Fabrication of lithium-drifted silicon detectors by constant temperature method

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Abstract. A new approach for lithium drifting in silicon is described where the silicon devices under drift are held at constant temperature and bias at normal air ambient, and the drift process is terminated at the end of an estimated time depending upon the thickness of wafers. A 4-channel lithium drifting unit with electronically controlled oven has been constructed for this purpose. Full details of the fabrication procedure are given. A sizable number of Si(Li) detectors have been fabricated using this approach. The quality of the detectors is tested with ^{241}Am alphas and conversion electrons from ^{209}Bi and ^{137}Cs sources. The detectors are regularly used for nuclear physics experiments at this Centre.

1. Introduction

Semiconductor detectors are being increasingly used for the last 25 years mainly because of their high resolution and compact size. The first detectors of this kind to be used were diffused P–N junction and surface barrier types using silicon single crystals. The thickness of the active region of the device where the ionizing radiation loses energy, contributing to the pulse height, is given by the relation,

$$W = 0.5(\rho V)^{1/2} \text{ microns,} \tag{1}$$

where ρ is the resistivity in ohm-cm and V is the applied bias. Therefore, the thickness of the depletion (active) region is limited by the resistivity of the starting material and the voltage which may be applied and this thickness does not exceed 1 to 1·5 mm in general. Such a thickness is found to be too small for many applications in charged particle spectroscopy. It is, therefore, necessary to go for an artificially created intrinsic region by lithium compensation of P-type silicon, thus realizing N–I–P–diode structure as was first demonstrated by Pell (1960). The rate of growth of compensated region in silicon

$$R = dW/dt = \mu_L E = \mu_L V/W,$$

or $\qquad W = (2\mu_L Vt)^{1/2},$ $\hspace{3cm}$ (2)

W, the thickness of the compensated region in cm is thus approximately given by (2) where the loss of lithium is ignored; μ_L is the mobility of lithium ion in silicon at the drifting temperature in cm^2/V-sec, V the applied reverse bias and t is the time of drift in seconds.

Many drift methods have been described in the literature, of them the ones of particular interest are the automatic lithium drift method described by Goulding and Hansen (1964) and drifting of silicon using a boiling liquid between 120 and 150°C by Mann *et al* (1962) and by Ammerlaan and Mulder (1963). In this article we describe a method where the drifting is done under normal atmosphere and the oven is maintained at a constant temperature. Batch production of lithium drifted silicon detectors is feasible by using this method. Since the drifting time increases as the square of the detector thickness, drifting very thick silicon detectors is difficult because lithium ions are lost at the surface and they precipitate in the bulk during drift. Hence a thickness of 5 mm is generally the upper limit for silicon detectors.

2. Fabrication of Si(Li) detectors

2.1 *Material selection and processing*

As lithium is an interstitial donor impurity in silicon, only P-type material can be used for lithium compensated devices. The required properties of silicon single crystal are given in table 1.

The silicon material having the above mentioned properties (supplied by M/s. Wacker Chemitronics, West Germany) was used. The required number of slices (in our case, four) about 0·5 mm thicker than the desired finished thickness (1 to 5 mm) is cut from the ingot using semiautomatic diamond wheel saw. The slices are hand-lapped scratch-free with 20 and then 5 μ alumina powder. The slices are cleaned free of any powder or dust using high purity trichloroethylene, acetone and water; and dried using nitrogen gas jet.

2.2 *Lithium diffusion*

Lithium is a fast diffusant in silicon having values of diffusion coefficient $1\cdot5 \times 10^{-8}$ cm²/sec at 350° and 3×10^{-8} cm²/sec at 400°C. In comparison the diffusion coefficient of phosphorus in silicon at 1200°C is about 10^{-12} cm²/sec. Lithium had earlier been diffused by painting one surface of silicon wafers with lithium emulsion in oil. But the modern method of lithium diffusion is evaporation and diffusion under high vacuum which is universally followed as reported by Mann *et al* (1962), Blankenship and Borkowski (1962) and Dearnaly and Lewis (1964). In our case,

Table 1. Specifications of silicon crystals for lithium drifting application.

