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Growth and Properties of LPCVD Titanium Nitride as a Diffusion Barrier for Silicon Device Technology

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ABSTRACT

Chemical vapor deposition has been used to deposit titanium nitride (TiN) on silicon wafers at low pressures in a cold-wall single-wafer reactor. Experiments are reported for pressures in the range of 100-300 mtorr and temperatures between 450°-700°C, with titanium tetrachloride and ammonia as reactants. Both hydrogen and nitrogen are evaluated as diluents. Deposition rates as high as 1000 Å/min have been achieved. The chemical nature of the films are evaluated by Auger and RBS techniques, while the morphology is depicted by SEM. For the most part, the films are stoichiometric and contain small quantities of oxygen, chlorine, and hydrogen. Film resistivities as low as 50 μΩ-cm are reported. Behavior of the TiN film as a diffusion barrier between silicon (boron doped) and aluminum, at annealing temperatures up to 550°C, is evaluated by measurements of contact resistance and diode leakage.

Coating of various metals with titanium nitride has been possible since the discovery in the 1920s of a chemical vapor deposition technique using electrically heated wires exposed to $TiCl_4 + N_2 + H_2$ gas mixtures (1, 2). In spite of the high temperatures required (>1000°C), commercial applications for jewelry coating (3) (gold color) and tool coating (4) (wear resistance) have seen considerable success. With the advent of reactive physical vapor deposition techniques (5-8), where a discharge in nitrogen is created with titanium atoms either evaporated or sputtered from one electrode, low temperature depositions (<500°C) became possible and applications of TiN coatings have become even more widespread. Other applications have been as a transparent heat mirror for architectural windows (9, 10), and as a high temperature diffusion barrier for silicon solar cells (11, 12).

Of particular interest, in the present paper, is the application of thin TiN films to silicon device technology. Because of the concern about exposure of circuits to high temperature processes, barrier films deposited by physical vapor techniques such as low temperature reactive sputtering (13-22) have been employed. However, as integrated circuit feature sizes shrink to submicron dimensions, the ability of physical techniques to uniformly coat high aspect ratio vias has become a concern. Accordingly, there has been an interest in developing a low temperature CVD process for deposition of thin films of TiN. One approach has been to explore plasma-enhanced CVD (PECVD) using $TiCl_4$ plus either nitrogen (23-26) or ammonia (27, 28). Another has been to investigate the low temperature thermal CVD process possible using $TiCl_4 + NH_3$, where depositions are possible at temperatures as low as 450°C. One recent investigation has focused on a low pressure CVD process (LPCVD) where many wafers are coated at one time in a hot wall tube reactor (29). Two others describe a LPCVD process in a cold-wall single-wafer reactor (30, 31). In this paper we elaborate on the latter approach.

Experimental

The titanium nitride depositions were carried out in a single-wafer cold-wall experimental reactor shown schematically in Fig. 1 (30). The chamber was stainless steel and enclosed a water and air-cooled lamp assembly used to heat the wafer. A single 10 kW tungsten halogen lamp was employed that can heat a 4 in. wafer to 700°C rapidly. A load lock was used to maintain an oxygen and water vapor-free deposition environment.

Reactive gases were introduced through a showerhead gas distributor several inches above the wafer. The $TiCl_4$ and NH_3 were introduced through two separate lines and mixed for the first time in the shower head. As long as these two gases are mixed close to the hot wafer on which the deposit is to be made, the formation of NH_4Cl can be avoided as has been demonstrated in another experiment similar to this one (31).

Before beginning $TiCl_4$ or NH_3 flows, a nitrogen flow was established in the $TiCl_4$ line downstream of the $TiCl_4$ bottle. Next the $TiCl_4$ flow was initiated, and after it was stabilized then the NH_3 flow was introduced. This procedure kept $TiCl_4$ and NH_3 from mixing in the $TiCl_4$ line and causing solid deposits there.

All of the gases were introduced through mass flow controllers (MFCs), including the $TiCl_4$ vapor which was obtained by heating $TiCl_4$ liquid to 40°C. The $TiCl_4$ line was heated to 50°C to prevent any condensation of this vapor. Since the pressure of the $TiCl_4$ vapor at 40°C was only 50 torr, we calibrated our Unit MFC (UFC 1100) by measuring the time it took for a specified nitrogen mass flow (supplied at 40 torr) to increase the pressure in our chamber from 100 to 200 mtorr. We then repeated this measurement when the nitrogen was supplied at 10 psi. By this method

