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LITHIUM-DRIFTED SILICON RADIATION DETECTOR PRODUCTION PROCESS

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PRODUCTION PROCESS**

**Berkeley, California**

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PRODUCTION PROCESS

Robert P. Lothrop and Harry E. Smith

June 11, 1965



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Lawrence Radiation Laboratory  
University of California  
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ABSTRACT

A process for the fabrication of lithium-drifted silicon radiation detectors with thin entrance windows is described. High-resistance p-type silicon is diffused, drifted, and processed to form devices useful in a wide range of charged-particle and low-energy gamma experiments.

## INTRODUCTION

The process described in these notes has been in use for approximately one year, during which many hundreds of useful devices have been produced. Most of these devices are used either in  $dE/dX$ -E counter systems for experiments with the Berkeley 88-inch cyclotron or in low-temperature high-resolution  $\beta$  spectroscopy. With silicon of sufficiently high quality 3-mm-thick devices having 4- to 6-keV line widths for  $Cs^{137}$  betas are made routinely for  $\beta$  spectroscopy.

These notes have been collected in recognition of interest shown by other laboratories. No effort has been made to explain the reasons for certain details of the process. Although the results of the descriptive sections may be obtained by other means, it is strongly recommended that the directions given in the imperative form be followed explicitly.

For convenience, the process is divided into four main phases:

- A. Predrift
- B. Drift
- C. Postdrift
- D. Surface treatment and electrical tests.

### A. PREDRIFT

1. Float zoned, p-type silicon having the following characteristics is chosen:

750 to 1500 ohm-cm,  
500  $\mu$ sec minimum lifetime,  
22 to 24 mm diam typical.

2. To evaluate new silicon stock, sample wafers are sawed, lapped (Lapmaster 1950), washed with water and methyl alcohol, dried (in all steps, "dry" or "dried" means blown dry in a jet of dry nitrogen), and copper-stained.

Copper stain solution:

Copper sulfate	20 g,
Water	1 liter,
48% HF	1 ml.

### Procedure

The clean, lapped wafer is placed in a dish and covered with copper stain solution. A bright light, such as a microscope lamp (preferably rich in red), is shone on the wafer for a period in the range of 30 seconds to 5 minutes. The wafer is removed, washed with water, then with methyl alcohol, and dried. Examination of the copper stain at low angles should show a light, uniform stain. A centrally located spot of heavily deposited copper may indicate an n-type core. If there is a central spot, concentric rings, spiral, or other reproducible nonuniformity, the material should be rejected; it has been found that, at best, such material yields devices of inferior quality.

3. To fabricate detectors, saw wafers  $X + Y + 15$  mils, where

X is the desired device thickness,  
Y is the saw blade thickness.

4. Machine lap both sides (Lapmaster 1950) to  $X + 10$  mils with no scratches. Clean wafers with water, then methyl alcohol, and dry.

5. For lithium evaporation, the wafers are mounted by steel wire clips to a flat copper plate about 4 in. in diameter. Aluminum foil prevents contamination of the silicon from the copper plate, which is used repeatedly for lithium evaporation. Water cooling coils are embedded in the copper plate, which in turn is bolted to an electric heating element. The assembly is mounted in an inverted position in a vacuum evaporator. Water lines, electric leads, and thermocouple are connected. The system is pumped down to about  $5 \times 10^{-6}$  mm Hg, and the temperature of the assembly is raised to  $350^\circ\text{C}$ . Lithium is then evaporated from a tantalum cup onto the entire surface of the wafers for 30 seconds. This period is begun and ended by removal and replacement of a metal baffle above the cup. After 2 min at  $350^\circ\text{C}$ , which includes the 30 sec of evaporation, the heating element is turned off and water is passed through the cooling coils. The present assembly drops from  $350^\circ$  to  $100^\circ\text{C}$  in 28 sec. The copper plate is 4 in. above the evaporation cup, and about 0.1 cc of lithium is used during each evaporation. This procedure yields uniform lithium diffusions of about 5 ohms (four-point probe), approximately 3 mils deep.

