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THE FABRICATION OF ENCAPSULATED GE (Li) DETECTORS FOR γ - SPECTROSCOPY

by

H. MEYER, H.L. ESCHBACH, W. NAGEL and E.W. KRUIDHOF

1968

Joint Nuclear Research Center
Geel Establishment - Belgium

Central Bureau for Nuclear Measurements - CBNM

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European Atomic Energy Community - EURATOM
Joint Nuclear Research Center - Geel Establishment (Belgium)
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The detector crystals are placed in aluminum capsules hermetically sealed by cold pressure welding. The important details of the welding process to ensure reliable welds are discussed. Proper crystal mounting, clean

helium atmosphere in the capsules and a carefully constructed feedthrough avoid biasing limitations and allow long term stability of the detector characteristics.

Background degradation due to the encapsulation of detectors irradiated with γ-rays was not detected.

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SUMMARY

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KEYWORDS

FABRICATION
GERMANIUM
LITHIUM
SEMICONDUCTOR COUNTERS
GAMMA SPECTROMETERS
CAPSULES
ALUMINUM
WELDING
HELIUM
STABILITY

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The Fabrication of Encapsulated Ge [Li] Detectors for γ-Spectroscopy

1. Introduction (*)

The application of germanium crystals instead of silicon for high resolution γ-spectroscopy has important advantages concerning counting efficiency and resolution, because of the higher density of Ge and its smaller bandgap leading to a better energy-to-charge conversion factor. But the small bandgap involves a low crystal temperature,(< 150° K) if the resolution is not to be determined by noise from thermally generated carriers in the crystal.

Other reasons, which have led to the use of liquid nitrogen temperature (77° K) for application and storage of detectors, are:

- maximum carrier mobility for minimum charge collection times resulting in small degradation of energetic resolution from recombination losses and high speed performance;
- small degradation of the degree of impurity compensation of the intrinsic detector volume by precipitation or outdiffusion of lithium; it is well-known that also local effects can influence overall performance remarkably.

Evacuated containers are used both to isolate the cooled detectors from the ambient and to have a surrounding medium of minimum mass and density; this assures small absorption of radiation, especially at low energies and avoids remarkable scattering effects degrading background of spectra. A problem, especially pronounced for cooled semiconductor detectors, is the treatment normally called edge protection for junction detectors (pn-devices). For silicon detectors used at ambient or temperatures down to solid carbon dioxide (dry ice) the necessary conditions of junction edges or surfaces of intrinsic regions can be maintained also at ambient atmospheric conditions by using epoxy resins or planar techniques (Si-oxide) for surface protection. For germanium detectors similar techniques are not applicable up to now. For instance,

(*) Manuscript received on July 2, 1968.

the greater temperature difference between detector preparation and use or storage would call for characteristics of a protecting material not easily obtainable; also germanium oxide has no suitable behaviour.
The method most commonly used is to keep the detectors during application and storage under clean vacuum conditions in the cryostats into which they were first introduced in connection with the first experiments planned. This procedure is recommended to avoid surface contamination which would ask for a retreatment of a detector by personnel trained for detector fabrication.
The main disadvantages of such a procedure are:

- For each detector an expensive cryostat with more or less expensive vacuum equipment is needed.
- The detection equipment cannot be adapted in an optimal manner to varying experimental conditions.
- Large quantities of liquid nitrogen are often needed also during storage of detectors.
- Transportation of detectors is complicated.
- For a repair of cooled preamplifier stages, directly associated to detectors for optimum performance, the cryostats must be opened (sensitivity of the FET-inputs of preamplifiers to voltage transients caused by accidents).

To circumvent the mentioned problems it was proposed to surround the detector by a tight container in which a gaseous atmosphere has to be maintained which guarantees unchanging correct conditions for the sensitive crystal surface. Cans of aluminium or steel, well evacuated or filled with pure helium before sealing, were used.[2,3,4]

2. General considerations

Desirable features of encapsulated detectors are the following:

- Low density of the encapsulating material for small absorption and scattering effects from radiation to be detected.
- Small capsule volume, not remarkably larger than the crystal volume to give maximum flexibility for the application to various experiments.
- Suitable and constant gas pressure in the capsule to avoid gas discharges also at high electric fields. Bias voltages up to 2000 Volt are desirable.
- Small thermal resistance between cooled capsule surface and crystal.
- Quick encapsulation procedure and no thermal heating during encapsulation to avoid a degradation of performance of prepared crystals.
- Stable capsule geometry at external pressure variations up to 1100 torr and insensibility of sealing quality at temperature variations up to 250°.
- Small increase of detector capacity due to encapsulation.

3. The fabrication of capsules

For the encapsulation of germanium detectors it is of primary importance to ensure a reliable vacuum tight seal between flanges to be joined. In principle many of the known welding and soldering techniques can be applied, provided that appropriate precautions are taken. Above all the crystals have to be protected from being heated during the sealing procedure. For instance, germanium detectors were sealed already into steel capsules by resistance welding [3]. In general, however, the most appealing method to procure vacuum tight seals seems to be the so-called cold pressure welding which is particularly easy to carry out if entire aluminum capsules are used. This method is clean, quick and reliable and no heat has to be dissipated.