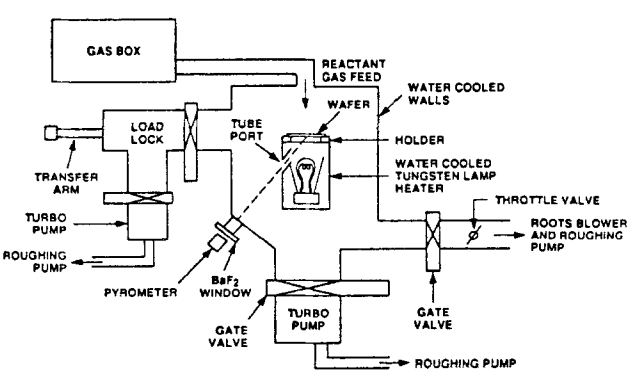

Fig. 1. Schematic of CVD reactor system

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we established that when our MFC was set at 50% we were realizing a flow of 42% if our supply pressure was only 40 torr.

Since it was felt that any native oxide on the silicon surface could influence the contact between the deposited titanium nitride and the substrate, a dry cleaning procedure was introduced. Each wafer prior to being loaded into the load lock was exposed to ozone generated by an array of mercury discharge lamps (32), in order to remove any carbon that might be on the surface. As has been shown (33), residual carbon on the wafer surface makes it much more difficult to remove oxide. Then after the wafer was placed in the chamber and before it was heated, it was exposed to hydrogen atoms to reduce the silicon oxide. Auger measurements of the titanium nitride film verified that there was no oxide peak at the interface.

Wafer temperature was measured with a Linear Labs TM1000 pyrometer sensitive to long wavelength infrared radiation (4-10 μm) in order to permit measurement of temperatures over the 200°-1000°C range. The pyrometer was set up to view the back of a wafer through a barium fluoride window in the reactor chamber. Calibration was done with a thick silicon wafer (1/4 in.) which had a radial hole drilled to its center (0.100 in.) into which a thermocouple was placed. Power to the lamp was controlled by a thyrister power control unit that was regulated by a Eurotherm 808 controller.

Turbopumps were used to evacuate the load lock and the main chamber. After bakeout, the base pressure in the main chamber was 3 × 10E-08 torr. There was no detectable oxygen and the water vapor partial pressure was no more than 1.5 × 10E-09 torr. During processing reactive gases were pumped by a Roots blower with a butterfly valve used to control pressure in the chamber. Process pressure was measured with a capacitance manometer and its output was used to set the butterfly valve.

Since it took about 30s to 1 min for a wafer to heat up to its deposition temperature, and a similar time period was necessary for the $TiCl_4$ flow to be stabilized, a specific sequence of events was followed for each deposition. At first, wafer heating was begun under vacuum and then nitrogen flow was started in the $TiCl_4$ manifold. Then, when the wafer was at temperature, the $TiCl_4$ flow was begun and monitored with a mass spectrometer. As soon as the flow was stabilized, the NH_3 flow was initiated and deposition begun.

After each deposition, crystalline growths always appeared on the wafer surface after the coated wafer was exposed to room air for more than 15 min. A typical crystalline growth approximately 25 μm across is shown in Fig. 2, and it contains some titanium, oxygen, chlorine, and nitrogen. A deionized water rinse immediately after each deposition removed whatever surface layer was causing these growths, and they did not reappear. Apparently, they appeared as a result of interaction of a surface layer remaining on the film with room air moisture. Since no *in situ* diagnostics were available, it was not possible to evaluate this surface film.

In the course of the present study, deposition characteristics were evaluated by depositing blanket films on 100 mm silicon wafers. Sheet resistance was measured with a Prometrix four-point probe. Deposition rates were determined by etching portions of a film away and then using a Dektak profilometer to measure step height. Knowing the film thickness, resistivity was also calculated. Chemical evaluation of these films was carried out with a combination of Auger spectroscopy and Rutherford back scattering (RBS). The latter provided accurate values of the Ti/N ratios, but we could not determine the oxygen or carbon concentrations by this technique. Therefore, oxygen and carbon contents were measured by Auger. The chlorine level was determined by both methods.

The fact that hydrogen existed in these films was pointed out many years ago (34). Since neither Auger nor RBS can detect hydrogen, we used a forward scattering technique originally developed at Lockheed Research Laboratories (35) that is being further refined at Charles Evans Laboratories. In this procedure, helium ions accelerated to 2.275 MeV impact a thin film on a substrate at a grazing angle of 15° and forward scatter hydrogen atoms out of the film. A detector positioned downstream at 15° from the sample surface can intercept the helium and hydrogen beam. A thin aluminum foil is placed over the detector to filter out the helium atoms so that only the hydrogen is detected.

