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THE PREPARATION OF LITHIUM-DRIFTED SEMICONDUCTOR NUCLEAR PARTICLE DETECTORS

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A method is described for the preparation of p-i-n structure semiconductor nuclear particle detectors using the process of ion drift. The procedure is described in detail and much attention is given to the measurements of parameters which control various steps in the procedure. The reproducibility, obtained by the method, turned out to be very high. The properties of the detectors are described briefly.

1. Introduction

In this paper a description is given of the preparation of semiconductor nuclear particle detectors, which have a p-i-n structure. The detectors are made from p-type, boron doped, silicon. In this material an n-type region is created by the diffusion of lithium. Intermediate between the p- and n-type regions an intrinsic region is formed by the process of ion drift, introduced by Pell[1]). In this process the lithium ions are made mobile by the combined action of increased temperature and a reverse bias of the p-n junction. The electric field in the p-n junction is made high enough to govern the lithium distribution. An automatic compensation of the negative boron ions by the positive lithium ions is then obtained.

It is the purpose of this paper to give the experimental procedure followed for the preparation of these detectors. The recipe depends for several parts on the work done by Mayer[2]), Elliott[3]), Dearnaley and Whitehead[4]). Since we know that on many places efforts are made to produce these p-i-n type semiconductor detectors it seemed worth while to publish in detail a method, from which a high degree of success is obtained.

Special attention is given in this paper to quantities, which can both be calculated and be measured, and which can serve therefore as a check on the followed procedure.

2. The Material

The silicon which was used, was ordered[5]) according to the following specifications:

a: p-type, with boron as impurity.
b: resistivity 100 Ωcm.
c: cylindrical bar, diameter 2 cm.
d: single crystal, a $<111>$ direction parallel to the cylinderaxis.
e: oxygen content below 10^{15} atoms per cm^3.
f: lifetime 100 μs.

The slices which were sawed from the bar, were optically polished on both sides. The optical polish was made using diamant powder of maximum grain size 0.7 μm. However, on the bright reflecting surfaces sometimes some very tiny lines could still be seen. The thickness of the polished slice was made about 300 μm larger than the required sensitive depth, because the thickness of the remaining insensitive lithium rich region is about equal to 200 μm and about 100 μm of Si is lost by etching. The thickness when measured on different spots of the slice, varied less than 10 μm.

3. The Front Contact

3.1.

The slice, which is from now on never touched by hands, is washed respectively in a synthetic

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[1]) E. M. Pell, J. Appl. Phys. **31** (1960) 291.

[2]) J. W. Mayer, N. A. Baily and H. L. Dunlap, Conference on Nuclear Electronics, Belgrade, May 1961.

[3]) J. H. Elliott, Nucl. Instr. and Meth. **12** (1961) 60.

[4]) G. Dearnaley and A. B. Whitehead, Nucl. Instr. and Meth. **12** (1961) 205.

[5]) Merck Sharp and Dohme International.

detergent, distilled water, methanol, trichloroethylene, methanol, distilled water and deïonised water (resistivity larger than 2 MΩcm).

The slice is then etched in an etchbath of composition HF (40%): HNO_3 (fumans) = 1:10 for 5 min. The same amount of HNO_3 is then added and the etching is continued for another 10 min. After that time the etch is diluted by pooring distilled water into it. Care is taken to avoid exposure of the silicon slice to the air during the early stages of the dilution.

The etch, which is contained in a polyethylene beaker, is prepared about 30 minutes before use and is cooled in ice. During the etching the liquid is kept in continuous movement by shaking the beaker.

The slice is washed again in methanol, trichloroethylene, methanol, distilled water and deïonised water. Maximal purity is pursued by drying the slice in a stream of nitrogen, blowing off as much water as possible.

3.2.

Contact to the p-type region, which is the front of the detector, is made with a thin layer of aluminum. The slice is mounted in a holder in which it lies down on a freshly split piece of mica. The upper side of the slice is left free for the Al-evaporation, with exception of a ring around the edge, about 1 mm in width, which is covered by a thin perspex mask.

The thickness of the evaporated aluminum layer is about 0.2 μm. The quantity of Al, needed for evaporation is calculated from geometrical considerations.

3.3.

A good ohmic contact between Al and Si is made by alloying for a few minutes at 650° C. The oven consists of a quartzglass tube around which the heating element is wound. The Si-slice is brought in at once from room temperature to the alloying temperature, lying down on a carbon strip. A stream of nitrogen, purified in concentrated H_2SO_4 and liquid air, is lead through the oven to keep air out. The temperature is measured with a NiCr-Ni thermocouple, which is lead into the oven in a separate smaller quartzglass tube. In order to avoid the introduction of crystal imperfections which lower the carrier lifetime, the temperature is decreased slowly after the alloying, the rate of temperature variation in the interval between 650° C and 450° C being − 2° C per minute.

