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THIN EPITAXIAL SILICON DETECTORS

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Presented at the 5th European Symposium
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Abstract: Manufacturing procedures of thin epitaxial surface barriers will be given. Some improvements have been obtained: larger areas, lower leakage currents and better resolutions. New planar epitaxial dE/dX detectors, made in a collaboration work with ENERTEC-INTERTECHNIQUE, and a new application of these thin planar diodes to EXAFS measurements, made in a collaboration work with LURE (CNRS,CEA,MEN) will also be reported.

1. Introduction

Techniques for fabricating thin silicon films for dE/dX diodes used in particle identification telescope systems have been reported in the 1960s [1,2]. Thinning was performed by very time-consuming processings: lapping, polishing and slow planar-etching. Many problems have arisen in handling very thin and brittle wafers and in the deep etching process which requires severe control of the etching rate to avoid thickness variations.

In 1970, it was found by H.J.A. Van Dijk [3] and M.J.J. Theunissen [4] that preferential electrochemical etching of suitable epitaxially grown structures can be used to make thin silicon crystals and can have application in thin device technology.

In 1971, R.L. Meek [5,6] described a method of thinning N/N⁺ epitaxial silicon wafers by selective anodic dissolution with some applications to integrated circuit technology and presented also a preparation of self-supported, large area, uniformly thin silicon films [7].

The first thin dE/dX surface barrier detector made on a thinned epitaxial silicon was produced by J.P. Ponpon et al. [8] in 1973 and an optimization of their experimental conditions was attempted and reported in the thesis of A. Tetefort [9].

Growing interest in using electrochemical etching has been observed in the late 1970s, and not only the feasibility of this technique has been confirmed [10,11,12,13] but thinned epitaxial detectors have also been commercially available.

Another thinning method, this time by chemical preferential etching of the epitaxial N/N⁺ structure, has been reported [14,15,16] and applied to the fabrication of thin detectors.

During the 1980s, the so-thinned epitaxial detectors disappeared both from the market and from many laboratories, probably due to a lack of reliability of the starting material which indeed was not of a detector grade quality.

In our Institute we have fabricated and used thin epitaxial dE/dX surface barriers since 1975 for heavy ion experiments [17], with thicknesses ranging from 1 to 50 μm and areas of up to 3 cm^2 . Our experience in a laboratory production of epitaxial silicon detectors allows us to say that electroetch thinning remains an easy, straightforward way to obtain economically and in a short time, especially with the today available starting material, a high quality thin detector with preset and uniform thickness over a large area.

The aim of this paper is twofold: first, to give a fast manufacturing procedure for a laboratory production of surface barriers with the related detection performances, secondly, to show that with the today high quality epitaxial silicon, new applications have also become possible. We will thus report some recent developments we have made in our Institute: a new planar epitaxial silicon detector for an industrial production and a new application to EXAFS (Extended X-ray Absorption Fine Structure) measurements.

2. Anodic dissolution of N/N⁺ epitaxial silicon

Early investigations in the electrochemical properties of silicon and germanium have been performed at the Bell Telephone Labs (USA) in the 1950s [18,19] and also at the Philips Research Labs (Europe) [20].

It has been found that P type and N⁺ type silicon can be electrochemically thinned in HF solutions. Anodic dissolution is governed by the supply of holes and by the mass transfer of fluoride ions to the electrolyte-silicon interface.

Holes are assumed to be generated at the heavily doped N⁺ type surface by avalanche or Zener breakdown by M.J.J. Theunissen [21], or by a surface generation process taking into account tunnel transitions in the very narrow space charge region of the electrolyte-silicon junction, by R.L. Meek [22].

Thus one can distinguish three resistivity regions, each with a different etching behavior: for lower values than 0.02 Ω .cm, complete dissolution is obtained; in the range between 0.02 Ω .cm and 0.3 Ω .cm, only partial dissolution occurs with formation of a thick and insoluble film; for resistivities higher than 0.3 Ω .cm the silicon is not attacked, due to the lack of holes.

The convenient structure for thin detector fabrication purposes is that of N/N⁺ epitaxial silicon where the resistivity of the N⁺ substrate is lower than 0.02 Ω.cm and that of the epilayer N, greater than 10 Ω.cm, with the condition of a steep impurity concentration gradient at the N/N⁺ interface. The uniform low resistivity condition of the substrate is a critical one because any region of higher resistivity will result in remaining islands of undissolved silicon on the thin epilayer film.

Fluoride ions reach the silicon surface by diffusion and convection from the solution bulk and are used up in the anode reaction to give fluosilicic acid, which is soluble and has no influence on further dissolution. The supply of fluoride ions can be increased by stirring, by nitrogen bubbling, by magnetic agitation or by using a rotating anode. It is well known in electrochemistry that mass transport from the electrolyte bulk to the anode surface can be controlled by the revolution rate of the anode disk [20]. One can also use a rotating anode moved slowly by hand, from time to time, to get a radial symmetrical dissolution [23]. Our anode holder contains only one sample of silicon to be dissolved and C.J. Maggiore et al. have used a multielectrode anode which contains a standard wafer on which nine films can be thinned at once and that is probably a solution more suited for an industrial production [11].

Some problems, such as remaining islands of undissolved substrate material, could arise in the anodic thinning of especially large area films. It is difficult to keep the dissolution rate of the N⁺ material uniform over the whole anode surface. In that case, it is preferable to have the dissolution proceeding radially outward. Therefore we have investigated electrolyte-jet techniques, close proximity cathodes, high anode rotation rates, virtual cathodes, and so on. The best results were obtained with a radially delaying action by proceeding step-wise in

using a sequence of annular masks with increasing inner diameters. Each mask was removed after a dissolution time of about 15 mn. A starting structure obtained for instance, with the use of two masks for a 3 cm^2 dissolution is represented in fig.1.

On the contrary, the other remaining electrochemical etching conditions are not so severe. Anode potentials of 5 and 6 V have been reported by R.L. Meek, and values ranging from 3 to 12 V have been used by other authors. The anode potential can be measured against a saturated calomel reference electrode. Current densities are related to the reaction rate at the electrolyte-silicon junction and thus will depend upon area, resistivity, HF concentration, agitation and temperature. With 5% aqueous HF solution and when working at room temperature, the current densities range from 50 to 150 mA.cm^{-2} .

3. Starting material

Since 1975 we have tried and used epitaxial slices coming from many manufacturers. In table 1. we give only the specifications of N/N⁺ slices with which we could obtain, with a high yield, good thin detectors, and which one could always buy in small quantities. The different trademarks are given in a chronological purchase order and one can notice the increasing resistivities with time.