The film densities were measured by using a microbalance to weigh a small piece of coated substrate (approximately 1 cm^2) before and after the titanium nitride was etched off.

Contact resistance and diode leakage studies were carried out on patterned wafers with p^+ (boron) doping. For contact to these diffusions, n-doped wafers were thermally oxidized to a thickness of 1 μm and then wet etched to open contact holes to the silicon. After this, boron ions were implanted at 30 keV and a dose of 5 × 10E15/cm^2. Implant activation was carried out at 1000°C for 30 min in dry oxygen. This produced a number density of activated boron atoms near the surface of 5.5 × 10E19/cm^2. Next 1 μm of PSG (phosphosilicate glass) was deposited and reactive ion etched to define the contact holes to the diffused silicon. Finally, a 500Å CVD TiN layer was deposited, and then 1 μm of aluminum (1% silicon) was sputtered onto the wafer and wet etched to complete the pattern delineation.

Contact resistance was measured using cross bridge Kelvin structures on the patterned wafers. Contact holes were 5 μm in diameter and diffusion lines were 15 μm wide. Diode integrity was evaluated by reverse biasing large diodes (95 by 395 μm) with either few [7], or many [702] 5 μm diam contacts, and measuring their leakage current.

For all of the measurements to be reported, the data points presented represent all of the data that was taken. Sufficient data were not available to permit evaluation of error levels in the measured values.

Results and Discussion

Film chemistry.—The titanium nitride films deposited exhibited a ~60Å thick oxide on the surface [oxygen ~ 15 atomic percent a/o)], but were otherwise uniform chemi-