3.1. Cold pressure welding

3.1.1. Welding principle

With the process of cold pressure welding the two metal parts to be joined are forced firmly together by merely applying high local pressure at ambient temperature. This brings the metal surfaces into close contact even on an atomic scale and during the subsequent plastic deformation of the metals in the weld a strong bond is produced.

The method of cold pressure welding has been known for quite a long time. For a historical review, see for example the compilation of F.C. Kelley [5]. Systematic studies of the welding process were carried out by Sowter [6], Kelley [5], Miller and Oyler [7] and Hofmann and Ruge [8].

A great variety of metals may be welded by the application of pressure, a.o., aluminum to aluminum, copper to copper, nickel to nickel, stainless steel to stainless steel. But cold pressure welds between different metals such as aluminum to copper, aluminum to mild steel, nickel to stainless steel, etc., are also feasible. The essential features of the method are illustrated by fig. 1 which shows schematically the welding of two metal strips of equal thickness d. The hardened ends of the punches are conical with an angle of 60°. The width 's' of the polished ends has to be varied with the materials to be welded. In the case that dissimilar metals have to be joined, the width 's' on the side of the harder metal has to be smaller than that of the counter punch. Fig. 1b indicates the situation after the full welding load has been applied. In the welding zone the total thickness of the two strips 2d has been reduced to a thickness 2r. The amount of deformation to produce a satisfactory weld is quite critical. The percentage of material left in a reliable and strong weld is called the 'figure of merit' F_M:

$$F_M = \frac{2r}{2d} \cdot 100\ \%$$

Some figures of merit are listed in table 1.

Table 1

Figures of merit of various cold welded metals

Metals welded	Punch width s (mm)	Fig. of merit
Al - Al	1.6	30
Cu - Cu	1.6	11
Ni - Ni	1.6	6
Stainless steel - Stainless steel	1.6	6
Al - Cu	Al: 1.6 Cu: 0.8	16
Al - Fe	Al: 1.6 Fe: 0.8	12
Cu - Fe	1.6	16
Cu - Stainless steel	Cu: 1.6 SS: 0.8	11
Fe - Ni	0.8	12
Stainless steel - Fe or Ni	0.8	6

During the welding procedure it can be observed regularly that from a certain amount of deformation on the thickness can be still further reduced without augmenting the load. This is due to a lateral flow of the metal. For some metals the load even tends to decrease. This has been measured by Hofmann and Ruge [8] for very pure annealed aluminium. In fig. 2, which is taken from their paper, the actual load is plotted against the reduction in the weld. Already at about 40 % reduction the flowing starts and a slight decrease of the load can be observed for higher reductions. The increase of the load at reductions higher than 60 % is due to the work hardening in the weld.

The speed with which the weld is performed as well as the duration of time that the maximum load is applied seem to be of little influence on the reliability of the welds.

It should be mentioned that vacuum tight and strong welds can also be produced when the deformation exceeds the optimum reduction corresponding to the figures of merit given in table 1. However, in this case the periphery of the necked-down section (see circle A in fig. 1b) may be unduly thinned and failures may occur, especially when cycling the capsules between ambient and liquid nitrogen temperature.

3.1.2. Choice of materials

A material of low density is to be preferred for detector capsules. Therefore, our own experiments with cold pressure welding were extended only to the bonding of different kinds of aluminum to aluminum and of aluminum to copper. At first the bonding of copper to aluminum was thought to be of special interest as this would allow to fabricate the lid of the cans of copper, and electrical feedthroughs could be placed easily by tin soldering. Although vacuum tight Cu-Al joints have been made repeatedly by cold pressure welding, they are nevertheless more difficult to produce and they seem to have less mechanical strength than Al-Al welds. In order to have both advantages, namely Al-Al welding flanges and soldering on copper for the feedthrough, the lids were fabricated from Cupal *), i.e., copper clad aluminum. Sheet material of 2 mm total thickness (1.6 mm Al 0.4 mm Cu) was used throughout the experiments. The purity of the aluminum used for the fabrication of Cupal is about 99.5 % as specified by the manufacturer.

3.1.3. Cleaning

A very critical part of the cold welding process is the cleaning of the metals before welding. In the case of aluminum

*) Cupal, Trademark of Hetzel & Co. Nürnberg

it is of special importance to remove the relatively thick oxide layer on the surfaces to be welded. Most authors agree that chemical cleaning is not satisfactory but mechanical cleaning is recommended.