Today available high epilayer resistivities allow also auto-depletion. These detectors can operate while fully depleted without any leakage current. Their internal built-in electrical field extends over the whole thickness and will make them more efficient when operating in a photovoltaic mode. With resistivities of up to around $2 \text{ k}\Omega.\text{cm}$ we intend to achieve in the near future auto-depleted diodes with thicknesses of up to $20 \mu\text{m}$.

4. Fabrication procedure of epitaxial surface barriers

After the electroetch thinning, the wafer was cleaned and was submitted to a surface treatment to insure stability in changing ambient conditions. We wanted, both to avoid putting varnish or epoxies for edge protection on thin crystals and also to keep the gold surface as small as possible to minimize the capacitance of the thin diode. For this purpose, we used a silver-glycol etch [24], a slow etch rate solution, which gave us surface barriers of long-term stability in the range of several years. It has allowed us to fabricate $5\ \mu\text{m}$ thin multistrip diodes for astrophysical applications and some other multidetector configurations. The silver-glycol etch has been used before at the Bell Telephone Labs for engraving silicon patterns.

We have obtained quite similar results with a tin oxide (SnO_2 or SnO) deposit of about $200\ \text{\AA}$ thick before the gold evaporation. It has been already reported that tin oxide increased vacuum stability of surface barriers [25].

The back contact can be achieved either by an Al deposit or by an amorphous Ge layer followed by a gold evaporation rather than Al, as reported by J.B.A. England [26,27,28]. The Ge-Au non-injecting back contact often allows us to put high overvoltage but seems not so stable with time.

Connections to the electrodes were made by means of silver paste and $50\ \mu\text{m}$ diameter gold wire. The bonding was made on the annular thicker frame of undissolved silicon which allows easy handling and encapsulation. Typical epitaxial surface barrier designs are represented in fig.2.

5. Performances of thin epitaxial surface barriers

5.1. Transmission dE/dX detectors

We have fabricated several hundred thin epitaxial surface barriers for heavy ion detection and especially today, for experiments realized at the heavy ion accelerator GANIL. As performances depend on thickness and area for high capacitance diodes, we have presented their dimensions in fig. 3, and one can see that our detector capacitances range from 500 pF to 3 nF.

The most significant sources of linewidth broadening of the ΔE signal, are electronic noise and straggling in energy loss. At shaping times of around 1 or 2 μs , principally used in nuclear physic experiments, the main contributor to noise when operating with high capacitance diodes is serial noise.

On the other hand, the contribution of the straggling in energy loss was measured with an α -particle thickness gauge, and to obtain information about thickness uniformity we operate with a parallel α -beam over all the detector surface. For this purpose we used 8.78 MeV α -particles from a thorium active deposit, Th(C+C'), a very thin, unsealed, high intensity source.

A Th(C+C') α -particle spectrum obtained with an epitaxial surface barrier is shown in fig.4. The serial noise due to the high capacitance of about 2050 pF, with our preamplifier, was 43 keV, and the energy-loss straggling of about 61 keV we measured, corresponds to that given by Bohr's formula.

The low leakage current, less than 70 nA, and the high capacitance of the detector, about 10.5 μm thick and with an area of 180 mm^2 , are plotted against bias voltage in fig. 5. One can notice the fast depletion due to a high resistivity starting material, $\rho > 500 \Omega\cdot\text{cm}$, obtained at about 0.5 V.

Another α -particle spectrum obtained with a 9 μm thick diode but of larger area, 250 mm^2 , which was realized five years ago, is given in fig.6. Many of these detectors were fabricated in 1984 and in 1985 for the experiments of the exotic radioactive decay of some radium isotopes by ^{14}C emission made in our Institute [29,30]. These detectors have to operate in the high cryogenic vacuum of the supraconducting spectrometer SOLENO for very long periods.

A Th(C+C') spectrum given by a thicker epitaxial dE/dX detector is shown in fig. 7. The lower energy α -particles, of 6.05 MeV, come to rest in the detector while the higher energy particles, of 8.78 MeV, pass through the counter. One can notice the good detection characteristics both in the transmission mode and in the total energy absorption mode.

5.2. Total energy absorption detectors

In nuclear particle experiments one often needs detectors which can discriminate short range particles from a lower specific ionization radiation background. Epitaxial surface barriers can be used in a total absorption mode in which the heavily doped substrate will play the role of a N^+ non-injecting back contact enabling high electrical field. We have realized fission fragments epitaxial detectors in circular or annular configuration with areas of up to 4 cm^2 . The leakage current and capacitance-voltage characteristics of such an epitaxial surface barrier, 32 μm thick with an area of 30 mm^2 , is given in fig.8. The detector is depleted at around 8 V and can withstand an overvoltage of 200 V with a leakage current of less than 90 nA. It is overdepleted at 200 V, about 25 times the depletion voltage, and the corresponding high electrical field will give very short charge transit times of less than 300 ps. The resistivity of the 32 μm thick epilayer was higher than 800 $\Omega\cdot\text{cm}$ which corresponds to an impurity concentration of less than

10^{12} cm^{-3} . The epitaxial surface barrier is also a low noise detector and can have high resolution applications as shown in fig. 9.

6. New developments

6.1. Planar epitaxial thin dE/dX detectors

We have realized in collaboration with ENERTEC-INTERTECHNIQUE planar epitaxial thin dE/dX detectors. The planar process of J. Kemmer [31] was achieved by ENERTEC. Electrochemical thinning and characterization of the detectors were performed in our laboratory. The detectors were tested at the heavy ion accelerator GANIL [32] where they showed particle identification performances as good as surface barriers. They were also submitted to high intensity synchrotron X-rays at the LURE facilities where they operated in a photovoltaic mode and have proved to be superior to ion gas chambers [33,34].

Fabrication procedures and early results have been reported in the thesis of L. Lavergne-Gosselin [33]. Transmission detectors, 10, 30 and 50 μm thick have been realized. They showed very low leakage current. We have not found and have not awaited much more particle or identification performances with respect to the surface barrier analogue, because the contribution of the leakage current in the linewidth broadening remains quite small with regards to the serial noise due to the high detector capacitance. With a smaller capacitance, the planar epitaxial diode will have a better performance than the surface barrier, for instance this could be the case for microstrip thin detectors. The major and well known advantages of planar diodes over surfaces barriers will indeed be expected also with planar epitaxial diodes: lower leakage current, low noise, ultra-high vacuum compatibility, annealing possibilities of radiation damages, more sophisticated electrodes patterning allowing heavy ion discrete localization down to the micron range, and

so on.