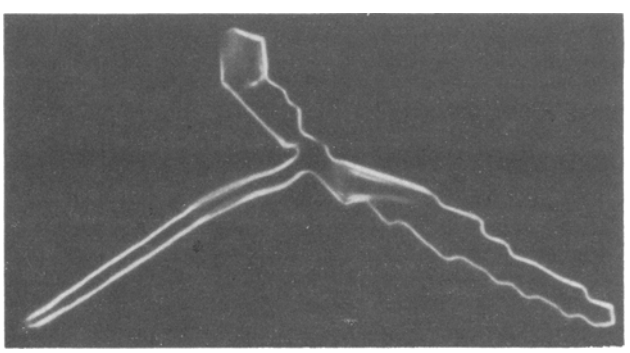

Fig. 2. Growth on surface of TiN-coated wafer

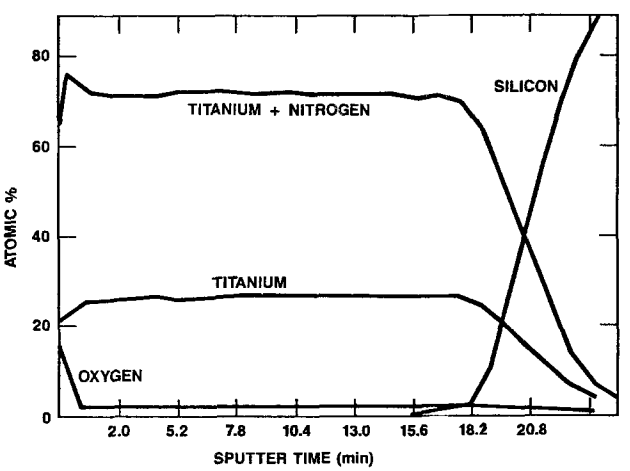

Fig. 3. Auger depth profile

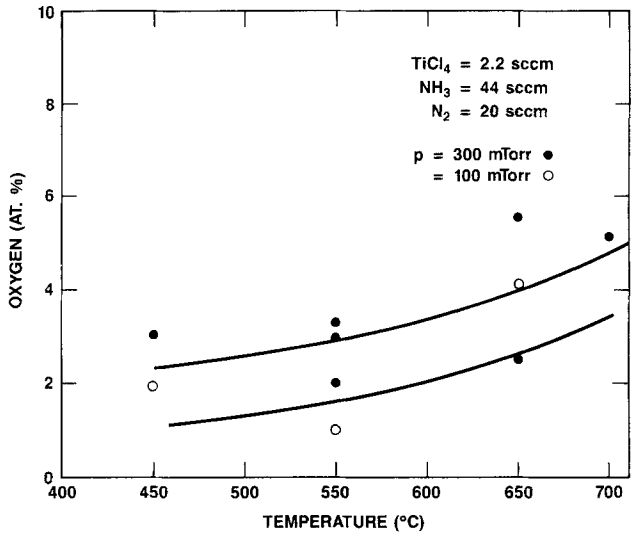

Fig. 4. Oxygen *vs.* *p* and *T*

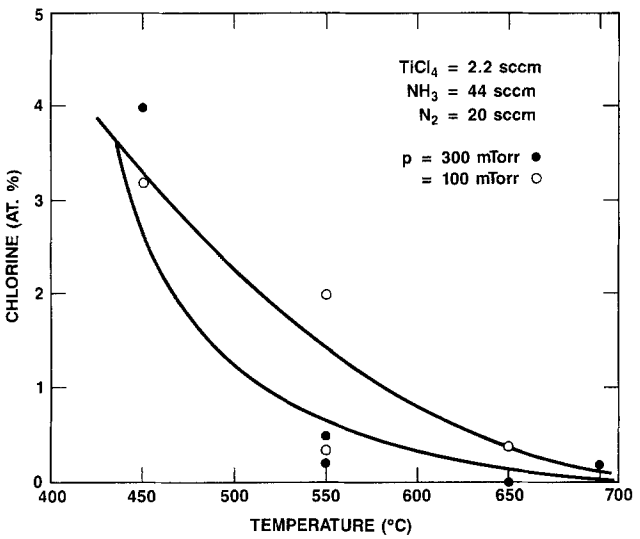

Fig. 5. Chlorine *vs.* *p* and *T*

cally. The lack of a measurable oxide layer at the TiN/Si interface is illustrated in the Auger depth profile presented in Fig. 3. Depending on the reacting gas mixture and deposition conditions, there are found varying amounts of oxygen, chlorine, and hydrogen in these films and their stoichiometry can change (TiN_x where $x < 1$ or $x > 1$). The behavior of the oxygen, chlorine, and hydrogen concentrations for different pressures and temperatures are shown in Fig. 4-6. The oxygen concentrations were observed to increase as temperature was increased, as well as with higher chamber pressures. However, they are comparable to the levels typically found in reactively sputtered films (17), and values as low as 1-2% are feasible. The chlorine content clearly is reduced at the higher deposition temperatures (<0.5% at $T > 600°$-650°C), but the trend with pressure is not clear. It is interesting to observe that our chlorine concentrations are substantially lower than other recently reported measurements [7.6% at 300 mtorr and 500°C (31), 50% at 1 atm and 450°C (10), 15% at 150 mtorr and 500°C (29)]. The hydrogen content of the films increases with temperature. Values over 10% are seen in the films deposited at the highest temperatures. Increasing the deposition pressure appears to lower the hydrogen concentration. Finally, the Ti/N ratio was close to one except for the 450°C film. At this lowest temperature we observed a nitrogen-rich film with Ti/N ranging from 0.84-0.92.

In addition to the flow conditions described in Fig. 4-6, additional experiments were done for different mass flows including the addition of hydrogen as a diluent. No clear trend, as far as chemical composition of these films ap-

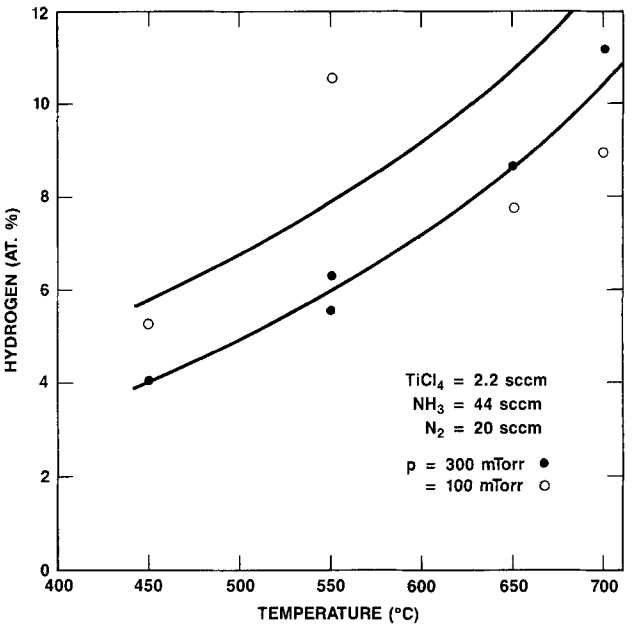

Fig. 6. Hydrogen *vs.* *p* and *T*

peared, other than a tendency to form titanium rich films when the $NH_3/TiCl_4$ ratio was reduced from 20:1 to 10:1. For example, we obtained a value of Ti/N = 1.4 for the following conditions

Flow of NH_3	= 16.5 sccm	p = 100 mtorr
Flow of $TiCl_4$	= 1.65 sccm	T = 550°C
Flow of N_2	= 20 sccm	

This last titanium-rich film had a decidedly silver-gold color, but it was the exception. All the other films were a typically gold color as long as they were thinner than ~800Å. The thicker films (1000-2000Å) were a dark rose-gold color, although there was no change in their chemical composition. On the other hand, of the films deposited in the atmospheric pressure cold-wall reactor (10) none were reported to have a gold color.