Although we produced satisfactory welds by scrubbing the flanges thoroughly with steelwool and rinsing them in acetone, another method of cleaning has been adopted afterwards. The flanges to be welded are carefully machined on a lathe taking off a very thin layer. No coolant or lubricant is used during this process and care is taken that the flanges are not contaminated again. In general the parts are used immediately after turning but in a few instances they have rested in air for two days before welding. Good welds could still be produced. This is in agreement with a more systematic test by Hofmann and Ruge [8] who have shown that only after several days a detrimental influence of the slowly growing oxide layer can be measured.

3.1.4. Welding tests

A series of welding tests were carried out using the final circular dies. The annular welding surface has a mean diameter of 52 mm and a width of 2 mm. Two kinds of aluminum (95 % and 99.5 % purity) were used and flange thicknesses of 1.0 and 1.5 mm were tested. Best results were achieved with 1.5 mm thick flanges of very pure annealed aluminum with welding loads between 8 and 8.5 ton. For the material used this gave a deformation corresponding to a figure of merit of about 30 to 40. A photomicrograph of a typical weld is shown in fig. 3. It should be noted that it was found necessary to hold the dies in perfect alignment to obtain satisfactory welds. Already a small misalignment may reduce the thickness of the critical region at the periphery of the weld (see circle A in fig. 1b) considerably.

3.2. Apparatus for encapsulation

The construction and main dimensions of the capsules used for planar diodes are given in fig. 4. The indicated form of the lid was chosen in order to avoid excessive buckling due to the lateral flow of the aluminum during the welding process. Before forming the lids part of the copper layer was taken off leaving a central circular area of about 30 mm in diameter. The capsules were fabricated out of Al-bars by turning. Both lids and capsules were carefully degreased and then degassed in a vacuum oven.

The dies employed for the welding are sketched in fig. 5. They are made of hardened 13 % Cr-steel. Three small lips are fixed to the periphery of the upper die to hold the lid during the evacuation cycle and helium filling period. With these dies and the aluminum used no pick up has been observed even after many welding experiments.

The unit being used for the encapsulation is shown in fig. 6. It consists basically of a crosslike recipient (1) made of stainless steel. A 270 l/s ion getter pump (Varian) (2) is attached to one of the main ports. The roughing line (3) and the gas inlet system (4) are fixed to the opposite port. The stainless steel bellows (5) on the top part of the cross transfers the welding load into the vacuum system and the four external springs (6) prevent that the bellows are compressed by the atmospheric pressure and by the weight of the heavy top plate. The complete assembly is mounted on a rolling table and can be placed between the pistons (7) of a 15-ton hydraulic press. All vacuum seals are made by OFHC copper gaskets.

3.3. Capsule tests

The complete encapsulation cycle beginning with the loading of the dies takes at maximum 65 minutes. Since the capsules are filled with helium they can be leak tested any time by placing them in a high vacuum bell jar which is connected to a helium leak detector. The detector available has a sensitivity of 5.10^{-11} torr l/s.

Several tests were carried out in order to check whether the welds remain vacuum tight after they had been cooled to liquid nitrogen temperature. For that purpose a small copper tube was soldered to the lid of the can in place of the electrical feedthrough. This tube could be connected to the leak detector directly. With the welding procedure finally adopted no leaks could be detected even after the capsules had been repeatedly immersed in liquid nitrogen and quickly warmed up again.
To avoid a voltage breakdown in a capsule when the detector is biased up to high voltages (2000 V) the leakage current over the feedthrough equipped with all contact parts (see fig. 7) was measured at various pressures of helium. According to Paschen's law the static breakdown voltage, i.e. the voltage at which a selfmaintaining current can flow in a gas, depends on the product of the gas pressure p and the electrode separation d. For a fixed electrode separation the breakdown voltage varies with the gas pressure. At low pressures the voltage decreases and at high pressures it increases with increasing pressure going through a minimum in an intermediate region. In the present case this minimum was found round about 5 torr. Sufficiently low leakage currents were observed at a He pressure lower than 10^{-2} torr and at pressures of 760 torr or higher. It is believed that occasional failures were due to sharp edges or points. For this reason all edges on the contact parts are rounded off carefully. The helium pressure in the final capsules is usually fixed to 1100 torr.

Before encapsulation all lids are tested for vacuum tightness and low leakage currents. With the capsules at liquid nitrogen temperature leakage currents < 0.1 nA are normally observed when applying bias voltages up to 2000 V.

As mentioned before, low leakage currents are also obtainable in well evacuated capsules. But due to the unavoidable outgassing of the crystals and the inner surfaces at room temperature and since the volume to surface ratios are usually very small, the pressure in the capsule might quickly rise into the critical region where the necessary bias voltage cannot be applied.