Property	Value
Type	P
Dopant	Boron
Resistivity	100–2000 ohm-cm
Minority carrier lifetime	500–2000 μs
Oxygen content	$< 10^{15}$ atoms per cm³
Etch pit density	< 500 cm^{-2}
Diameter	20–50 mm
Orientation	$\langle 111 \rangle$

evaporation and diffusion are carried out simultaneously by preheating all the four slices to a temperature of 350–400°C in the vacuum evaporator at a pressure of 1×10^{-5} torr and then evaporating about 250 mg of lithium metal from a tungsten filament kept at a distance of 10 cm above the slices. The evaporation is done for about 90 sec and the diffusion is carried out for a further 3 minutes, after which the slices are suddenly cooled by passing water through a cooling coil welded to the hot plate. The estimated rate of lithium evaporation is about 100 Å/sec resulting in a heavy deposition which serves as a constant source for diffusion and a reservoir for drifting at a later stage. The wafers are removed from the vacuum evaporator and dipped in methyl alcohol to dissolve excess lithium at the surface. The number of diffused lithium atoms must be larger than the number of acceptors in the desired compensated thickness W. Blankenship and Borkowski (1962) had pointed out that the sheet resistance R_s, per unit area, must be much less than $1/N_A \mu_e Wq$ where N_A is the acceptor concentration in the starting material, μ_e is the electron mobility in silicon and q is the electronic charge. Diffusion at the above mentioned parameters yields junction depths of 100–150 μ and a sheet resistance of 1–10 ohm per square which correspond to a surface concentration of more than 10^{17} per cm^3 of lithium.

The lithium-diffused region constitutes a dead layer for the charged particles at the back of the detector whose thickness is required to be known for some applications. This thickness, which is nothing but the junction depth, can be calculated by using complementary error function distribution for the case of diffusion from an ultimate source:

$$N(x, t) = N_0 \, \mathrm{erfc} \left[X/2(Dt)^{1/2} \right]$$

yielding a junction depth of

$$X_j = 2(Dt)^{1/2} \, \mathrm{erfc}^{-1} (N_A/N_0), \tag{3}$$

where D is the diffusion coefficient, t is the diffusion time and N_0 is the surface concentration of lithium. The diffusion depth can also be experimentally determined once the device is completely drifted by using a conversion electron source like [137]Cs. This method is completely non-destructive.

2.3 *Pre-drift processing*

The chemicals mentioned in the following procedure are invariably BDH make (electronic grade), the deionized water is 18 Meg-ohm-cm resistivity and gases are Indian Oxygen make (IOLAR-I grade).

(i) Mount the wafers on a carbon block with lithium diffused face upward using Shellac Wax. With the help of ultrasonic impact grinder cut groove of 12 mm I.D. 0·75 mm width and upto a depth 1 mm less than the total thickness of the wafers but about 0·5 mm deep in case of 1 mm wafers.

(ii) Remove the wafers from carbon block, clean in methyle alcohol. Lightly hand lap the wafers on both faces and wash using distilled and deionized water and jet dry using nitrogen gas. With the help of masking machine draw a circular strip of about 14 mm I.D. on the opposite face to the lithium diffused one using piecine wax dissolved in xylene with a brush. Mount the wafers on a teflon etching stick with the masked face upward using piecine wax.

(iii) Etch the wafers in 3 : 1 HNO_3 : HF solution for 3 min, quench in isopropyl alcohol and then wash in running deionized water. Clean the wafers in trichloroethylene and then in methyl alcohol and jet dry.

(iv) Evaporate 200 Å gold on the etched "well" side at a pressure of about 1 $\times 10^{-5}$ torr. This thin gold evaporation on etched silicon serves as surface barrier ohmic-contact for the lithium P–N junction diode and also as a thin window entrance for the charged particle once the device is completed. Mask the central etched region and evaporate 600 Å gold layer over the rest of the area. Evaporate 600 Å gold on full area of lithium-diffused face also.

(v) Protect the lithium-diffused face with piecine wax except the grooves and mount the wafers on teflon etching stick. Remove the gold layer from the grooves with the help of Aqua Regia solution, wash in deionized water and dry. Etch the grooves for 3 min in 3 : 1 HNO_3 : HF, Quench in isopropyl alcohol, wash and clean the wafers as metioned earlier. Treat the grooves with methyl alcohol for 30 sec and jet dry. Now the wafers have been converted into P–N junction diodes and are ready for drifting.

2.4 *Lithium drifting*

As mentioned earlier a method of constant temperature drift under normal ambient has been developed where even batch production of detectors is possible. A 4-channel drifting apparatus has been constructed. Here the drift oven temperature is controlled electronically to give a continuous control of temperature within $\pm 0.2°C$ at the operating temperature for about a month at a stretch. The oven is heated by a number of cartridge heaters of 220 V and 50 W rating each and measuring 6 mm diameter and 50 mm long, thus enabling the flexibility in heater design and very quick replacement in case of heater failure. The oven can accommodate four devices at a time. Drifting of larger number of devices is possible by suitably modifying the oven and the drift power supply current rating.