Physical characteristics of film.—The physical nature of the films was examined by SEM. In every case they appeared to be crystalline with columnar grain structure. As can be seen in Fig. 7, for films <1000Å, the crystal diameters are ~500Å.

As noted earlier, a key issue is how conformally will these films cover very small (<1 μm diam) and very deep (>1 μm) contact holes. Accordingly, we patterned some PSG-coated wafers with contact lithography and dry-etched contact holes. In Fig. 8 we show a 2 μm diam contact hole approximately 1 μm deep, and it appears to have

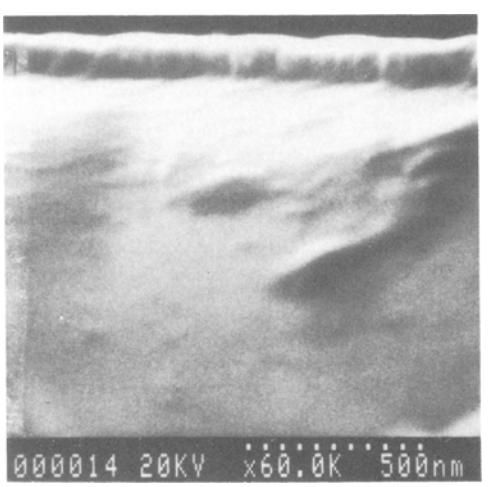

Fig. 7. SEM of TiN film

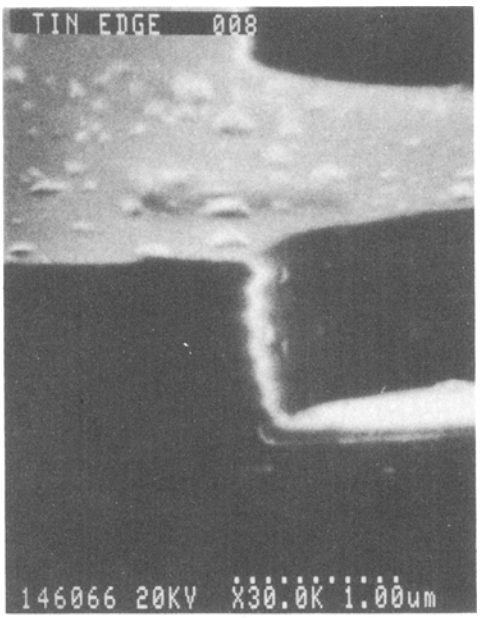

Fig. 8. SEM showing conformality of coverage

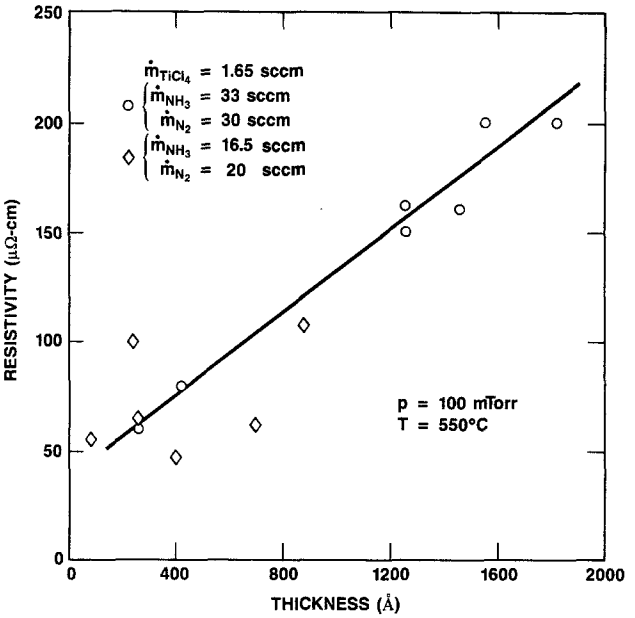

Fig. 10. Resistivity vs. film thickness

a very conformal coating. Excellent coverage on even smaller geometries has recently been reported (29, 31).