6. Remove surplus lithium by flushing with water or methyl alcohol.

7. Etch the entire wafer for 30 sec, quench in running distilled water, and place in a beaker of methyl alcohol. Wash and scrub (cotton swabs) with trichloroethylene, repeat with methyl alcohol, and dry. In all cases the etch is three volumes of reagent grade nitric acid and one volume of 48% electronic-grade hydrofluoric acid.

8. Evaporate the gold drift geometry onto the side not lithium-diffused. A thin washer 5 mm o. d.  $\times$  3 mm i. d. laid on the wafer will prevent deposition on a 1-mm-wide ring. This contact arrangement allows the continuous measurement during drift (by the automatic drifting apparatus), of the distance of the drifted region from the back of the wafer.

9. Mask the gold back contacts and the mesa (typically 14 mm diam) with etch resist (Picein 80 in trichloroethylene). A "LeRoy" draftsman's lettering pen mounted over a rotating chuck is useful for applying the Picein 80. Etch for 6 min and quench in running distilled water. Agitation should be sufficiently vigorous to remove any bubbles formed during the entire etch process. Remove masking with trichloroethylene and dry.

10. Wash and scrub (cotton swabs) with trichloroethylene, repeat with methyl alcohol, and dry.



## B. DRIFT

An eight-station drift unit as described by Goulding and Hansen<sup>1</sup> is used for the drift operation. This drift unit has been in continuous operation for about 17 months. The unit (see Fig. 1) operates as follows:

1. The prepared wafer is placed in a station consisting of a separate high-voltage contact, a copper block which contains a power transistor, the two back contacts to the wafer, and a thermocouple. The assembly is covered to reduce heat loss and to eliminate light.

2. Heating, and therefore drifting, is initiated by selecting demand current other than zero. The control circuitry is arranged so that it controls the drift current by heating (or cooling) the wafer. Typical drift conditions are 1 mA at 500 or 1000 V, with the resulting temperature in the range of 120 to 130°C. Increasing the demand current by a factor of two raises the temperature about 10°C.

3. The resistance between the two back contacts, and hence the distance of the drifted region from the back, is monitored continuously. The resistance sense signal is compared with that of the preset control and when the device has drifted to the preselected distance from the back, will turn off the heater, thus stopping drift automatically.

## C. POSTDRIFT

1. Remove the gold drift geometry by lapping (Lapmaster 1950), wash with water and then methyl alcohol, dry, and copper stain. The exposed drifted region stains deeply compared with the surrounding p-type material, and should be of approximately the same diameter as the mesa.

2. Mask the wafer so that the copper-stained drifted region and several millimeters of surrounding p-type material will be etched to form a shallow well.

3. Etch for 3 min, quench in running distilled water, wash with methyl alcohol, and dry.

4. Remove the masking with trichloroethylene and dry.

5. Time the following for late afternoon:

Etch the entire wafer for 30 sec, quench in running distilled water, and place in a beaker of methyl alcohol.

6. Wash and scrub (cotton swabs) the entire wafer with trichloroethylene, giving particular attention to the well area. Repeat with methyl alcohol. Soak the wafer in 48% hydrofluoric acid for 30 sec minimum. Wash with methyl alcohol, and dry.