6.2. Thin planar epitaxial diodes for EXAFS measurements

A conventional EXAFS experimental set-up is composed of two ion chambers, operating in current mode: the front ion chamber is a transmission detector which measures the incoming X-ray intensity, I_0 , the back ion chamber measures the value I transmitted by the sample under test as given in fig.10. The modulation of the absorption coefficient μ , $\mu x = \log(I/I_0)$, near an absorption edge K or L, is obtained against the energy of the X-rays.

The advantages of solid state detectors over gas ionization chambers are well known: high quantum efficiency, linearity, a wide dynamic range of about 10^{10} , small size, low cost, high vacuum compatibility, thin windows, low noise, cooling possibilities and without the disadvantages of ion gas chambers (microphonic noise, high voltage, gas supply and so on).

Photodiodes have already been used as back detectors in transmission, or absorption EXAFS experiments, where the front detector remained an ion chamber.

Another alternative is to use two planar diodes: the front detector will be a thin epitaxial planar diode and the back detector can be a 200 μm thick conventional planar detector and both can operate in a photovoltaic mode. The Cu K-edge EXAFS spectrum which we have obtained is given in fig.11. This first absorption EXAFS experiment, using two silicon planar detectors, the front detector being a new planar epitaxial 30 μm thin diode, has been done in a collaboration work between the laboratories of LURE and IPN of ORSAY and ENERTEC-INTERTECHNIQUE [35].

6.3. Future prospects

The highly developed planar technology associated with selective electroetch thinning can achieve epitaxial transmission diodes with more or less sophisticated electrode patterns, which could find applications in many investigation fields.

In the heavy ion detection field, ultra-thin microstrip epitaxial diodes could be of interest in heavy ion tracking.

For X-ray synchrotron applications, the possibility of EXAFS measurements with planar detectors has been demonstrated but could still be improved. Auto-depleted thin epitaxial transmission diodes will be more efficient and could also be used in X-ray intensity measurement or X-beam localization. Another point of interest is that epitaxial silicon has very low dielectric relaxation time and could be used in fast timing with a pulsed X-ray synchrotron beam or any other high intensity radiation.

It would also be interesting to investigate about the fabrication and use of epitaxial ultra-thin transmission picosecond photoconductors or picosecond switches. With thicknesses in the micron range they will probably be suitable for laser and heavy ion applications and in the range of 5 to 10 μm they will be more suited to X and γ or high energy particles. Gallium arsenide is already used in that new class of high-speed radiation detectors but like silicon, it could also be thinned by means of a selective anodic dissolution [7].

7. Conclusion

Today available high resistivity epitaxial silicon allows to realize high performance thin detectors either with surface barrier technology or with planar

technology. A fast and easy manufacturing procedure of thin epitaxial surface barriers has been described and can be applied to the fabrication of some special purpose detectors which could still be made in the user's laboratory. The so-called planar process, which is achieved by ENERTEC, can also be applied to thin epitaxial diodes and will offer all the well known advantages and possibilities of the high detection quality planar diode.

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TABLE 1.

Thickness X (μm) and resistivity ρ ($\Omega\cdot\text{cm}$) of the epilayer of some starting material used at Orsay.

<i>Manufacturer</i>	<i>Epilayer thickness</i> X (μm)	<i>Epilayer resistivity</i> ρ ($\Omega\cdot\text{cm}$)
Toyo Silicon Co.	2-14	10-50
ASM	6-10	30
Semimetals, Inc.	10-30	50-150
Epitaxy, Inc.	10	200
General Instrument	8-40	100-300
Spire Corporation	10-40	300-800

FIGURE CAPTIONS

- Fig.1 - Starting structure of a large area N^+ substrate after a pre-etch with first and second mask.
- Fig.2 - Typical epitaxial silicon surface barrier designs.
- Fig.3 - Area plotted against thickness of thin epitaxial dE/dX surface barriers which have been realized for heavy ion experiments in our laboratory since 1975,●, and those which are reported in the following references: Δ [8], \circ [9], + [10], * [11,13], Δ [14].
- Fig.4 - $Th(C+C')$ α -particle spectrum obtained with a $9 \mu m$ thick epitaxial dE/dX detector. Bias 4 V, leakage current $0.1 \mu A$, capacitance 2050 pF, area $180 mm^2$, shaping time 2 μs , collimation 15 mm diameter aperture.
- Fig.5 - Leakage current and capacitance as a function of bias voltage of an epitaxial dE/dX detector. Area $180 mm^2$, thickness $10.5 \mu m$.
- Fig.6 - $Th(C+C')$ spectrum obtained with an epitaxial dE/dX diode with a noise linewidth of 112 keV due to a capacitance of 2900 pF. Bias 8 V, leakage current $0.25 \mu A$.
- Fig.7 - $Th(C+C')$ spectrum obtained with a $37.4 \mu m$ thick epitaxial dE/dX diode. Bias 5 V, leakage current $0.1 \mu A$, area $170 mm^2$.
- Fig.8 - Leakage current and capacitance versus bias voltage of an epitaxial overdepleted E detector.
- Fig.9 - $Th C$ spectrum of a low noise epitaxial surface barrier. Area $30 mm^2$, thickness $32 \mu m$, bias 50 V, leakage current 60 nA, shaping time 1 μs .
- Fig.10 - Typical EXAFS experimental set-up.
- Fig.11 - Cu K-edge EXAFS spectrum obtained with two planar silicon diodes operating in a photovoltaic mode. The front detector is a new epitaxial planar diode, $30 \mu m$ thick and with a rectangular area of $26 \times 7 mm^2$.