In order to evaluate quantitatively film stress, it would be necessary to deposit a film of uniform thickness over an entire unpatterned 4 in. wafer. Unfortunately, with the present lamp heater design the films were only uniform over the central 2 in. diam of the wafer, so that no measurements of film stress were possible. However, all of the films were very adherent to both silicon and silicon dioxide and could not be removed by the scotch tape test. Therefore, for the thin films studied here (<2000Å) adherence of the films does not appear to be a limitation.

Film resistivity.—In general, the resistivity of low temperature CVD TiN thin films has been reported to be quite high compared to bulk material (18 μΩ-cm) as well as films prepared by reactive sputtering with substantial ion bombardment (36 μΩ-cm at 100V bias). Atmospheric pressure CVD films (10) were reported to have values on the order of 300 μΩ-cm when deposited at 650°C, and a recent low pressure study (35) gave a value of 525 μm-cm for a 500°C process. The lowest value, 47 μΩ-cm, has been reported for a low-pressure process (29) operated at 750°C. In the present study, measurements of resistivity have been made over a range of pressures, temperatures, and reactant mass flows. Two effects have been observed. One, as shown in Fig. 9, is that the higher the temperature and pressure the lower the resistivity. Two, the thicker the film the higher the resistivity. To illustrate this phenomena, we show in Fig. 10 how the measured resistivity changes with film thickness for two separate flow conditions. Within the accuracy available, the chemical composition of all of these films were the same, so this could not explain the measured results. Careful determination of the film density did show a systematic lowering of this parameter as the films grew thicker (Fig. 11), and this apparently is the cause of the lowered resistivity. Presumably, this could also explain the change in color as the films grow thicker. A similar phenomena has been described for reactively sputtered TiN films (17).

Deposition rates.—In the single-wafer reactor format being studied here, high deposition rates are necessary to enable acceptable wafer through puts. Compared to deposition rates of 30-65 Å/min obtained in a hot-wall low-pressure system (29), we have observed rates as high as 1000Å/min. In addition, we have found that there is an "incubation" time before noticeable deposition begins. As shown in Fig. 12, at 100 mtorr deposition pressure this incubation time can be over 1 min. Similar phenomena have been known for the deposition of tungsten from tungsten