4. Encapsulation Procedure

4.1. Crystal preparation

Crystals for encapsulated detectors can be handled practically in the same way as those for non-encapsulated devices. We evaporate the lithium for diffusion on etched crystal surfaces. The diffusion is performed at 410°C to 420°C and the crystals are drifted in boiling pentane [9]. Special care is taken to maintain a high enough density of mobile lithium atoms in the diffused n-region of the crystal also after drift — if necessary by rediffusion or reheating. For that purpose, the resistance of the n-side is measured very carefully to avoid high resistivity regions from local imperfections.
Clean up cycles have to be fully completed before encapsulation; a probable outdiffusion of lithium could necessitate a retreatment of a crystal which is not possible after encapsulation.
For a final crystal test before encapsulation charge compensation on the intrinsic crystal surface is performed in a suitable cryostat; the main parts of the procedure applied, H_2O_2 dip and storage at controlled air humidity, were mentioned earlier in literature [10].
Acceptable test results include low leakage currents up to high bias voltages, small slope of detector capacity v.s. bias voltage and also small degradation of energetic resolution up to high γ-energies.

4.2. Crystal placement and contacts

The arrangement of a planar detector in a capsule is shown in Fig. 7.

A general problem is given by the variety of geometrical configurations of crystals which are possible. Circular and trapezoidal cross sections are often used; but the final dimensions of different detector crystals are never the same because unusable parts containing irregularities or imperfections are cut or lapped away during the fabrication process.

The thin indium disc at the front-side of a planar detector, normally the n-side of a not fully compensated crystal, will assure a good surface contact. The spring pressure on the back of the crystal will help to assure a fixed shock insensitive crystal position in the capsule. The spring length is adapted to the crystal thickness.

To simplify the equipment for encapsulation, only capsules having the same diameter are used. They can serve for crystals with a maximum radial extension of about 40 mm.

For coaxial detectors, a spring with a modified contact part will be used as center contact. A special spring arrangement is foreseen between the lithium enriched outer n-surface of a crystal and the cylindrical capsule wall.

As shown in fig. 7, special care has been taken to avoid sharp edges on the feedthrough and around it to allow high bias voltages without voltage break-down.

4.3. Encapsulation cycle

To assure a minimum time interval, during which a crystal to be encapsulated is at ambient temperature, a fixed time schedule is followed for encapsulation. After outgassing and cleaning of the capsule parts, the crystal is taken out of the test cryostat at the latest moment for a short cleaning and surface handling.

At controlled humidity the crystal is placed carefully in the capsule and then transported in a closed container to the encapsulation apparatus. In the meantime the equipment has been prepared in the following way:

The system is first pumped by a double stage rotary pump which is baffled by a simple nitrogen cooled trap. This roughing period is kept as short as possible. When a pressure of $< 10^{-2}$ torr is obtained, the rotary pump is shut off and the precooled sorption pump is connected to the system. At a pressure of about 5.10^{-4} torr the ion getter pump is started, the roughing line is closed, and the system is evacuated to the low 10^{-7} torr region. This process can be aided, if necessary, by a mild bake at about 150°C using heating tapes.

To load the system with the prepared capsules it is flushed with pure and carefully dried nitrogen. The loading is carried out via the top port of the recipient. Opening the top port, loading the dies and closing the top port again takes about 5 minutes. Then the roughing valve can be opened. After a roughing time of generally 16 minutes the ion getter pump can be started. After another 40 minutes a pressure of about 5.10^{-7} torr will be reached. This can be measured by an inserted Bayard-Alpert gauge. At this pressure the ion getter pump is shut off and pure helium (99.995 % He, as specified by the supplier) is slowly bled in via a needle valve. As an additional precaution, a trap filled with silicagel and a liquid nitrogen cooled trap filled with molecular sieve are incorporated in the helium line to prevent any water vapour to enter the system. At a helium pressure of about 1.5 atmospheres the welding is carried out. The encapsulated crystal can be taken out again through the top port.

Immediately after a short visual inspection the capsule is cooled down in about 15 minutes to a temperature, lower than 150°K by immersing a copper bar on which the capsule is fixed in liquid nitrogen.

5. Handling and test of encapsulated detectors

If the external surface of the feedthrough is freed sufficiently from frozen condensed water vapour a preliminary test of the detector performance in liquid nitrogen is possible. Normally, only the leakage current vs. detector bias is measured and compared with results before encapsulation. Capacitance measurements and comparative tests with γ-radiation are performed in a test cryostat. A small but defined increase of the detector capacity, not larger than 1-2 pF for suitable feedthroughs, cannot be avoided. The test cryostat should be equipped with a sensitive helium leak detector. A quick transfer of a capsule from the nitrogen bath to the cryostat is recommendable, especially, if this is a current procedure of the experimenter. For that purpose the cryostat is equipped with spring contacts assuring short installation time during which the cryostat is held at room temperature and filled with dry nitrogen gas. In such a way, leakage currents on the surface of the feedthrough and evacuation problems because of frozen condensed water vapour are avoided. A fast cooling of the detector holder in the cryostat — via the cold finger during evacuation — will contribute to reduce a certain small probability that rest gases condensed on the capsule walls might be freed and diffuse to the sensitive detector surface, degrading performance.