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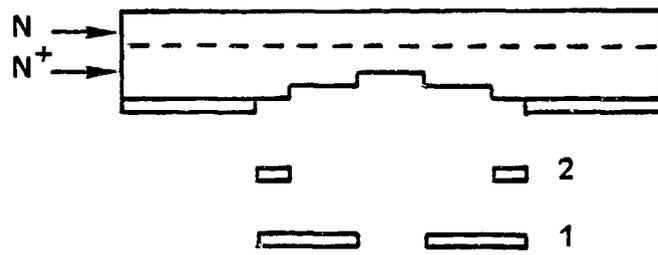


Fig.1

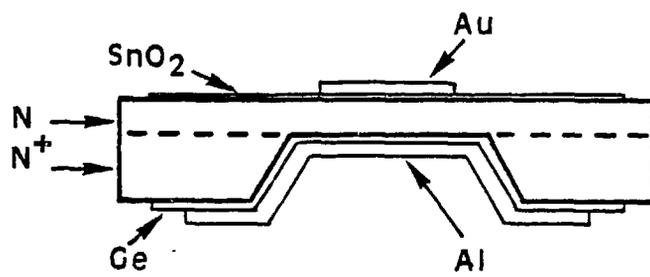
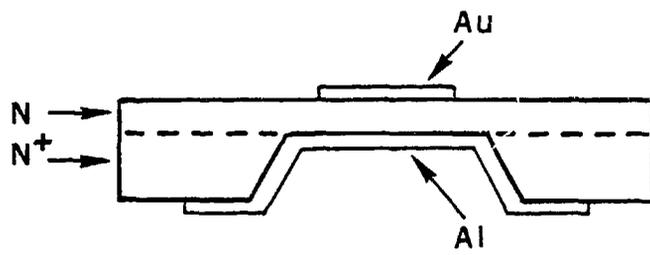


