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Abstract

A manufacturing process for passivated p-n junction detectors is described. The role of oxygen during the diffusion process is emphasized; careful control of oxygen is a feature of the process. The results obtained justify the belief that this technique may result in the availability of cheap high-quality detectors, which may make possible the construction of large detector arrays.

1. Introduction

Early attempts to apply to diffused junction particle detectors oxide passivation techniques similar to those used in planar transistor technology proved unsuccessful owing to electrical effects at the silicon dioxide — silicon interface. Although these effects are tolerable in transistors in which the bulk material resistivity is low, they are much more important in the high-resistivity material used in particle detectors. We have carried out a lengthy investigation of the electrical properties of the SiO_2-Si interface under various diffusion conditions and have devised some understanding of the parameters involved in controlling these properties. The result of this work, as applied to semiconductor detectors, is the subject of this note. The technique described permits production, with high yield, of passivated junction detectors simultaneously exhibiting excellent energy resolution and long-term reliability. The basic method of compensating surface states has been described previously,[1] but the better understanding we now have of parameters that affect the final surface has resulted in greatly improved reproducibility as compared with our earlier work.

Several authors[2, 3] have measured the surface states produced at SiO_2-Si interfaces in diodes produced by a planar technique using standard diffusion methods for junction production. These measurements, which agree

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[1] J. Appl. Phys. 34, No. 5, 1570-1571 (1963).

[2] M. M. Atalla, E. Tannenbaum, and E. S. Scheibner, Bell System Tech. J. 38, 749-84 (1959).

[3] L. M. Terman, An Investigation of Surface States at a Si-SiO_2 Interface, Stanford Electronics Laboratories Technical Report No. 1655-1, Feb. 1961.

with our own, show that the interface is characterized by a layer of donor states with a density of about $5 \times 10^{12}/cm^2$ of area. In n-on-p diodes, this surface inversion layer constitutes a "skirt" or "channel" resulting in large junction leakage currents and excessive noise. It has generally been assumed that these donor states at the SiO_2-Si interface are produced during the oxidation process, and the constancy of the density of states reported by different workers supports this assumption. However, we observed some time ago that the density of surface states under the oxide was smaller in diodes made by diffusing phosphorus through a hole in an oxide mask into p-type silicon in a completely nonoxidizing environment than when the diffusion was carried out in an oxidizing environment. This observation suggested that the role of oxygen during the phosphorus diffusion step was important, and pointed to the possibility that the presence of oxygen frozen into the SiO_2 lattice after cooling might be a major factor in determining the electrical properties of the SiO_2-Si interface. The obvious solution to the problem might appear to be to carry out the diffusion in a nonoxidizing environment, but oxygen seems to be necessary during part of the process in order to allow the phosphorus to enter the silicon lattice in sufficiently high concentrations at the shallow diffusion depths used ($< 0.5\ \mu$). The procedure described below takes these factors into account, giving consistent control over oxygen during the process and, consequently, reproducible behavior of the surface states, which allows us to compensate quite accurately for their electrical effect.

2. Manufacturing Procedure

Four basic high-temperature steps are used in producing detectors. All are carried out in tube furnaces with 2-in.-diameter clear fused silica tubes.

Wafers are initially prepared with the usual precautions, etched in 3:1 HNO_3-HF mixture, and washed in deionized water. Following each high-temperature treatment the furnace is allowed to cool in its natural time to 600°C to anneal the wafers, which are then removed from the furnace. The guard-ring geometry[4] is used on all detectors. This structure can tolerate greater departures from correct surface compensation than the simple diode structure, and measurement of the interelectrode impedance provides an accurate check on the degree of compensation, thereby allowing adjustment of the compensation in successive batches if required. In practice, this is most important when establishing new compensation conditions for a material of different bulk resistivity from previous batches.

The four high-temperature steps are carried out in the following sequence[5] and with the following conditions.

2. a. Boron diffusion

The purpose of this diffusion is to produce a good p^+ back contact for the diode. It is carried out for 2 hours at 1000°C following a 1-hour predeposit at the same temperature. The boron source is BI_3 vapor obtained from BI_3 solid at room temperature over which the carrier gas flows. The carrier gas, consisting of 50 cc/min of hydrogen and 450 cc/min of nitrogen, provides a dry reducing ambient for the diffusion. Oxygen is removed from the carrier gas by palladium catalysis and dried by a molecular sieve and a liquid nitrogen trap. The predeposit time quoted gives the maximum boron concentration without leaving a visible boron deposit and gives a surface sheet resistance of 15 ohms/square.

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[4] F. S. Goulding and W. L. Hansen, Nucl. Instr. Methods 12, 249 (1961).

[5] The sequence is described for p-type bulk material in the resistivity range > 1000 ohms/cm.

Following the boron diffusion one side of the wafer is etched (masking the other side with Picein in trichloroethylene) to remove the boron from it. We refer to the boron side as the back of the wafer. The wafers are washed in deionized water.

2.b. Gallium diffusion

The purpose of this diffusion is to provide a layer of acceptors that will just compensate the donor states to be formed later by oxidation. The wafers are placed in a closed silica boat containing 99.999% pure elemental gallium. This is inserted in the cold zone of the furnace tube which is twice evacuated and filled with carrier gas. The boat is then pushed into the furnace hot zone. The carrier gas and its flow rate are the same as used in the boron diffusion. Gallium diffusions are carried out for 1 hour and the furnace temperature is adjusted to give the correct gallium concentration. For example, if the bulk resistivity is 4000 ohms/cm, the correct gallium concentration gives a sheet resistance of about 3000 ohms/square on high resistivity n-type silicon, and this is produced at a furnace temperature of about 880°C. The use of elemental gallium as a source-limited diffusant gives a shallow, low-concentration layer which will be almost totally gettered during the oxide growth. This combination of low source concentration and gettering by the oxide provides the control necessary to achieve almost exact compensation of the oxide-induced acceptors.