In fig.8 a γ spectrum of ^{137}Cs measured with an encapsulated planar detector is demonstrated. No increase of background due to the encapsulation can be detected.

6. Possible improvements and extensions

It is planned to encapsulate also coaxial detectors with a sensitive volume up to about 50 cm^3 in the near future. In order to reduce the influence of the mass of the capsule on detector performance, (for instance beryllium capsules welded in a suitable way to the applied aluminum covers and(or) thin effective windows) especially for planar detectors by profiling the capsule front end, should be tried. The stiffness of the capsules has to be maintained for pressure differences of up to 1.5 atmospheres under worst case conditions (capsule in vacuum at room temperature).

7. Acknowledgements

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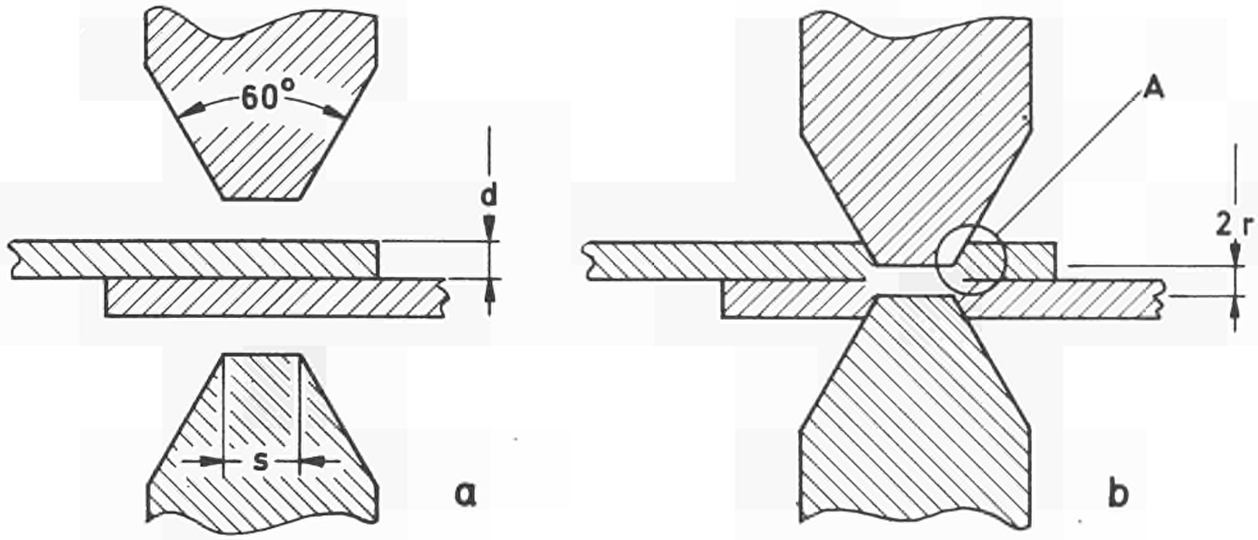

Fig.1 Schematic illustration of the cold pressure welding process.
a) Welding of two metal strips of equal thickness d.
b) Position of the punches at maximum load.
Circle A indicates a critical region adjacent to the welded part.

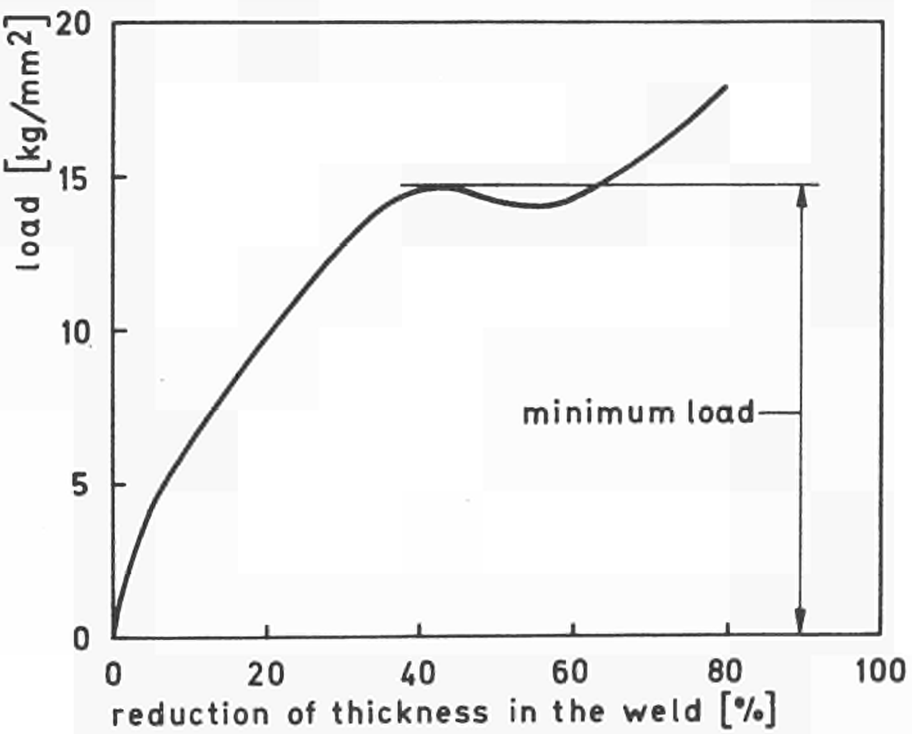

Fig.2 Variation of load with the final reduction of thickness in the weld for very pure annealed aluminium.
(After Hofman and Ruge [8])