Fig. 2

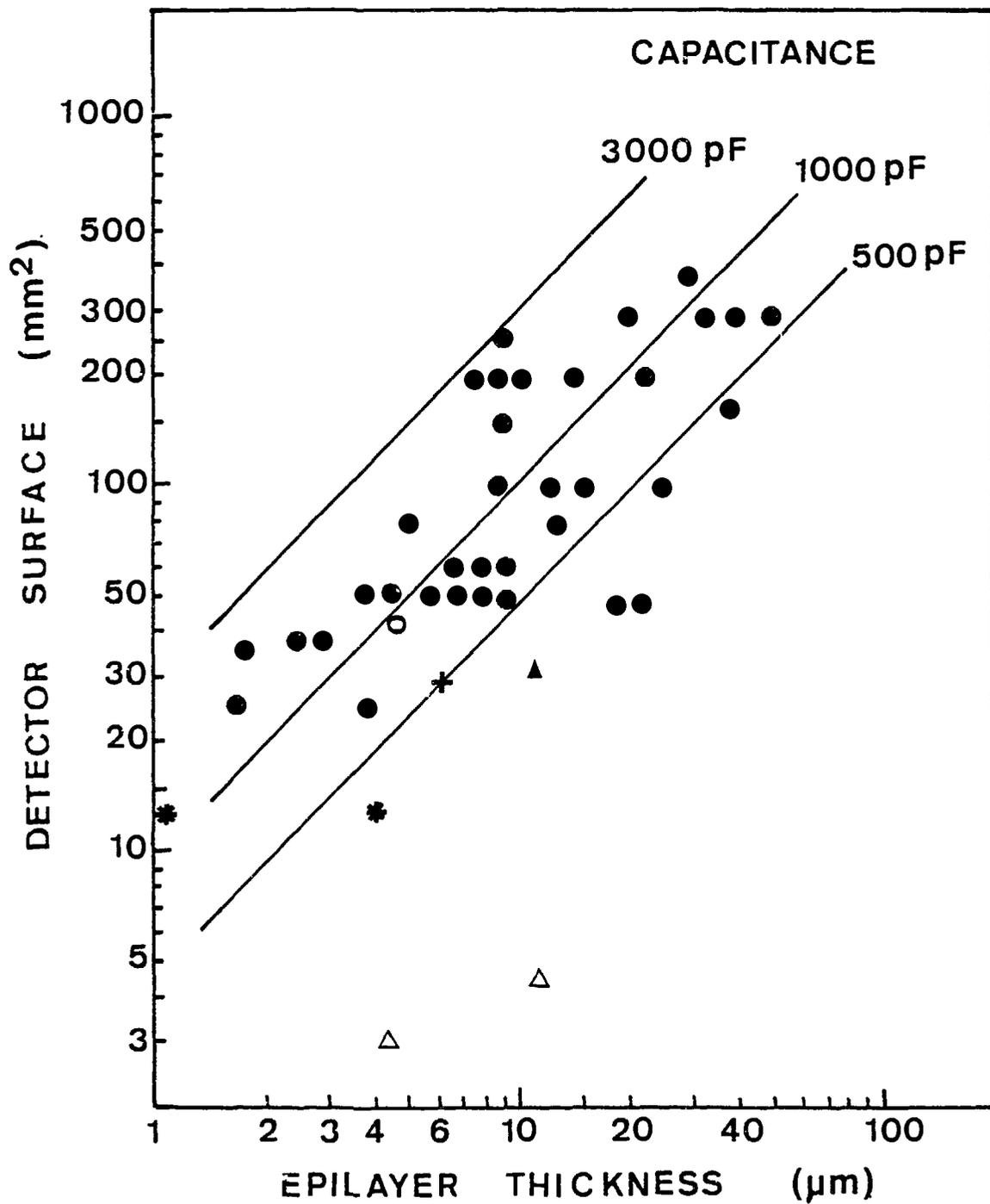


Fig. 3

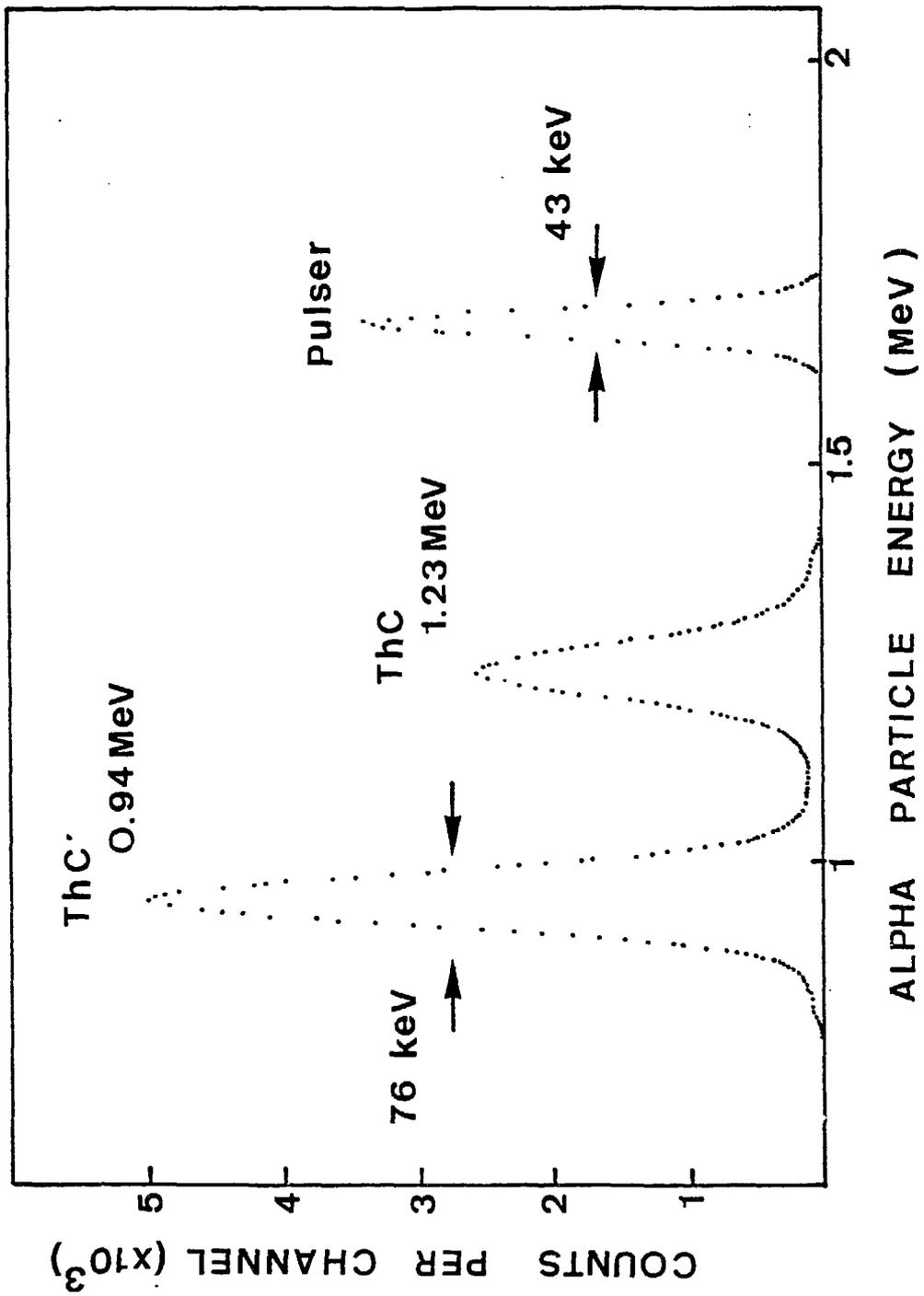


Fig. 4