The wafers are enclosed in the oven at room temperature. A reverse bias of 200 volts is applied on the lithium-diffused face and the leakage current is monitored for each device. Now the oven temperature is slowly raised in steps of 10°C to the required drift temperature of 110°C or 120°C. This period of temperature raising is generally dependent on the thickness of the wafer and is about a week for 5 mm device. Once the required temperature is achieved the bias is increased in steps of 100 volts over 3–4 hr to an ultimate value of 500 volts. Now the temperature and voltage are kept constant and the compensation of acceptor impurities by lithium ions is accomplished as a function of time. The drift time can be estimated by using equation (2) taking value of 8×10^{-11} cm^2/V-sec for μ_L at 110°C. But it may vary slightly from crystal to crystal because of variance of lithium losses in them. Experimentally-determined average values of drift times for good crystals in the units of kilo-volt-hours for different thicknesses and drift temperatures are given in table 2 which can serve as a guide for termination of the drift schedule.

The drift characteristics of a 5 mm thick detector at 110°C are shown graphically in figure 1. Here the ordinate is the drift-current but the abcissa is subdivided into three successive variables, i.e. temperature, voltage and time. First the increase in leakage current is determined by the increase in device temperature. Once the temperature is stabilized at 110°C the bias is increased from 200 to 500 volts. The drift current does not

Table 2. Drift time in kilo-volt-hour for different thicknesses of silicon at various temperatures.

Device thickness (mm)	Drift time at temp (°C)			
	110	120	125	130
1·0	9·75	6·0	5·0	4·0
1·5	23·5	14·5	12·0	9·5
2·0	43·0	27·0	22·0	17·5
3·0	98·0	62·0	50·0	40·0
4·0	180·0	110·0	90·0	74·0
5·0	280·0	174·0	140·0	115·0
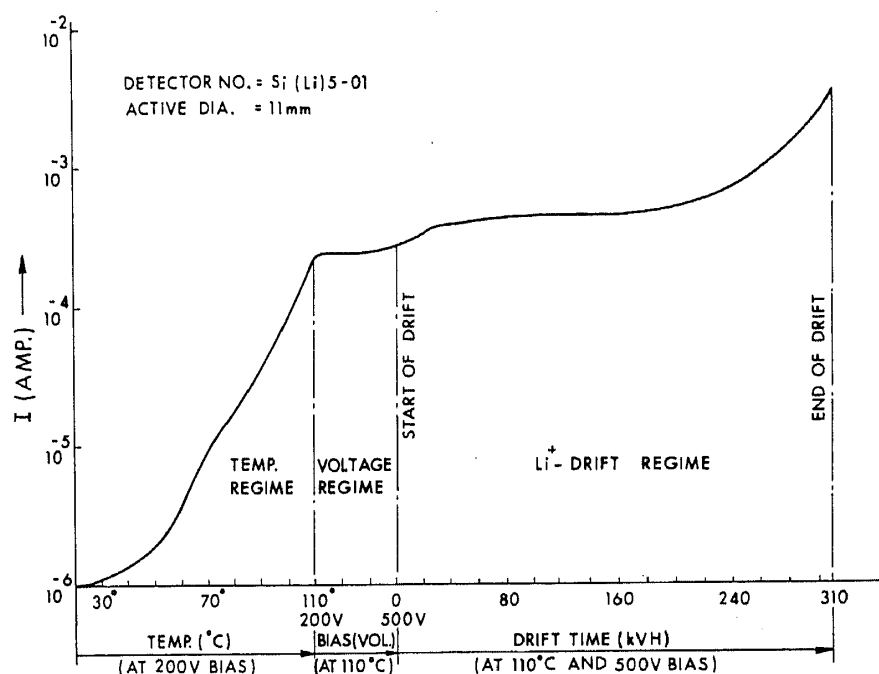
Figure 1. Drift-current characteristics of 5 mm thick Si(Li) detector.

increase appreciably with increase in bias as is clear in the voltage-regime of the graph which is a healthy sign of a good diode. In the time-regime the stability or very slow rise in drift current is a reflection on the starting material, proper lithium diffusion and the pre-drift procedures followed. Towards the end of the drifting when the compensated region under bias approaches the back contact, heavy injection of charge carriers occurs if the ohmic contact is not good; consequently a sharp rise in drift current is seen in such cases and the devices are invariably spoilt. Once the estimated time is over, the heater power is put off either manually or through a pre-set timer. Now the devices are allowed to cool slowly under bias to the room temperature and are removed from the oven after switching off the bias. The grooves are etched in 3:1 HNO_3:HF etchant for about 30 sec as described earlier and are treated with methyl alcohol after removing wax and cleaning in deionized water.

3. Testing of drifted detectors

The first concern once the drifting process is completed is to find out whether the drifted region has reached the back surface. This can be confirmed by one of the following two methods:

3.1 *Copper staining method*

The back surface is hand-lapped to remove the etched "well" region. The wafer is then immersed in 0·01 % copper sulphate solution to which a few drops of hydrofluoric acid are added. A thin layer of copper gets electroless plated on lapped silicon surface within a short time when a strong white light is shone on the surface. If the drifting in the central region had reached the back surface the differential copper plating on P- and intrinsic regions would clearly delineate these areas. This effect is shown in figure 2 where the central circular region is clearly visible which is the drifted region surrounded by the undrifted P-type region. The copper plating is removed with the help of nitric acid and the central well area is again etched and a thin layer of gold is evaporated and the detector is restored to its working condition but, of course, with a reduced thickness. The copper staining method is semidestructive and, therefore, is resorted to only if the second method does not give a positive result.

