatband and the interface state density vs. bandgap energy profile. As the oxide thickness increased, both the interface state density and the flatband interface charge density first decreased and then went through a maximum. The oxidation rate was also found to undergo a changeover at the same value of the oxide thickness. The observed correlation between the oxidation rate and the interface defects underscores the need to take into account the influence of the interface charge density and the built-in interfacial potential, while formulating a model for the oxidation kinetics of the initial phase.

To realize circuits with ultralarge scale integration, certain features will approach submicron dimensions. For the metal-oxide-semiconductor (MOS) field effect transistor (FET), decreasing channel length requires a smaller drain voltage and therefore a smaller threshold gate voltage. The threshold voltage, $V_{\rm T}$, is determined by the following parameters in the case of an n-channel at zero drain-to-source voltage

$$V_{\rm T} = \left[(\Phi_{\rm M} - \chi_{\rm Si})/q \right] - \Phi_{\rm p} + \left[(Q_{\rm scinv} + Q_{\rm itinv} + Q_{\rm F})/t_{\rm ox}/\epsilon_{\rm ox} \right] \qquad [1]$$

 Φ_M is the work-function of the gate metal, χ_{Si} is the electron affinity of silicon, Φ_p is the bulk Fermi potential ($\Phi_p = (E_F - E_v)/q$, where E_F is the Fermi level and E_v is the valence bandedge in bulk silicon), Q_{scinv} is the silicon space charge density and Q_{itinv} the interface state charge density at the onset of strong inversion, Q_F the fixed charge density, t_{ox} the oxide thickness, and ϵ_{ox} the oxide permittivity.

Previous workers have scaled down the oxide thickness $t_{\rm ox}$ to reduce $V_{\rm T}$. The assumptions underlying this approach have been that the fixed charge density, $Q_{\rm F}$, and the interface state density distribution, $D_{it}(E)$, are invariant of the oxide thickness. However, when the oxide thickness is reduced to a value less than, say, 200 Å, these assumptions are not valid. It is generally assumed that the positive fixed charge, $Q_{\rm F}$, is spread over several monolayers in the oxide, extending up to tens of Å from the silicon/silicon dioxide interface (1). If that is the case, it would be expected that $Q_{\rm F}$ will increase with increasing oxide thickness in this thin regime.

Second, sources of the interface states are likely to undergo a transition during the initial oxidation phase, before a steady state is reached. For example, the density of dangling bonds will go down as the silicon surface is covered by the initial monolayers of the oxide. Similarly, strain/stress-induced defects and defects such as stacking faults are expected to appear at the interface after a critical oxide thickness is reached. These factors are likely to lead to a dependence of $D_{it}(E)$ on the initial oxide thickness.

Electrochemical Society Active Member.

¹ Present address: Microelectronics Development Division, Department of Electronics, 1 Eastern Avenue, Maharani Bagh, New Delhi-110065, India.