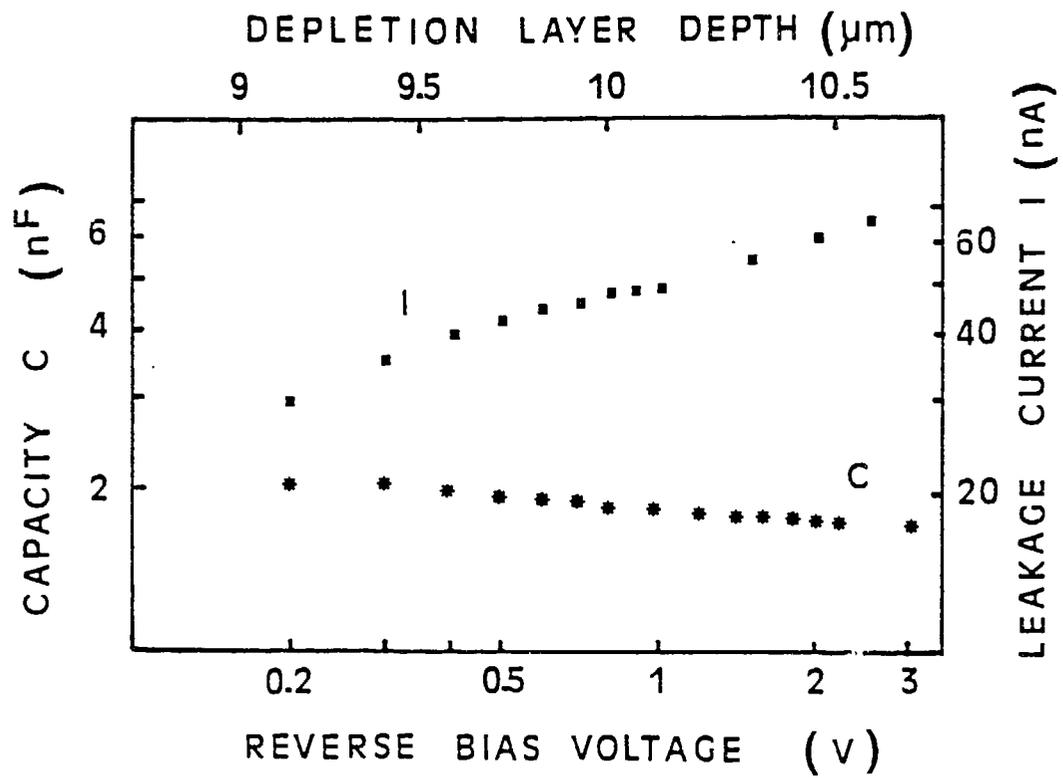


Fig. 5

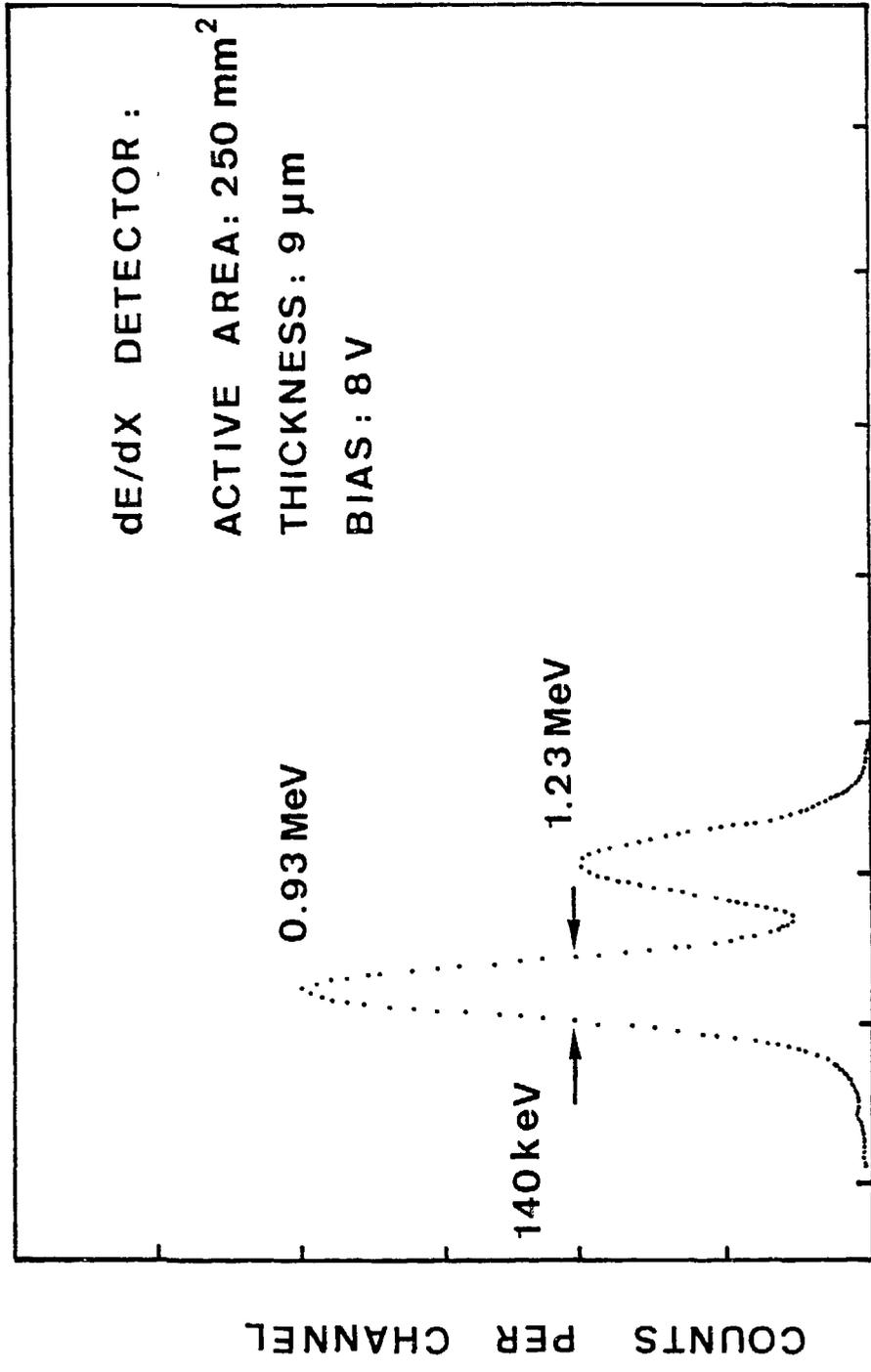


Fig.6