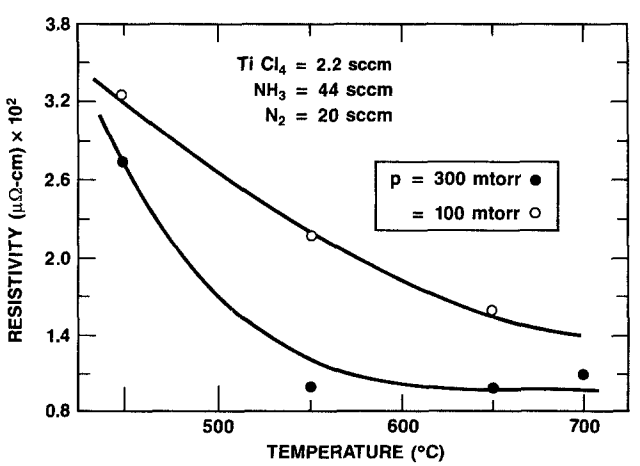

Fig. 9. Resistivity vs. p and T for 800A films

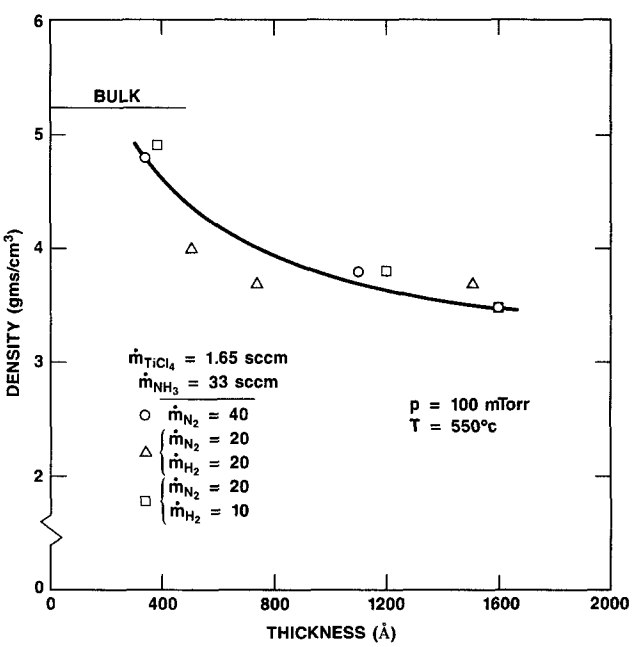

Fig. 11. Film density vs. film thickness

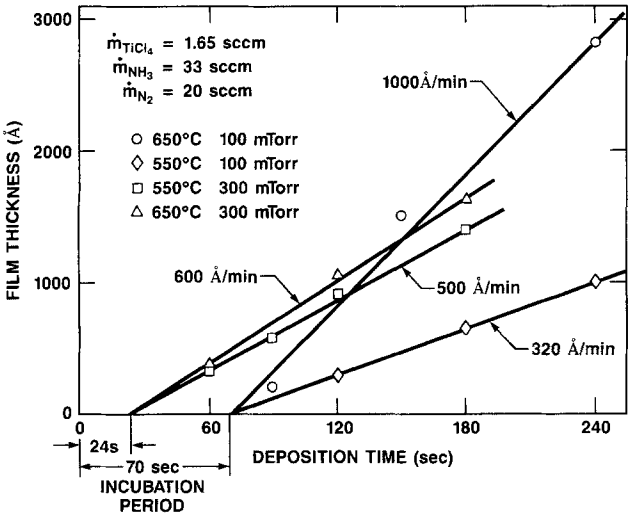

Fig. 12. Deposition rates and incubation time

hexafluoride and hydrogen, depending on the type of surface onto which the deposition is being done (36). Presumably, the incubation time phenomenon occurs in our experiments because our reaction is not very well catalyzed by a silicon surface. In fact, depositions on wafers that had an initial TiN film (sputtered) did not exhibit any incubation time.

As can be seen, the lower pressure deposition is very much surface controlled and an increase of temperature of 100°C causes an almost 300% increase in deposition rate. The higher pressure case appears to be more diffusion controlled so a similar 100°C temperature rise only causes a 20% rise in rate. However, because of the shorter incubation period, at 550°C and 300 mtorr a 1000Å film can be deposited in 2 min, but would take 4 min at 100 mtorr. When the pressure and temperature as well as the flows of the reactive gases ($TiCl_4$, NH_3) were held constant it was found that doubling the N_2 flow increased the deposition rate from 320 to 500Å/min. Finally, reducing the flow rate of NH_3 by a factor of two seemed to increase the length of the incubation time by the same factor.

Diffusion barrier behavior.—A good diffusion barrier must first of all prevent intermixing of aluminum and silicon even at elevated temperatures. Second, it would be desirable if it also exhibited a low contact resistance.

Experience has shown that good ohmic contacts are hardest to achieve on p^+ doped silicon. Measurements were therefore made of contact resistance to p^+ doped substrates. After annealing a wafer with Cross bridge Kelvin resistor patterns and Si(p^+)/TiN/Al contacts for 30 min in forming gas (10% H_2 in N_2) at 500°C, we measured 13Ω on the 5 μm diam contacts. Taking into account an accurate model of the Cross bridge resistor (37), this value converts into a contact resistivity of 2.4 × 10E-06 Ω-cm². In general, reactively sputtered TiN films between Si and Al are reported to give very high contact resistance values (19) (*i.e.*, 1-3 × 10E-05 Ω-cm²). The present measurement, however, is consistent with reported values of 2 × 10E-07—1 × 10E-06 Ω-cm² for p^+ doped contacts with 2 × 10E20/cm³ active boron atom concentration (38), if we recognize the exponential dependence of contact resistance on doping level (39). The very high contact resistance normally observed for these films may be due to a native oxide layer between the TiN and Si which we have avoided by our *in situ* cleaning procedure.

A measure of the quality of the diffusion barrier behavior of the TiN films was obtained by examining diode integrity when reverse biased at 10V. If it exhibited less than 1 μA leakage it was considered acceptable. All the diodes that were annealed at 450° and 500°C passed this test. None of the diodes that were annealed at 550°C survived. The degree of interaction between the aluminum and silicon was evaluated by stripping of the aluminum, TiN, and oxide layers. An SEM photograph of a contact annealed at 500°C