7. Store overnight, well side up, in a clean covered dish.

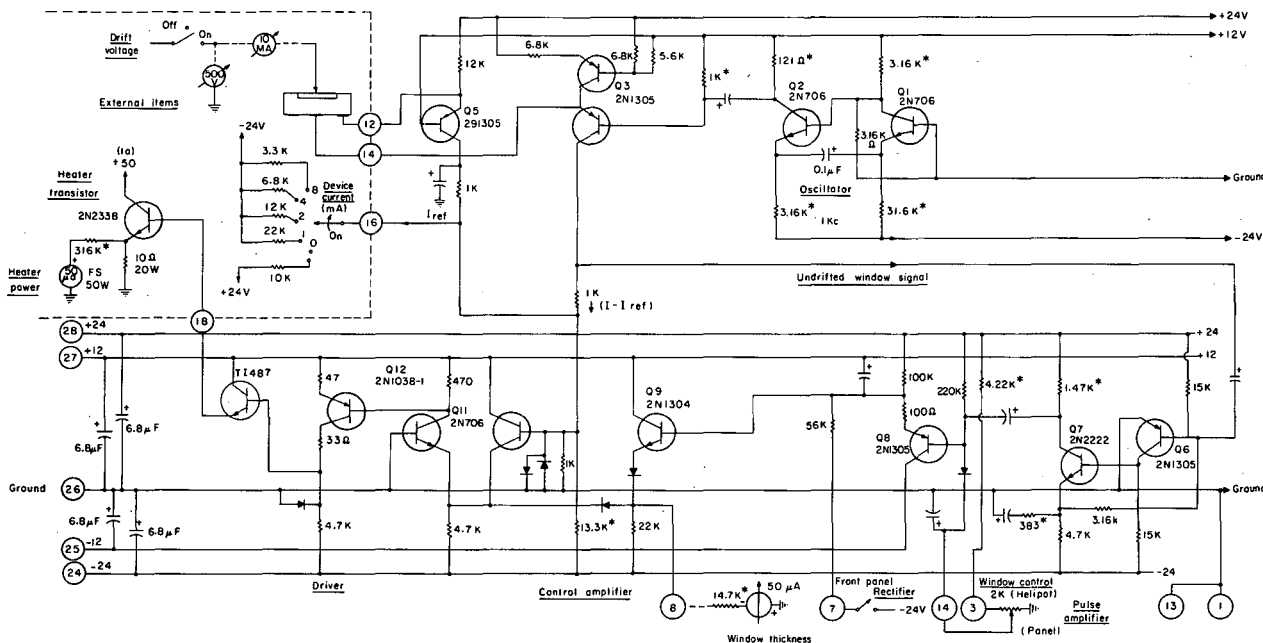


Fig. 1. Lithium-drifting temperature controller.

Unless stated otherwise,

All capacitors:  $6.8\mu\text{F}$ ,  $\pm 20\%$ , 30 V, tantalum;

All resistors: (i) Carbon, 5%, 1/4 W;

(iii) \*Metal film, 1%, 1/8 W;

All diodes: IN 904.

8. Early the next day, evaporate a gold contact over the entire well side and a gold contact on the lithium-diffused mesa. See Fig. 2.

#### D. SURFACE TREATMENT AND ELECTRICAL TESTS

A block diagram of the test apparatus is shown in Fig. 3. The calibration and test procedures are as follows:

##### System calibration

An Am<sup>241</sup> alpha source is used as the calibration reference. A detector known to have good characteristics is placed in the test chamber, the chamber is evacuated, and the detector properly biased. The oscilloscope gain is set to 1 V/cm. The linear amplifier is then adjusted so that the 5.47-MeV alpha pulse is 5.47 cm high.

##### Noise test

Calibrate the system, as described above, and then set the oscilloscope gain to 0.1 V/cm. We require that signal for the peak-to-peak noise be less than 1 cm high at or above the operating voltage. The minimum operating bias we observe for completed devices is

$$V_{\min} = 100 T + 50,$$

where T is the device thickness in mm.

The minimum bias is raised at least 50 V for tests before final protection is applied. For a 3-mm-thick device with 14-mm sensitive diameter, the noise signal should be less than 1 cm at 350 V. With finished devices of this size, we typically obtain 400- to 500-V operation with leakage of 2 to 10  $\mu$ A.