2.c. Oxidation

The wafers are transferred directly from the gallium furnace to the oxidation furnace with no cleaning. A 2-hour 1000°C steam oxidation is used for all detectors, the wafers being inserted into the furnace tube already filled with steam. The steam is replaced by dry nitrogen during the cooling period following oxidation.

After cooling, the guard-ring detector geometry is produced by painting with Picein in trichloroethylene and removing oxide in the regions where phosphorus is to be diffused by use of $NH_4F \cdot HF$. Satisfactory interelectrode spaces are obtained by using a ruling pen and a small turntable. Spacings less than 4 mils are obtained by this technique. Following this treatment the Picein is removed by trichloroethylene and the wafer is thoroughly washed in deionized water.

2. d. Phosphorus diffusion

As mentioned earlier, the role of oxygen in the phosphorus diffusion is most important. For this reason, extreme care is taken to establish the same conditions in this furnace for each diffusion. In principle, the schedule in this furnace consists of a 1-minute predeposit in oxygen (1 liter/min) saturated with $POCl_3$ followed by 29 minutes of diffusion in dry nitrogen (1.5 liters/min). The sheet resistance of the diffused layer is about 8 ohms/square.

It is essential to avoid the presence of oxygen during the 29-minute diffusion part of this schedule. To accomplish this, two steps are taken:

(i) A special glass valve is used to switch from the oxidizing predeposit gas flow to the dry nitrogen; there are no side necks from which oxygen can diffuse.

(ii) A silica wool plug is used at the entrance to the furnace tube to break up laminar flow and prevent back-streaming.

3. Detector Characteristics

Many detectors of various shapes and sizes have been made by this process and no difficulties have arisen in maintaining excellent yield and

performance over a period of 4 months. Detector leakage currents are typically 10^{-7} A/cm^2 at 300 V when the detectors are used as guard-ring devices. A typical leakage curve is shown in Fig. 1. Observed α-particle (Am^{241}, 5.5 MeV) resolutions, with a preamplifier employing a 7788 tube at its input, range from 13 keV for a 0.2-cm^2 detector to 18 keV for a 2-cm^2 detector. On a production run of 104 detectors (2 cm^2) a criterion of 18-keV resolution (full width at half maximum) resulted in the rejection of only four devices.

We have observed that long-term storage of detectors with only oxide protection generally results in a degradation of characteristics, owing to surface contamination. Washing in water restores the original characteristics in these cases. To prevent this degradation, which may possibly be due to accumulation of electrical charge on the surface of the oxide, we now coat the oxide surface with a varnish of the epoxy or silicone (Dow-Corning 991). Detectors treated in this way appear to retain their characteristics for at least several months under various storage and operating conditions.

4. Conclusions

The process described here offers the prospect of the manufacture of silicon p-n junction detectors exhibiting excellent performance at relatively low prices. The high cost of diffusion equipment is offset by its ability to process large quantities of devices simultaneously. Batch quantities of up to 100 seem quite possible with existing techniques. This factor, combined with the long-term reliability inherent in these devices, may make practicable the use of large arrays of solid-state detectors. Only diffused passivated detectors offer this prospect at the present time.

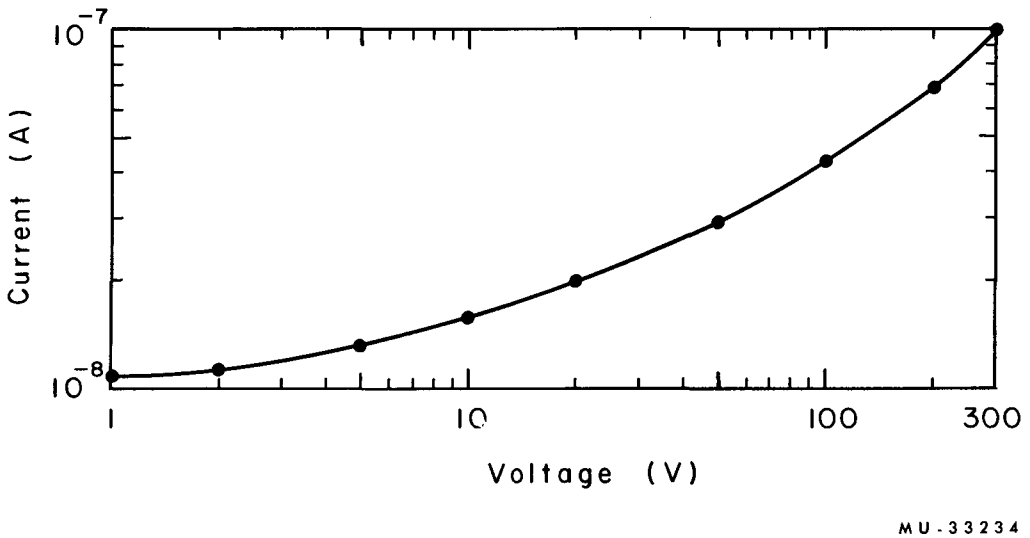

Fig. 1.

Acknowledgment

Blair Jarrett and Morris Roach assisted in the construction and evaluation of the devices used in this study. The contribution of Robert P. Lothrop to the earlier phase of this investigation is gratefully acknowledged.

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Figure Caption

Fig. 1. Typical I-V characteristic of oxide-passivated guard ring silicon detector—2 cm^2 area.