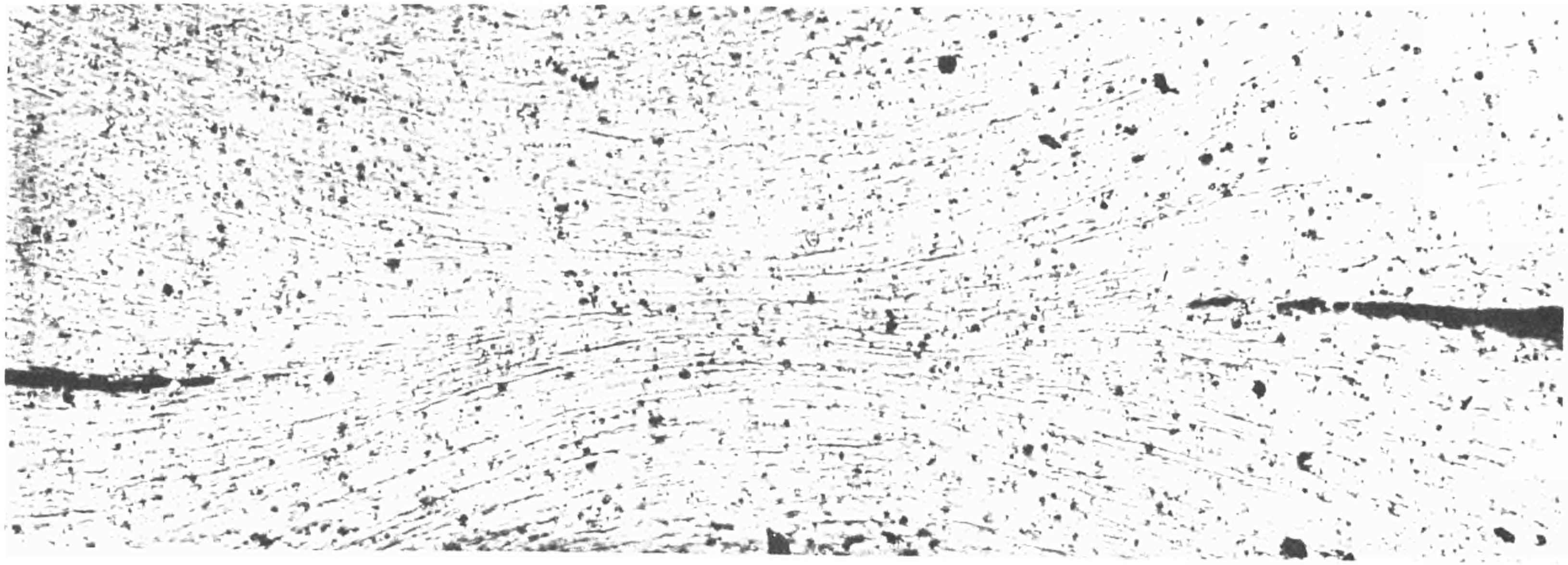

Fig. 3 Photomicrograph of a strongly etched aluminium to aluminium cold pressure weld at 60 % deformation. (magnification 120 x)