##### Surface treatment

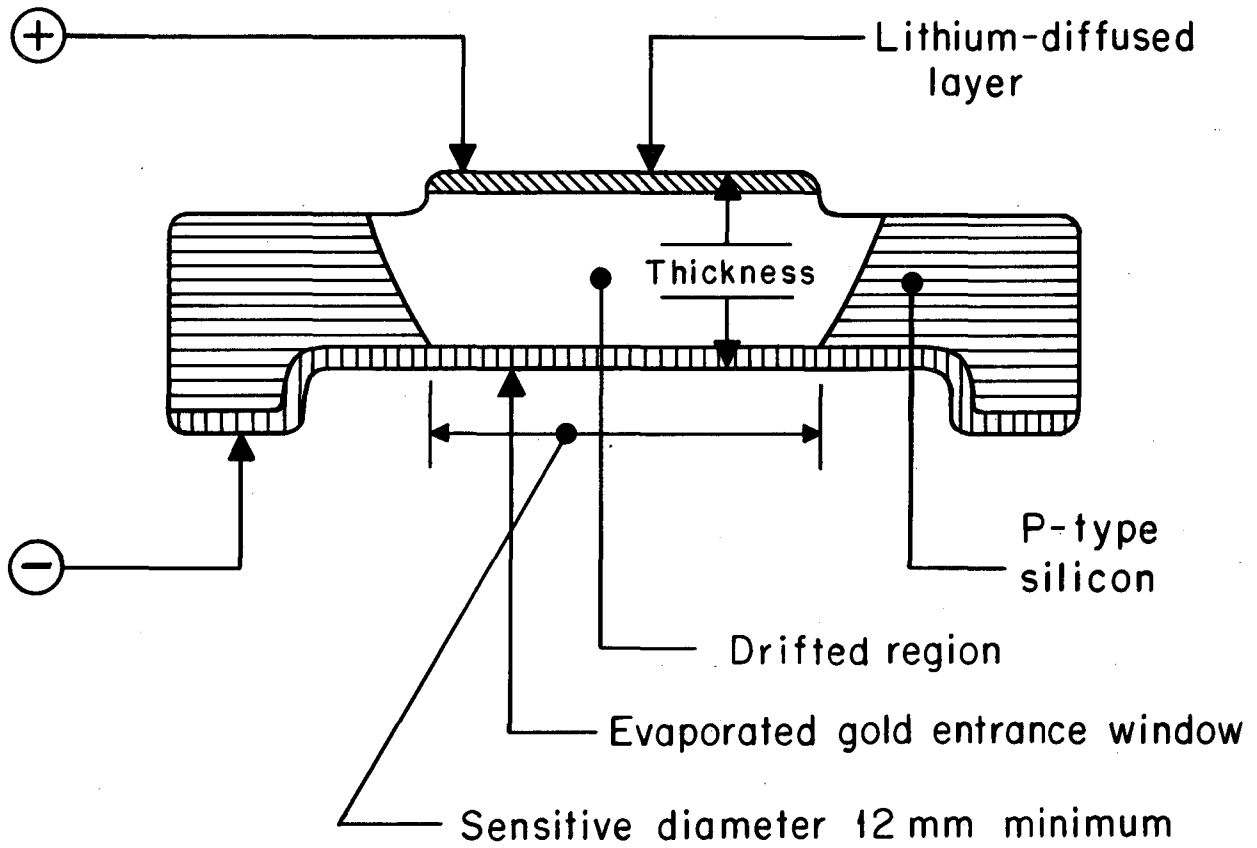
1. Wash and scrub the junction edges and exposed drifted surface with trichloroethylene. Repeat with methyl alcohol, and soak in 48% hydrofluoric acid for approximately 5 to 30 sec. Remove the wafer from the HF. Wash with a minimum of methyl alcohol, and dry.

2. Test, observing the noise and leakage current as a function of applied voltage.

Further washing with methyl alcohol will usually decrease both the leakage current and the voltage at which the noise rises rapidly.