3.2 *Alpha pulses method*

The completion of drifting process can easily be tested using an alpha source like ^{241}Am. The alpha particles are directed on the back-etched surface of the detector kept inside the test chamber under a pressure of the order of 10^{-2} torr. The alpha pulse height through the standard preamplifier and amplifier is compared on the oscilloscope against the pulse height of a control detector having the same capacitance. This method is completely nondestructive and is sufficient for most of the detectors. The cross-section of a completely drifted Si(Li) is shown in figure 3 where the thickness may be 1 to 5 mm.
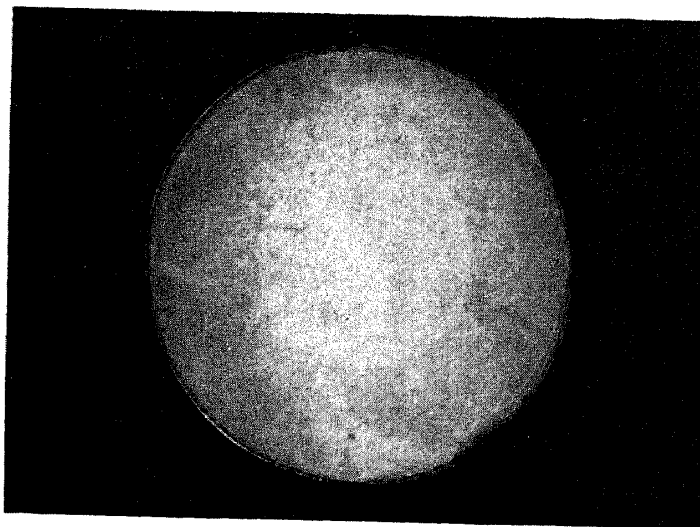

Figure 2. Photograph of a copper stained Si(Li) detector.
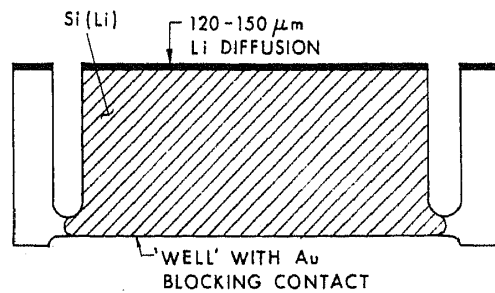
Figure 3. Cross-section of a Si(Li) detector with groove.

The degree of compensation achieved as a result of the drifting process which is reflected in the bias required for full depletion of the detector depends upon many factors like the starting material, lithium concentration at the surface, loss of lithium during drift and; the drift current and its stability. Full depletion voltages for different thickness of nicely compensated detectors are shown in table 3.

3.3 *Detector leakage current measurement*

The reverse bias leakage current of these devices has a direct bearing on the quality of the detectors as nuclear radiation spectrometers and, therefore, is invariably measured. The detectors are kept under a pressure of 10^{-2} torr for these measurements and the minimum bias to which the devices are subjected are shown in table 3 according to their thickness. The I–V characteristics measured for representative detectors of 2 mm and 5 mm thickness are shown in figures 4A and 4B respectively.

3.4 *Alpha resolution determination*

The detector and a standard 0·1 micro curie [241]Am alpha source are enclosed in a test chamber under 10^{-2} torr pressure. The detector is raised to the minimum bias voltage required for its thickness as given in table 3. Alpha spectrum is recorded using a standard test set-up consisting of pre-amp., spectroscopy amplifier and multichannel analyser. The full width at half maximum (FWHM) in keV for the 5·486 MeV [241]Am alpha peak is the standard yardstick quoted for indicating the quality of the detector as an alpha spectrometer. Figure 5 shows the alpha spectra of [239]Pu and [241]Am of a 3 mm

Table 3. Full depletion voltage and minimum testing voltage for different thickness of Si(Li) detectors.