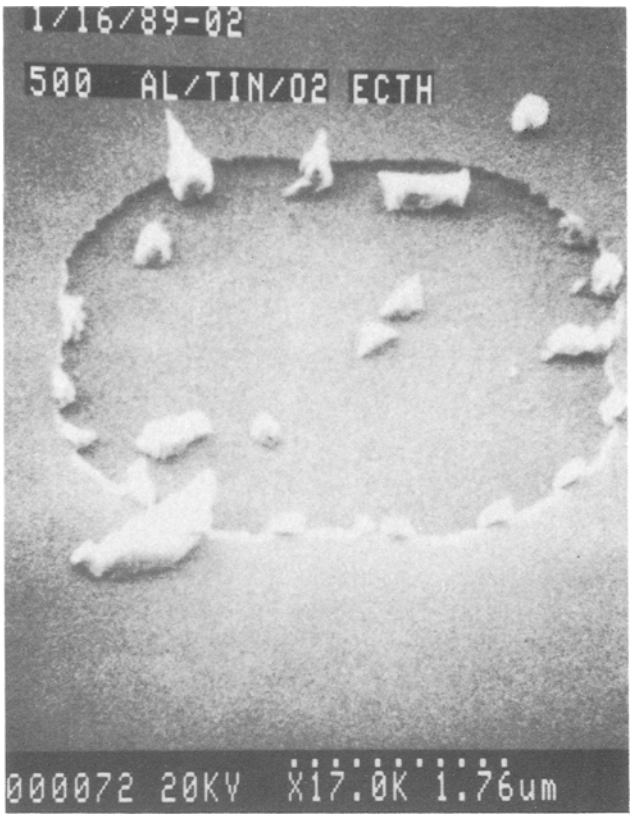

Fig. 13. SEM of silicon TiN contact surface after annealing at 500°C for 30 min.

is shown in Fig. 13, where the nodules remaining on the surface are simply silicon left over from the 1% silicon doping of the aluminum film. No evidence of attack is seen. When annealed at 550°C, however, the contact is completely consumed as shown in Fig. 14.

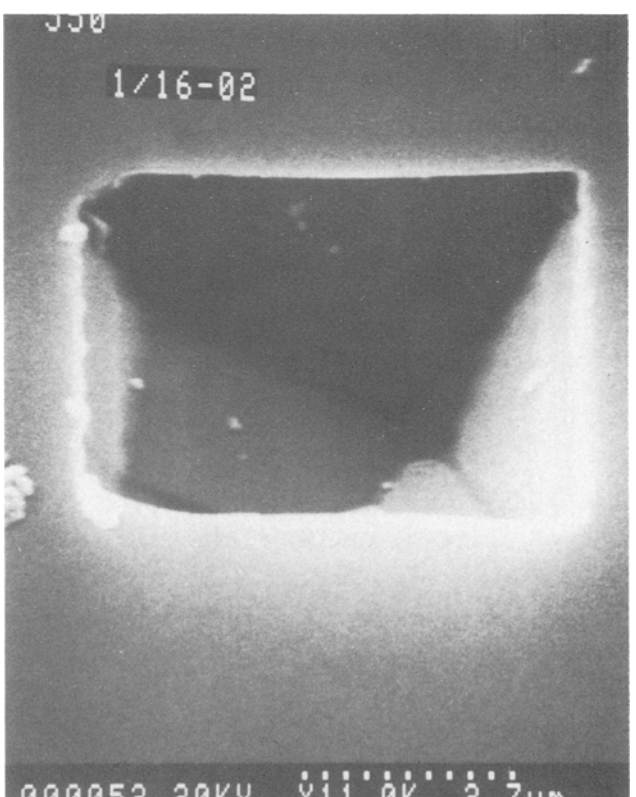

Fig. 14. SEM of silicon TiN contact surface after annealing at 550°C for 30 min.

Summary and Conclusions

The feasibility of depositing a thin film of TiN in a low temperature-low pressure cold-wall single-wafer reactor has been demonstrated. The chemical nature of the film has been explored, and we conclude that the films are largely stoichiometric and contain small quantities of oxygen, chlorine, and hydrogen. Physically, the films are crystalline (columnar crystals), conformal, and adherent. Their resistivity ranges from 50-320 μΩ-cm, with the lowest values being obtained for the thinnest (~500Å) films. High deposition rates are observed (~1000Å/min), and there is an incubation period before deposition begins.

The suitability of these films as diffusion barriers between silicon and aluminum has been examined briefly for p^+ (boron) on n diodes. First indications are that diodes annealed at 500°C yield contact resistivities of ~2.4 × 10E-06 Ω-cm^2 and exhibit less than 1 μA leakage current when biased 10V.

Acknowledgments

The author would like to thank B. Cooper, G. Reynolds, M. Salimian, P. Gaczi, and D. Hodul for general discussions, S. Felch for help in preparing the patterned wafers, and J. Hayes for his assistance with the electrical measurements. He would also like to acknowledge the assistance of H. Gilliland and C. Holloway in fabricating and assembling the reactor system, and for help in gathering and evaluating the data.

Manuscript submitted Aug. 31, 1989; revised manuscript received Dec. 11, 1989.

Varian Research Center assisted in meeting the publication costs of this article.

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