3.4.

The thickness and the variations in thickness of the aluminum layer can be measured, both before and after the alloying, using the four point probe method. When the appropriate correction factors[6, 7]) are applied the sheet resistivity yields values of about 0.14 Ω. The agreement between thickness of Al-layer, calculated from the geometry used in evaporation and from the electrical measurements, is within 10%.

The method for the preparation of the Al-contact can be tested by making this contact on both sides of a slice. When the contacts are made in the right way the measured resistance agrees with the resistance calculated from the resistivity of the material, the thickness and diameter of the slice.

4. The Lithium Diffusion

4.1.

Into the other side of the slice, which becomes the back of the detector, lithium has to be diffused. The lithium is available as a lithium in oil suspension[8]). A thin layer of this suspension is painted with a brush on the Si-slice. The layer must be thin enough in order to prevent smearing out during the heating steps which follow. The layer on the slice is dried by driving off the oil at a temperature of about 250° C. This takes 5 to 10 min. When the surface looks dry the slice is immediately brought at a temperature of 450° C, at which temperature it is kept for 5 min. During this time the diffusion takes place. After the diffusion the slice is cooled down at once to 250° C at which temperature it is kept for about 10 min. This step is followed by further cooling to room temperature.

Two separate ovens are wound on one quartzglass tube. The one designed to give the tempera-

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[6]) F. M. Smits, Bell Syst. Techn. J. **37** (1958) 711.

[7]) L. B. Valdes, Proc. of the I.R.E. **42** (1954) 420.

[8]) Lithium Corporation of America.

ture of 250°C has a small window in the middle through which the Si-slice can be observed. A small stream of purified nitrogen is blown through the ovens during the whole process.

4.2.

The slice, now a diode, is cleaned by washing in methanol, distilled water and deïonised water. The slice is dried by blowing off the water with a jet of nitrogen, as before.

4.3.

The Li-distribution after the diffusion and the depth of the p-n junction can be calculated from known parameters, as diffusion constant[9,10] and lithium-solubility[11].

The uniformity of the Li-diffusion can be checked and the depth of the p-n junction can be calculated from four point probe measurements[12]. The measured sheet resistivity has a value of about 2 Ω.

Another method for determining the position of the p-n junction is by chemical stain. A slice is cut in two about equal pieces, using a diamant saw. The edges are polished with Al_2O_3 grinding powder. The edges are wet with HF with a very small amount of HNO_3 (one drop of HNO_3 in 20 cm^3 of HF). Then within a few seconds the p-region turns dark and the n-region light. In this way the p-n junction is made visible and its position can be measured with a microscope.

4.4.

The concentration of the Li-atoms as function of the depth into the silicon is given by: $N_{Li}(x) = N_{Li}(0) \cdot \mathrm{erfc}\,[x/2(Dt)^{\frac{1}{2}}]$. On the basis of a linear-graded junction approximation the capacity of the p-n junction can be derived. The result is: $C = \varepsilon_0 \varepsilon_r A (N_B e d / 24 \varepsilon_0 \varepsilon_r D t V)^{\frac{1}{3}}$, in which d, the depth of the p-n junction, can be found from $N_B = N_{Li}(0) \cdot \mathrm{erfc}\,[d/2(Dt)^{\frac{1}{2}}]$. The symbols are: C = capacity, A = detector area, ε_0 = dielectric constant of free space, ε_r = relative dielectric constant of Si ($\varepsilon_r = 12$ at room temperature), N_B = boron concentration, e = electron charge, D = diffusion constant, t = diffusion time, V = reverse bias of the p-n junction and $N_{Li}(0)$ = surface concentration of the Li in the Si.

The capacity dependance on the voltage is therefore according to a $V^{-\frac{1}{2}}$ power law. This is only true when the linear-graded junction approximation is valid[13] and when room temperature drift can be neglected. For this reason the capacity must be measured soon after the diffusion.

5. Preparation for the Drift Process

5.1.

The diode is washed in methanol, trichloroethylene, methanol, distilled water and deïonised water. The slice is dried by blowing off the water.

5.2.

The central part of the Li-side and the whole Al-side of the diode are now covered with black wax. Care is taken not to cover the region where the p-n junction reaches the surface. The black wax is used solved in toluol and is dried during 12 hours. The edges of the diode are then etched, during two minutes, in an etch of composition HF (40%): HNO_3 (fumans): CH_3COOH (100%) = 2:2:5. The etching is done in the same way as described earlier.