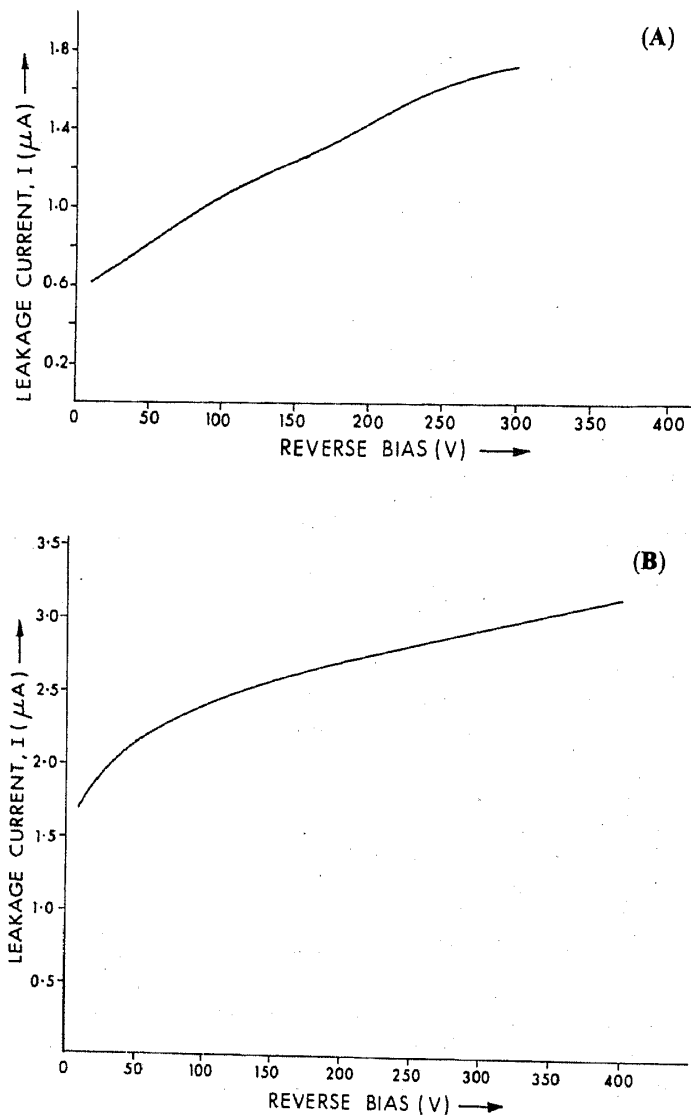| Detector thickness (in mm)	Full depletion voltage (volts)	Minimum testing voltage (volts)
1·0	40	200
1·5	60	250
2·0	80	300
2·5	100	350
3·0	120	400
4·0	140	500
5·0	160	600

Figure 4. I–V characteristic of Si(Li) detector, A. 2 mm. B. 5 mm.

Si(Li) detector. An FWHM of 38 keV for 5·486 MeV alpha peak is obtained in this case. Resolutions of 30 to 70 keV are generally achieved.

The alpha resolution of a detector is affected by the drift current behaviour and consequently by the I–V characteristic. A detector which drifts at a lower value of drift current generally exhibits a better resolution than the one which drifts at higher current value.

