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LOW TEMPERATURE EPITAXIAL DEPOSITION

OF SILICON BY PLASMA ENHANCED CVD

FINAL TECHNICAL REPORT

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L. Rafael Reif

June 1, 1984

U. S. ARNY RESEARCH OFFICE

Contract Number: DAAG29-81-K-0087

Massachusetts Institute of Technology

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LOW-TEMPERATURE SILICON EPITAXIAL DEPOSITION OF SILICON BY PLASMA ENHANCED CVD

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A reactor system has been developed to deposit specular epitaxial silicon films at temperatures as low as 650°C using a low pressure chemical vapor deposition process both with and without plasma enhancement. This represents the lowest silicon epitaxial deposition temperature ever reported for a thermally driven chemical vapor deposition process. Experiments performed at 775°C indicate that the predeposition in-situ cleaning of the substrate surface is the critical step in determining whether epitaxial deposition will occur. Surface cleaning in these experiments was done by sputtering in an argon plasma ambient at the deposition temperature while applying a dc bias to the susceptor. This is the lowest pre-epitaxial cleaning temperature ever reported for a thermally driven chemical vapor deposition.

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LOW TEMPERATURE EPITAXIAL DEPOSITION OF SILICON BY PLASMA ENHANCED CVD

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The development of low-temperature processes for fabricating VLSI chips has become essential to the continuing development of smaller, faster solidstate devices and circuits. Of the low temperature processes that have been reported in the literature, such as Molecular Beam Epitaxy (MBE), Ion Beam Epitaxy (IBE), and Plasma Enhanced Chemical Vapor Deposition (PECVD), only PECVD appears to be compatible with manufacturing requirements such as high throughput and conformal coverage. The most promising work to date has been the work of Suzuki [1], who has reported depositing good quality epitaxial silicon films at temperatures as low as 750°C. Suzuki and others have surmised that reactive radicals in the plasma and/or in-situ surface cleaning by the plasma make it possible for the PECVD process to achieve low-temperature epitaxy. However, no definitive experiments have been performed to determine the relative importance of the various parameters that contribute to the deposition process in a PECVD system.

We have developed a low pressure chemical vapor deposition process using silane capable of depositing uniform, specular epitaxial films on 2-in. silicon substrates at temperatures as low as 650°C, both with and without plasma enhancement during the deposition [2]. In both cases, it was found that the pre-deposition in-situ argon plasma sputter cleaning was the critical factor in determining whether or not the ensuing deposition would

be epitaxial, and was also important in determining the quality of the deposited film. We have thus shown that it is possible to deposit good quality epitaxial silicon at low temperatures using a low pressure chemical vapor deposition process without plasma (and consequently without plasma radicals and ions) if the surface is properly cleaned in-situ prior to the deposition. The presence of a plasma during the deposition had little influence upon the surface quality of the resulting deposition, if the predeposition cleaning was performed properly, but it had a significant effect upon the deposition rate at the lower deposition temperatures.

The deposition system is a vertically aligned, radiantly heated reactor[2]. The wafer lays on a silicon-carbide coated graphite susceptor, facing the radiant heater. An infrared pyrometer monitors the wafer temperature, with a feedback loop controlling the lamp power. The chamber consists of a quartz tube sealed to stainless steel end plates by silicon gaskets. Research purity gases are fed into the deposition system through mass flow controllers. The rf plasma is generated by a 13.56 MHz generator, the output of which passes through a matching network prior to coupling to the gas. The rf electrodes consist of a copper electrode wrapped around the exterior of the upper 1/3 of the quartz chamber, with a silicon coated stainless steel false-bottom serving as the other rf electrode. For the deposition conditions used, this arrangement generates a very uniform plasma within the chamber. A dc voltage source is connected through an rf filter to the susceptor and the false bottom in such a way that positive ions generated within the plasma are accelerated towards the susceptor and the substrate. The chamber is pumped on by a 510-1/s turbomolecular pump backed by an 11cfm mechanical pump. The system base pressure is less than 10^{-7} torr.

Before doing a deposition sequence, the chamber and its contents are coated with silicon using a silane plasma. This provides a uniform silicon ambient for the subsequent depositions, and prevents large temperature fluctuations from occuring as the quartz in front of the radiant heater first coats. The wafer is cleaned using a standard organic/ionic cleaning process, the chamber is backfilled with nitrogen, the previous run is unloaded, the wafer is given a final dip in a 15:1 HF solution, the wafer is loaded, and the chamber is pumped down. When the pressure within the chamber reaches 10^{-7} torr, the chamber is cyclically purged with argon, the lamps are turned on to bake out the chamber rand stabilize the deposition temperature, and the chamber is once again cyclically purged with argon. The wafer is then sputter cleaned for five minutes with an argon plasma of 50 W at a pressure of 5 mtorr, while applying a 300 V dc bias to the susceptor with respect to the internal rf electrode. At the conclusion of the argon sputter, the rf power is either turned off or reduced to 20 W, the silane flow is established (14 sccm, about 14 mtorr). If a dc bias is to be applied to the susceptor during a plasma deposition, the applied potential remained at 300 V. Otherwise, the dc bias line is open-circuited, allowing the susceptor to electrically float. The films were analyzed using x-ray and transmission electron diffraction, as well as optical and scanning electron microscopy, SIMS, and spreading resistance analysis.