5.3.

After the etching the black wax is removed with toluol. The diode is furthermore washed in methanol, distilled water and deïonised water and dried as before.

5.4.

The diode is mounted in a steel holder with spring contacts.

6. The Drift Process

The drift process takes place in a rapidly stirred oilbath. Silicon-oil MS 200 with a viscosity of 100 cS is used. The temperature of the oil is measured with a thermocouple (Fe-Constantan) and regulated with the drift current.

To avoid thermal runaway the power dissipation

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[9] E. M. Pell, Phys. Rev. **119** (1960) 1222.

[10] E. M. Pell, Phys. Rev. **119** (1960) 1014.

[11] C. S. Fuller and J. A. Ditzenberger, Phys. Rev. **91** (1953) 193.

[12] G. Backenstoss, Bell Syst. Techn. J. **37** (1958) 699.

[13] H. Lawrence and R. M. Warner, Bell Syst. Techn. J. **39** (1960) 389.

in the diode is kept below 1 W per cm^2 by regulating the temperature for a fixed drift voltage.

With 100 Ωcm material the maximum allowable drift temperature is 160° C, in order to keep the intrinsic carrier concentration n_i[14]) below the impurity hole concentration in the B-region.

The increase in the thickness of the intrinsic region is calculated from $W_2^2 - W_1^2 = 2\,\mu V_d \cdot (t_2 - t_1)$. ($W_1$ = initial thickness of the intrinsic region, W_2 = final thickness, μ = mobility of the Li-ions[9,15]), V_d = drift voltage, $t_2 - t_1$ = drift time).

The drift process is stopped, now and then, for measurements of the reverse current i and the capacity C at room temperature (20° C). These measurements give a rough determination of the thickness W of the intrinsic layer, since i is proportional and C is inversely proportional to W. If the detector is sufficiently clean to ensure that the surface leakage current is small compared to the space charge generated current the detector current i is equal to $AWn_ie/2\tau$ in which τ is the, generally unknown, lifetime of the charge carriers. Because of the analogy with the parallel plate condenser the capacity of a p-i-n structure is given by $C = \varepsilon_0 \varepsilon_r A/W$. For the capacity measurements a high frequency bridge is recommended in order to decrease the phase angle due to the ohmic component of the space charge generated current.

The presence of oxygen in the silicon slows down the growth rate of the intrinsic region through the formation of the LiO$^+$-complex[16,17,18]).

At the end of the drift process the "window"-thickness on the B-side of the detector is determined accurately with the 6.06 MeV and 8.78 MeV α-particles of a ^{212}Pb-source. The silicon-oil is washed off in gasoline.

7. The Mounting

When the thickness of the intrinsic region, which is the sensitive part of the detector, is sufficiently large, or alternatively, when the "window"-thickness of the detector is sufficiently small, the drift process is stopped.

The detector is cleaned by washing or etching and then mounted in a perspex holder. The electric contacts to the diode are made with small copper wires. These wires are attached with Ag-paste on both sides of the detector, somewhere near the edge on the Al-side and in the middle of the Li-side.

8. Detector Properties

Detectors have been made with thicknesses ranging from 0.7 to 4.4 mm and with areas between 1 and 3 cm^2. The thickest ones were able to stop 30 MeV protons.

The best energy resolution obtained for energies above 20 MeV was about 1%. The greater part of this spread is due to the energy spread in the cyclotronbeam. From noise measurements an average line width of about 100 keV may be expected.

The "window"-thickness of the detectors was determined with the 6.06 MeV and 8.78 MeV α-particles of a ^{212}Pb-source, which have a range in Si of 32 μm and 54 μm respectively. The particles were however already observed when the thickness of the p-region exceeded the range-values appreciably. This effect is attributed to the diffusion of electrons, created in the p-region, to the intrinsic region. More measurements concerning this subject are in progress.

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[14]) F. J. Morin and J. P. Maita, Phys. Rev. **96** (1954) 28.

[15]) C. S. Fuller and J. C. Severiens, Phys. Rev. **96** (1954) 21.

[16]) E. M. Pell, Solid State Physics in Electronics and Telecommunications, Volume 1 (Academic Press, 1960) p. 261.

[17]) E. M. Pell, J. Appl. Phys. **32** (1961) 1048.

[18]) H. Reiss, C. S. Fuller and F. J. Morin, Bell Syst. Techn. J. **35** (1956) 535.