3.5 Electron resolution

The response to conversion electrons at room temperature is also measured using [207]Bi and ^{137}Cs electron sources. Figures 6A and 6B show these spectra. Resolutions of the order of 14 to 25 keV have been achieved. A system for cooling the detectors up to 77°K is under construction which will help in studying the behaviour of cooled lithium drifted silicon detectors for conversion electrons.
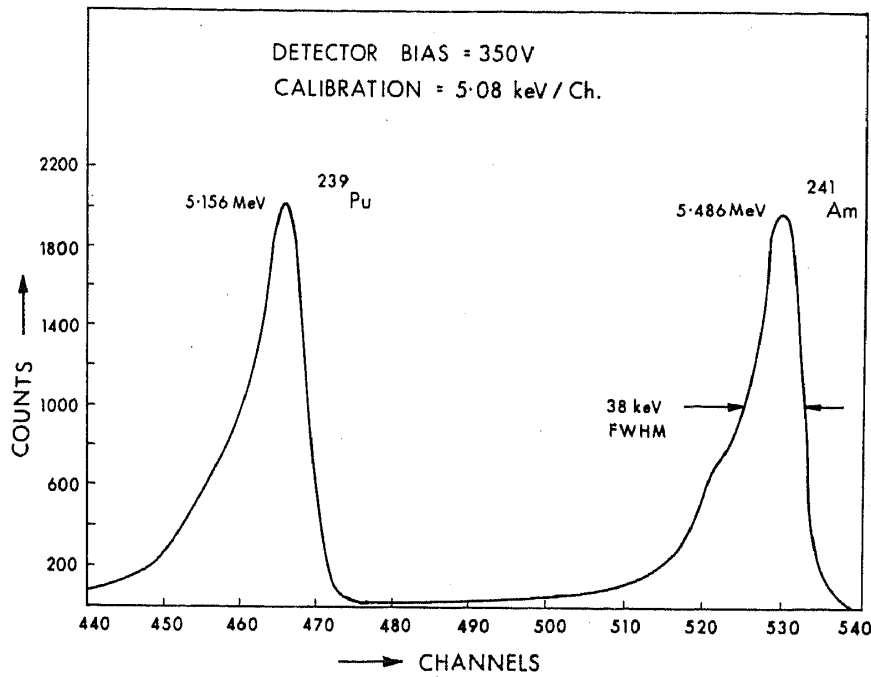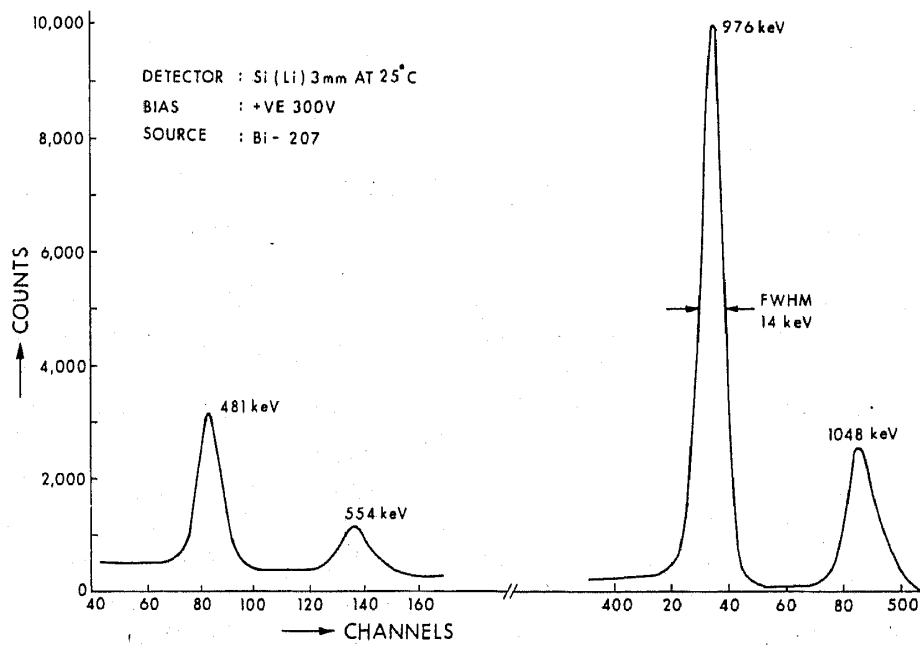
Figure 5. Alpha spectrum with 3 mm Si(Li).

Figure 6A. ^{207}Bi Monoenergetic electron spectrum with Si(Li) detector.

The grooves of the detectors which are found suitable are protected with KMER (Kodak micro etch resist). The finished detectors are then stored under oxygen ambient and at a reverse bias of 150 to 200 V.
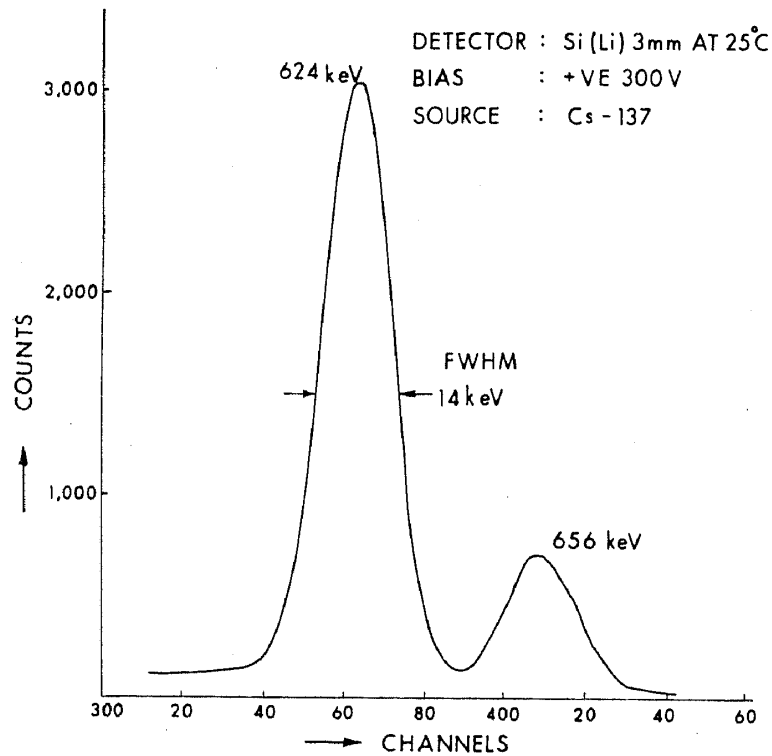
Figure 6B. ^{137}Cs monoenergetic electron spectrum with Si(Li) detector.