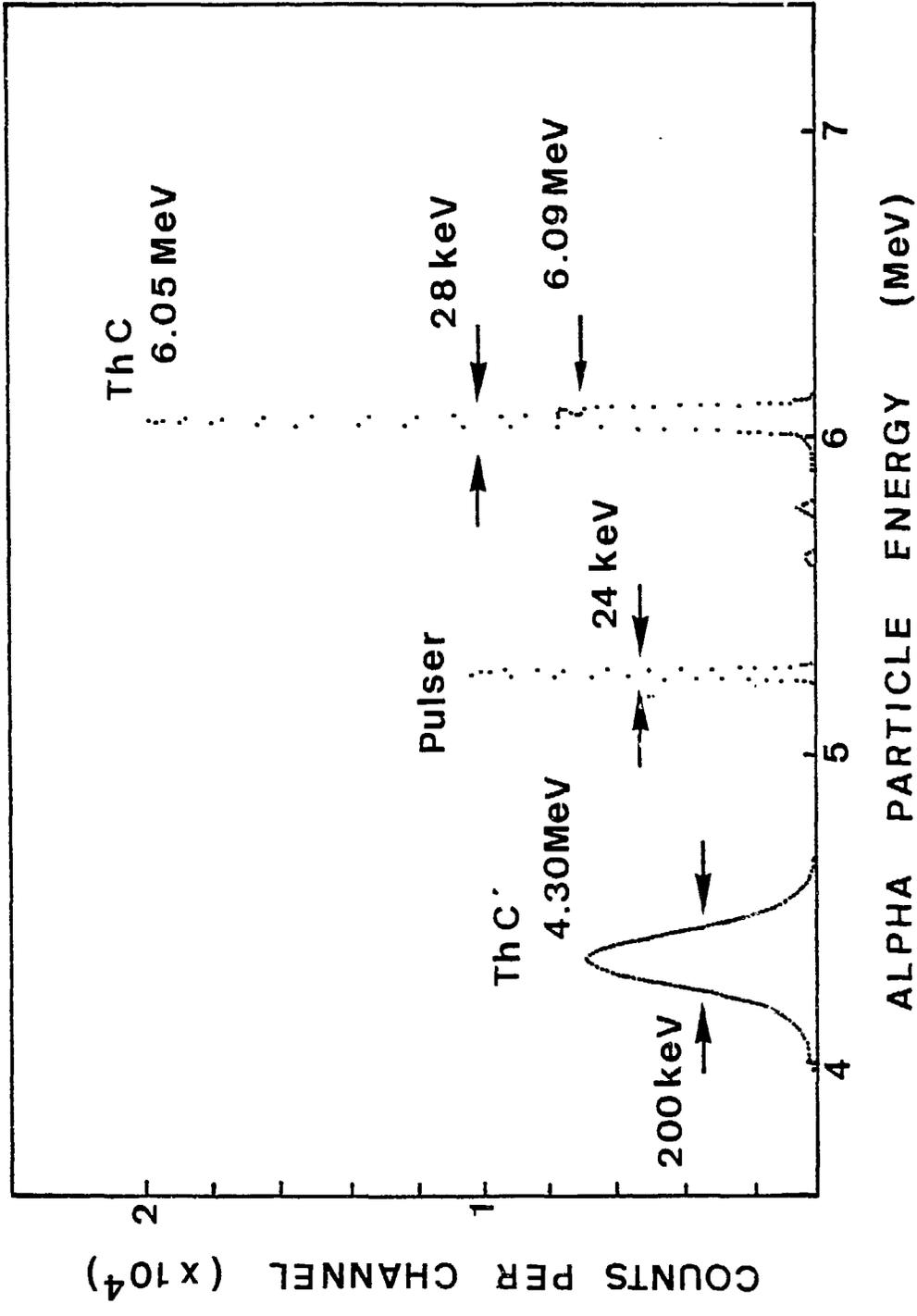


Fig. 7

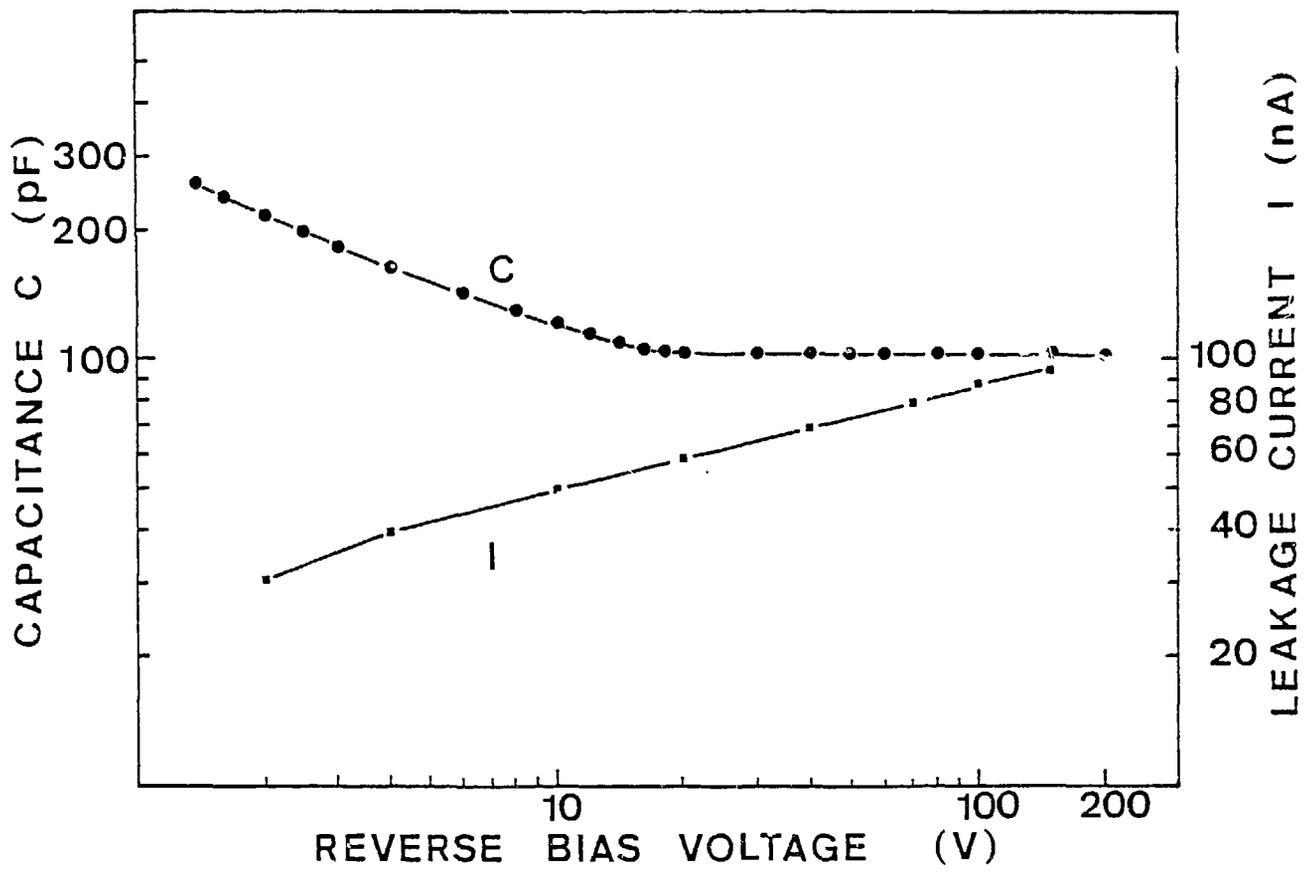
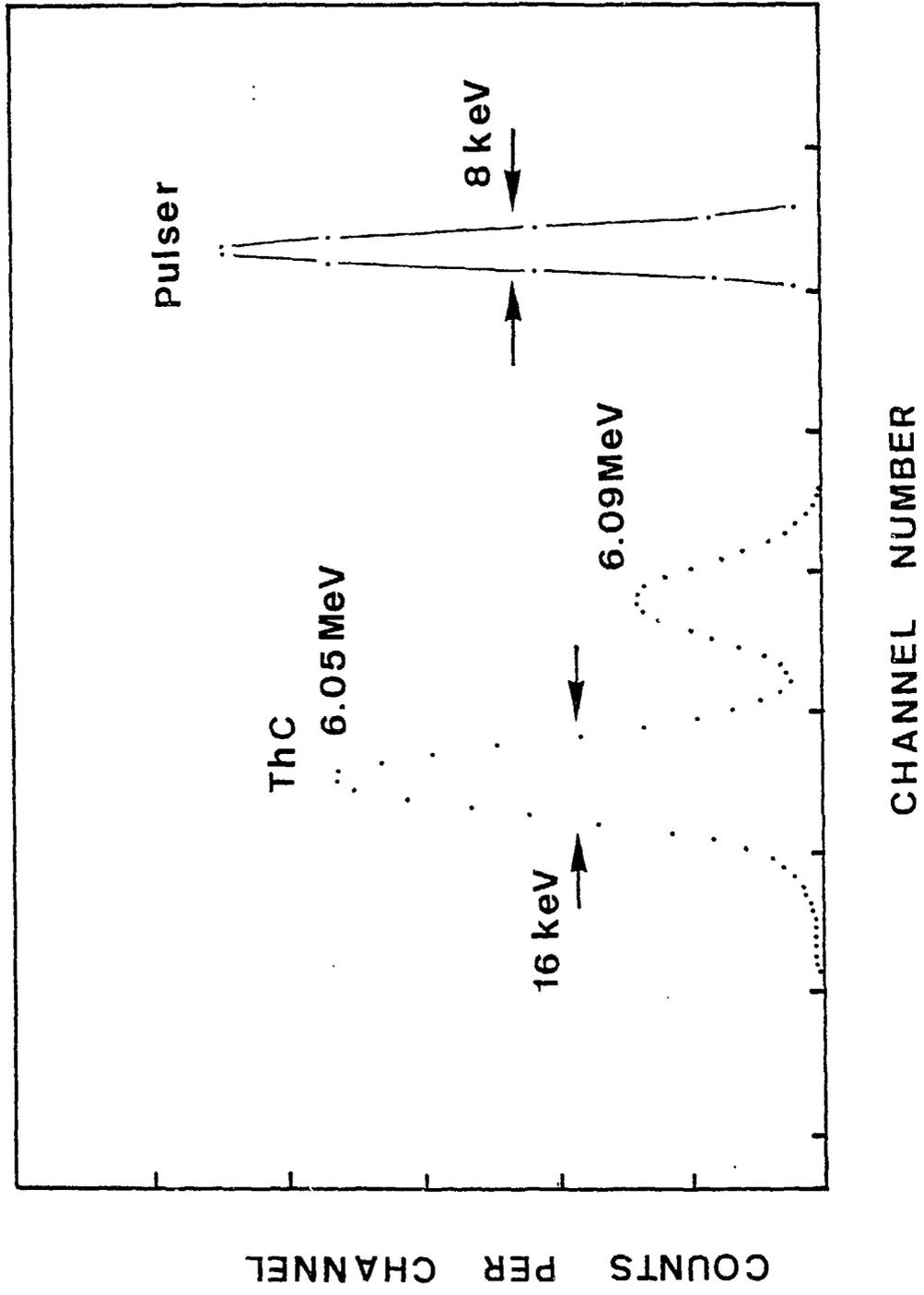