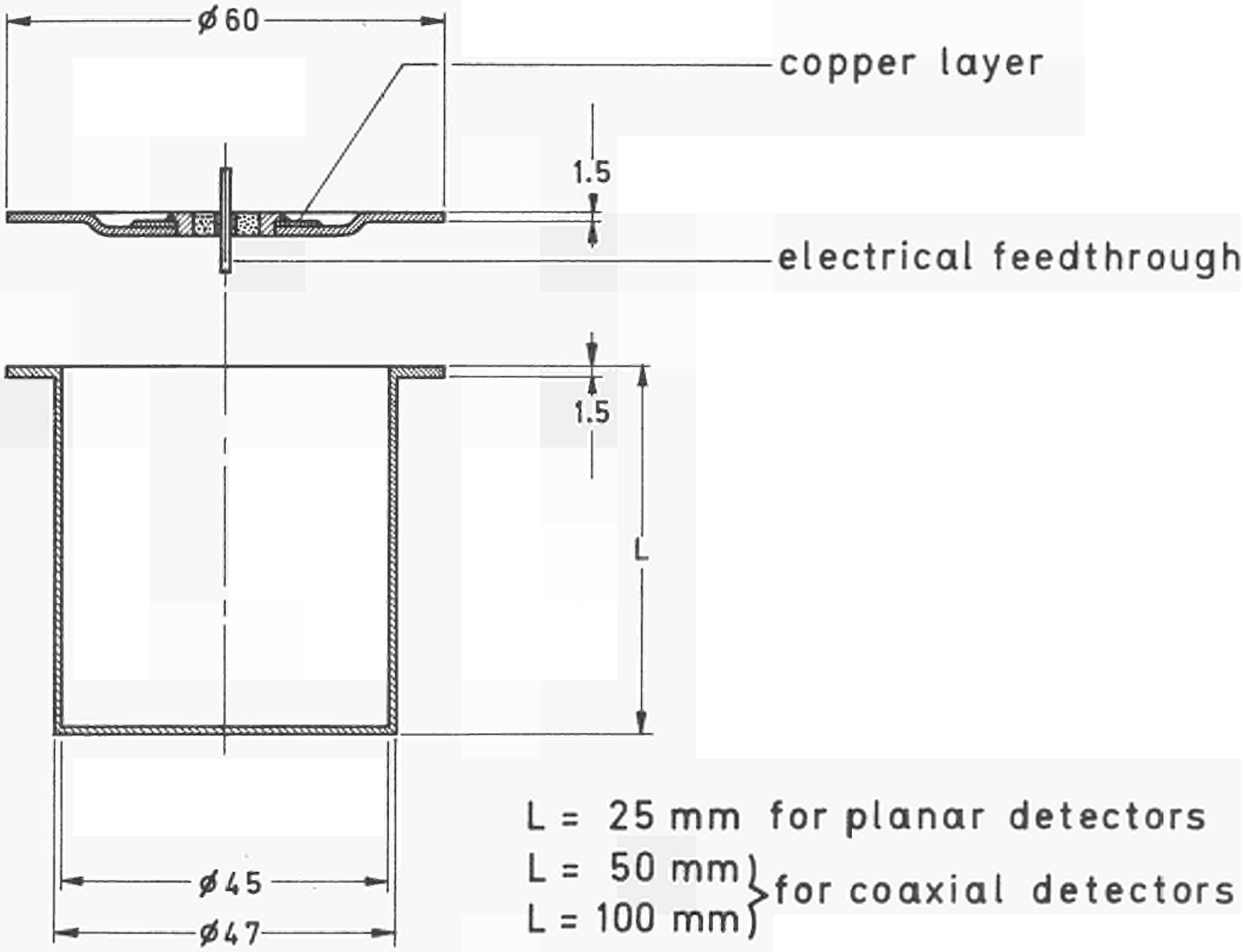

Fig. 4 Can and lid for the encapsulation of germanium detectors.