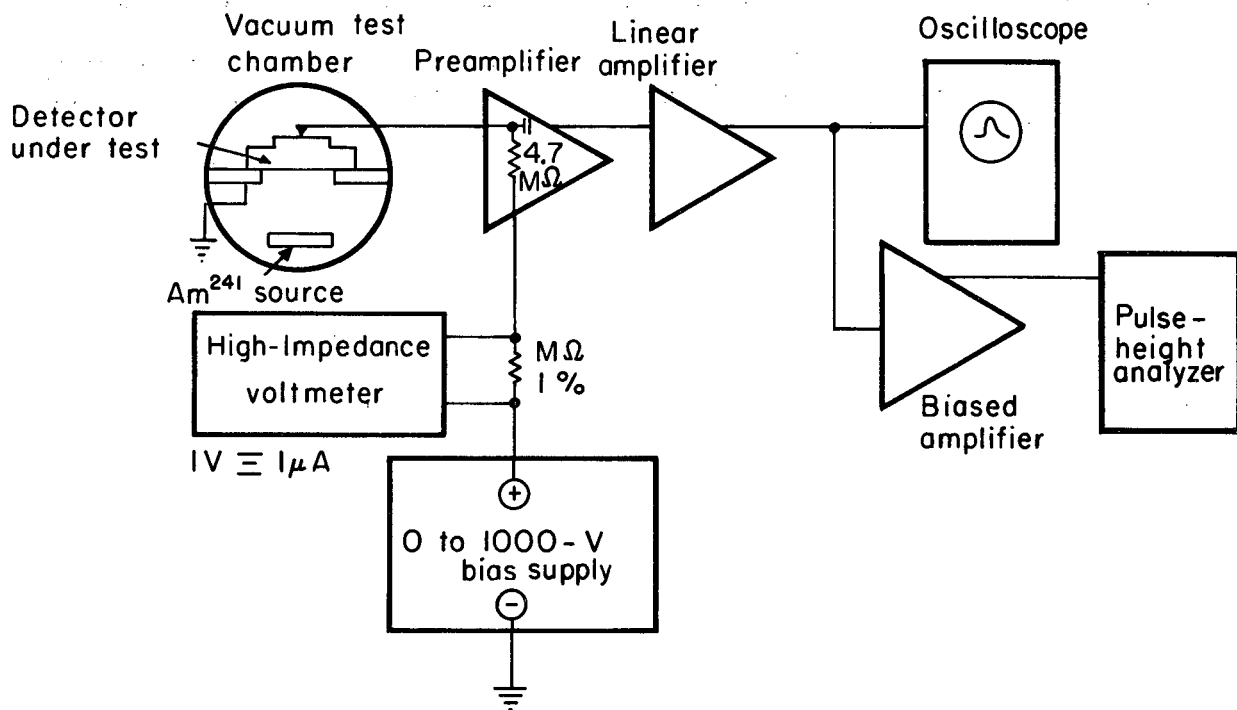
Longer soaking in hydrofluoric acid followed by a wash with a minimum amount of methyl alcohol will effect the reverse of the above.

When a suitable compromise between leakage current and noise at the proper bias voltage, has been obtained, proceed with step 3 below.



MUB-4762

Fig. 2. Lithium-drifted silicon device. Thickness: 0.5 to 3 mm in 0.5-mm steps.



MUB-6682

Fig. 3. Block diagram of apparatus.

3. Coat the junction edges and drifted surface with polyurethane varnish (Sherwin-Williams Marvethane), and allow to dry overnight in a clean, covered dish.

FOOTNOTE AND REFERENCE

\*Work done under the auspices of the U. S. Atomic Energy Commission.

1. Fred S. Goulding and William L. Hansen, An Automatic Lithium Drifting Apparatus for Silicon and Germanium Detectors, IEEE Trans. Nucl. Sci. 786, June 1964.

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