Fig. 8

Fig. 9



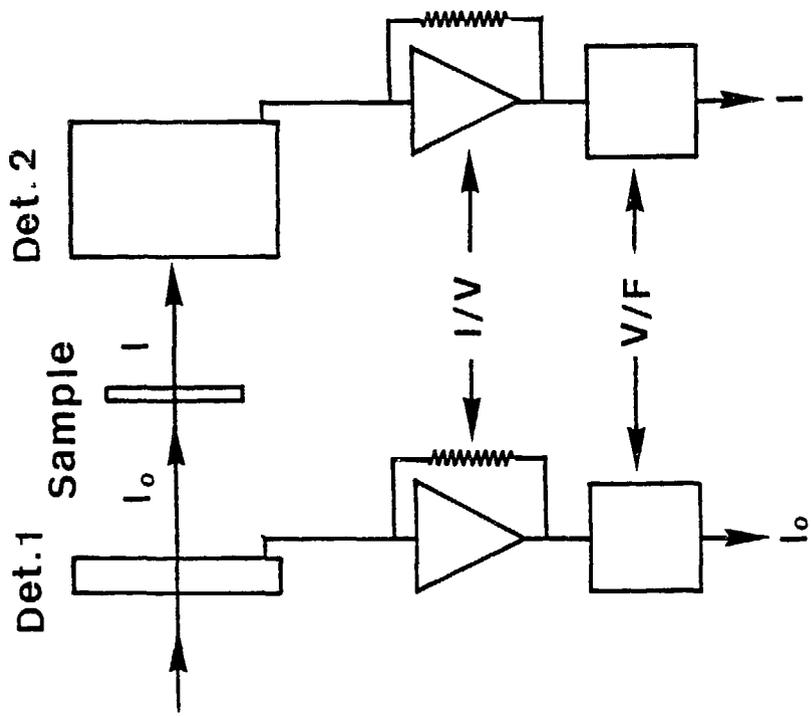


Fig. 10

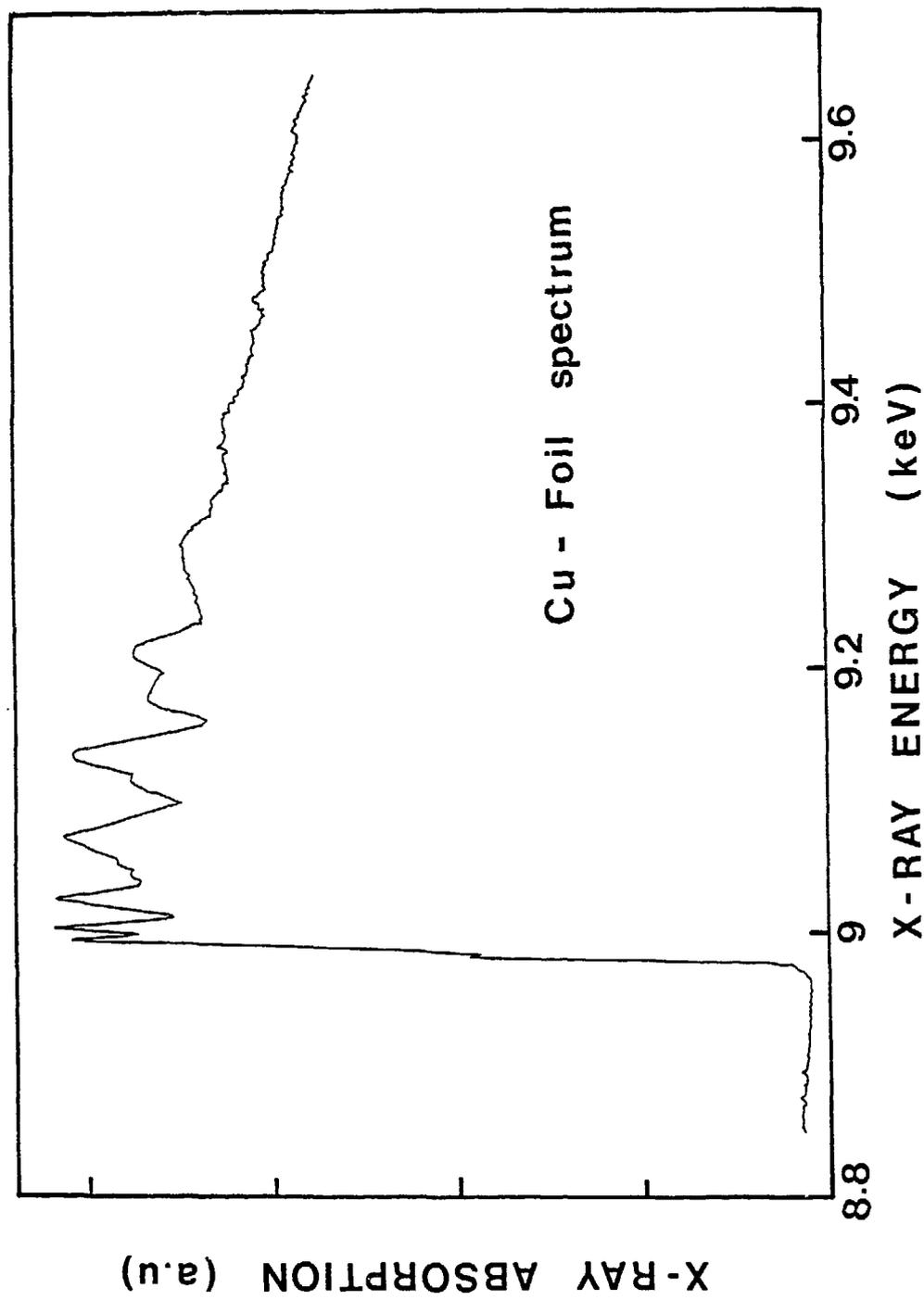


Fig. 11