The most critical stage of the process is the 5-min in-situ cleaning in the argon plasma. Experiments performed at 775°C indicate that if either the dc bias during the argon plasma was removed or if the argon plasma was eliminated altogether, then only polycrystalline films are deposited. With only 100 or 200 V of applied dc bias we were able to get hazy epitaxial

films with rough surfaces, but only if the plasma was used during the deposition. With a 300 V bias applied during the sputter cleaning, smooth, specular epitaxial films are deposited with or without plasma during the deposition. With the 300 V bias applied during the plasma deposition, the same result was obtained. The thickness of the films deposited at $775^{\circ}C$ ranged from 1 to 2 um as determined by weight, and were uniform to within 5% as determined by depositing on an oxidized wafer and using surface profilometry. The growth rates were 350 A/min (over three wafers) without the plasma, and 450 A/min (over four wafers) with the plasma.

To further investigate this difference in growth rates, and to attempt to determine the minimum epitaxial temperature using these deposition conditions, depositions were performed at 650° C (we have been unable to obtain epitaxial deposits at lower temperatures). At 650° C the growth rate without the plasma was about 22 A/min, while the growth rate with the plasma was about 180 A/min. This illustrates the ability of the plasma to enhance the deposition rate from what is possibly by strictly thermal decomposition. The plasma deposition rate is less sensitive than the strictly thermal deposition rate to the deposition temperature: the activation energy for the plasma deposition is about 14 kcal/mole, while that for the non-plasma depositions is about 41 kcal/mole.

Spreading resistance data indicates that the film intrinsic resistivity is in the 10 to 40 ohm-cm range, while SIMS analysis indicates that the major impurities in the film are oxygen and carbon.

To summarize, we have deposited specular epitaxial films at temperatures as low as 650°C using low pressure chemical vapor deposition both with and without plasma enhancement. This is the lowest silicon epitaxial depos-

ition temperature and the lowest pre-epitaxial cleaning temperature ever reported for a thermally driven CVD process. Furthermore, we have demonstrated that the predeposition cleaning rather than the plasma effects during the deposition is essential to obtain epitaxial silicon growth at these low temperatures.

References

- [1] S. Suzuki and T. Itoh, J. Appl. Phys., 54, 1466 (1983).
- [2] T. J. Donahue, W. R. Burger, and R. Reif, Appl. Phys. Lett., 44, 346 (1984).

PUBLICATIONS

- R. Reif, "Phosphorus Incorporation During Silicon Epitaxial Growth in a CVD Reactor", J. Electrochem. Soc., <u>129</u>, pp. 1122-1128, May 1982.
- T. J. Donahue, R. Reif and W. R. Burger, "Plasma-Assisted Chemical Vapor Deposition of Crystalline Silicon", <u>Electrochem, Soc. Spring</u> <u>Meeting</u>, Abs. 217, Montreal, Canada, p. 350, May 9-14, 1982.
- W. R. Burger, T. J. Donahue and R. Reif, "Plasma Assisted CVD of Thin Polysilicon Films", <u>Proceedings of the First International Symposium on</u> <u>VLSI Science and Technology</u>, The Electrochemical Society, Proceedings Vol. 82-7, pp. 87-93, 1982. Shorter version in <u>The Electrochemical</u> <u>Society Extended Abstracts</u>, Fall Meeting, Detroit, MI, Abs. 149, pp. 235-236, October 17-21, 1982.
- 4. R. Reif, "Chemical Vapor Deposition of Epitaxial Silicon", <u>Proceedings</u> of <u>The Flat-Plate Solar Array Workshop on the Science of Silicon Mater-</u> <u>ial Preparation</u>, JPL Publication 83-13, pp. 253-264, Jet Propulsion Laboratory, Pasadena, CA, February 1983. <u>Invited paper</u>.
- 5. W. R. Burger, T. J. Donahue, and R. Reif, "Properties of Thin Polycrystalline Silicon Films Deposited by Plasma Assisted CVD", <u>Proceedings of</u> <u>the Fourth European Conference on Chemical Vapor Deposition</u>, J. Bloem <u>et al.</u>, editors, Eindhoven Druk B. V., pp. 265-272, 1983.
- T. J. Donahue, W. R. Burger, and R. Reif, "Low-Temperature Silicon Epitaxy using Low Pressure Chemical Vapor Deposition with and without Plasma Enhancement", Applied Physics Letters, <u>44</u>, pp. 346-348, 1 February 1984.

PATENT APPLICATION

R. Reif, T. J. Donahue and W. R. Burger, "Growth of Epitaxial Films by Chemical Vapor Deposition", to be filed in June 1984.

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