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March 6, 1962

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I. Introduction

Present methods for predepositing phosphorus in silicon lack sufficient control and reproduciability to meet the exacting requirements of large-area silicon radiation detectors. Unlike transistor fabrication techniques--in which diffusion at high temperatures and long times are common--radiation detectors, for certain applications, require shallow diffusions and therefore low diffusion temperatures: temperatures in the range of 600 to 900°C, and junction depths of the order of 300 to 5000 Å.

The source commonly used for the predeposition of phosphorus is phosphorus pentoxide. In order to improve the control over that obtainable with phosphorus pentoxide, the following phosphorus compounds were screened: PCl_3, PF_5, $POCl_3$, PH_3, and the azeotrope of P_2O_5 and H_2O. The method used in the last case and the results obtained proved to be of interest and are reported here.

II. Source Preparation

The azeotrope of phosphorus pentoxide and water has the following properties:[1]

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[1] J. R. Van Wazer, Phosphorus and its Compounds, (Interscience Publishers, Inc., New York, 1958), Vol. 1, p. 773.

(a) Composition: 92.4% P_2O_5, 7.6% H_2O;

(b) Boiling point: 864°C;

(c) Vapor pressure: $\log P_{mm} = 8.7788 - \frac{5.9080 \times 864}{T}$,

where P is the pressure in mm of Hg, and

T is the temperature in °K.

The azeotrope is readily prepared by heating metaphosphoric acid in a platinum boat at 800°C for several hours. Since the source is an azeotropic mixture, its composition is conveniently maintained by its frequent use.

III. Method of Obtaining Predeposit

The prepared source in a platinum boat was used in the furnace, and located upstream from the wafers. Nitrogen gas flowing at the rate of 1/2 liter per minute from boiling liquid nitrogen was used as the carrier in a 2-in.-diameter quartz tube. To minimize phosphorus contamination near the furnace area the exhaust gases were led to a fume hood.

Satisfactory predeposits have been obtained by subjecting suitably cleaned wafers to the vapors for periods of 60 min to 16 hr, and at temperatures in the range of 600 to 800°C. Temperatures and times different from the indicated ones may also be used.

IV. Results

1. Predeposit at 800°C for 60 min

Wafers of approximately 1500 ohm-cm, p-type silicon gave the following surface sheet resistances:[2]

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[2] Sheet resistances were measured with a four-point probe with equally spaced points.

Wafer #	ohms/□	Wafer #	ohms/□
627	68	541	77
628	75	542	64
629	104	545	64

2. Predeposit at 600°C for 16 hr

Wafers of approximately 1500 ohm-cm, p-type silicon gave the following sheet resistances:

Wafer #	ohms/□	Wafer #	ohms/□
537	4.5×10^3	538	4.5×10^3
539	4.5×10^3		

3. Predeposit at 800°C for 60 min followed by diffusion at 900°C for 30 min

Wafers of approximately 1500 ohm-cm, p-type silicon gave the following sheet resistances:

Wafer #	ohms/□	Wafer #	ohms/□
658	25	518	32
659	25	519	31
517	32	520	30

V. Discussion

Silicon wafers predeposited at 800°C for 60 min do not have the heavy phosphosilicate glass layer commonly obtained by the use of P_2O_5 or other sources. However, a hydrophilic thin layer is present and may be removed by washing with 25% HF. The layer is sufficiently thin that contact to the silicon may be made by pressure points. Uniform large-area predeposits were obtained in all cases. Wafers diffused by this procedure also made radiation detectors with desirable characteristics.

The observed variation in surface sheet resistances were probably due to a nonuniform furnace temperature profile.

VI. Conclusion

The azeotrope of phosphorus pentoxide and water provides a unique source of phosphorus whose composition is invariant and whose vapor pressure is controlled by furnace temperature. Its use gives reproducible results particularly valuable at the low temperatures required for the shallow diffusion of silicon radiation detectors.

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