4. Utilization of detectors

The variable energy cyclotron (VEC) at Calcutta is designed to accelerate protons, deuterons, alphas and suitable heavy ions. The machine consistently delivers alpha beams of 25 to 75 MeV to the users since January 1981. The lithium-drifted silicon detectors developed at the laboratory here have been used for a wide range of nuclear physics experiments. Figure 7 is a representative spectrum of elastic-inelastic scattering of alpha particles from ^{12}C and ^{16}O in a 500 μg/cm^2 thick Mylar target (Dutta *et al* 1985). Similarly figure 8A is the representative two-dimensional plot of ΔE vs E showing P, d, t, ^{3}He and alphas as nuclear reaction products of ^{27}Al with 40 MeV alpha projectiles. Figure 8B shows the linear energy spectrum of tritons from ^{27}Al (α, t) ^{28}Si showing the ground state and a few excited states of ^{28}Si (Banerjee *et al* 1984).

Si(Li) detectors are also useful for beam diagnostics. Figure 9 shows the alpha-RF time spectrum obtained using the elastically-scattered alphas from gold target. This spectrum shows the multi-turn extraction nature of the machine. It was taken to obtain the time structure of the VEC beam and subsequently gate the energy spectrum with respect to a specific portion of the time spectrum to improve the beam energy resolution. The effective incident beam resolution improved from 127 keV to 63 keV for 40 MeV alphas using this technique (Bhandari *et al* 1985).
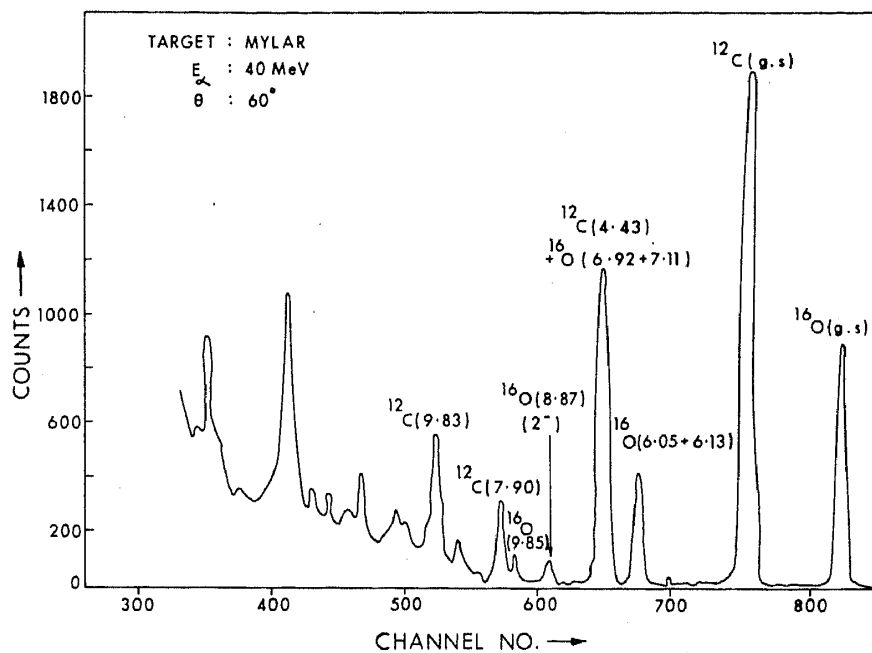
Figure 7. Elastic-inelastic scattering of alphas from ^{16}O and ^{12}C in a Mylar target.

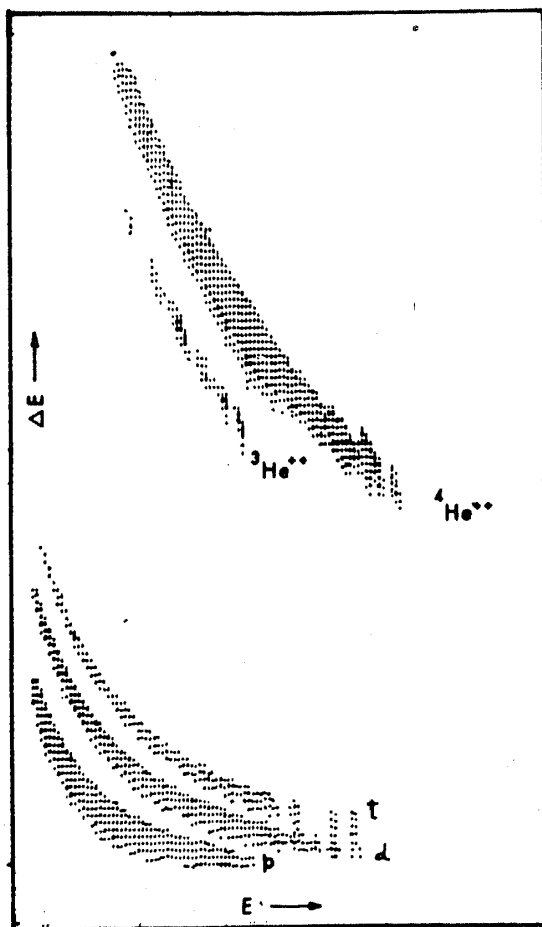**Figure 8A.** Two-dimensional ΔE vs E plot for transfer reaction study of alpha on ^{27}Al($E_\alpha = 40$ MeV).

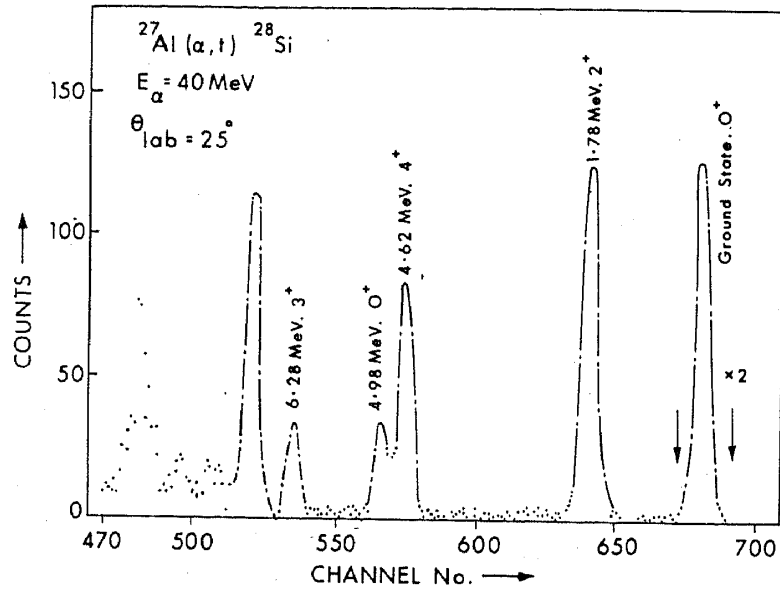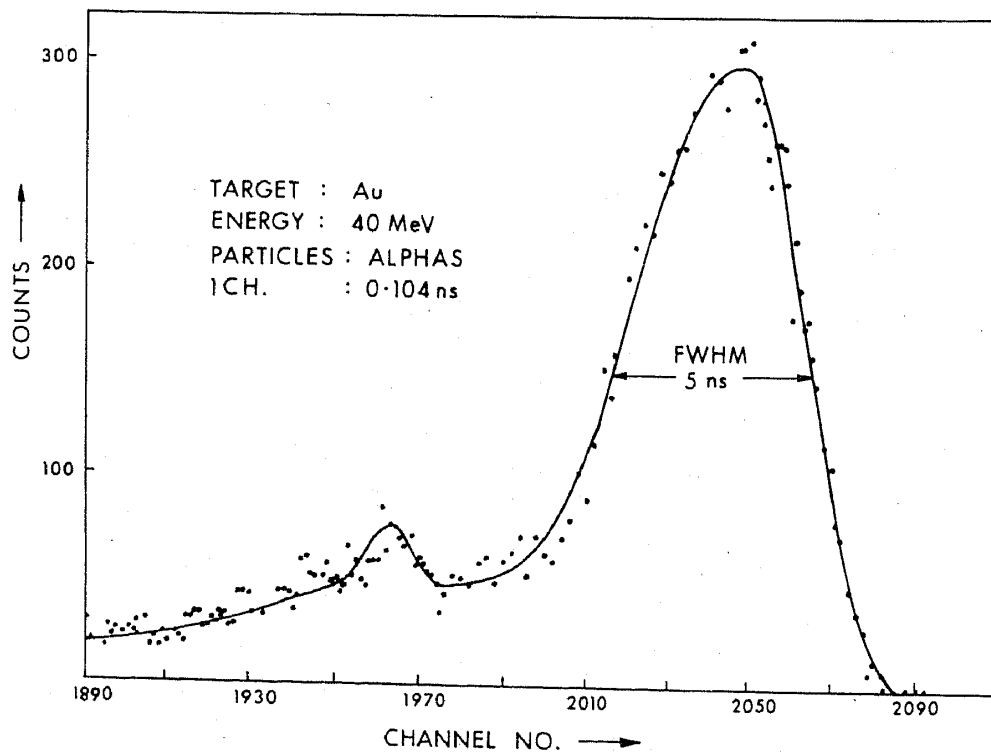**Figure 8B.** Triton energy spectrum for the transfer reaction $^{27}Al(\alpha, t)\ ^{28}Si$.

Figure 9. Alpha-RF time spectrum for VEC external beam.

5. Conclusion

Over 50 Si(Li) detectors have been fabricated using the constant temperature method described in this paper. These have been used satisfactorily by various experimental groups working with VEC. Efforts are on to further improve the quality of these detectors and for fabrication of larger area detectors.

Acknowledgements

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