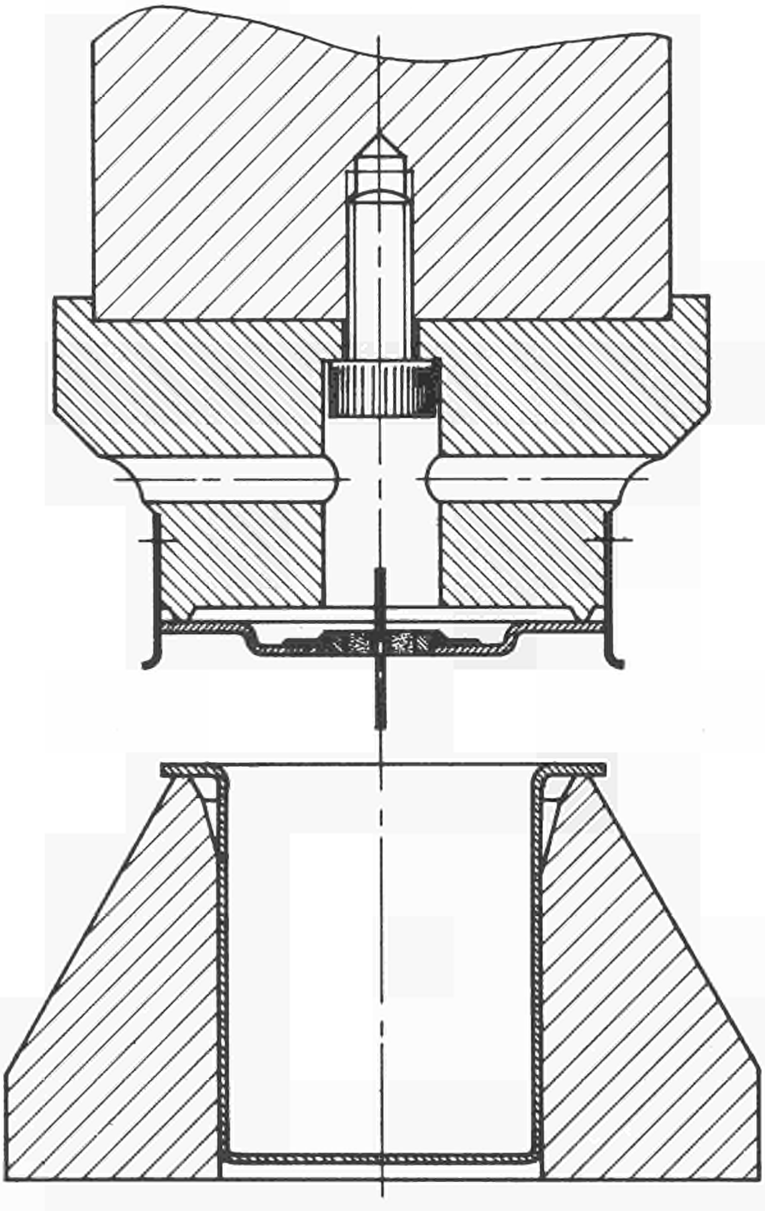

Fig.5 Dies for the cold pressure welding of detector cans.

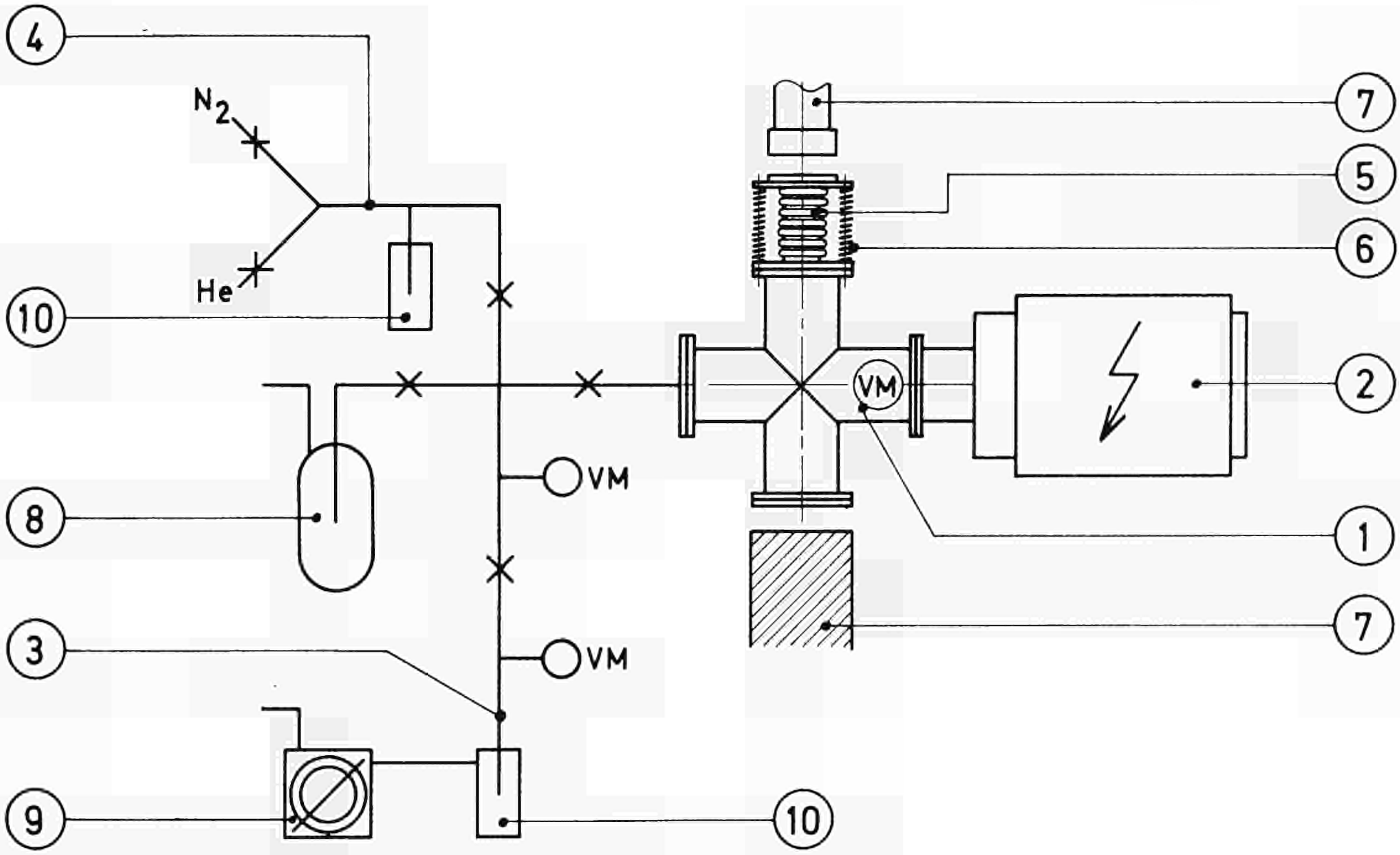

Fig.6 Encapsulation unit.

① recipient
② ion getter pump
③ roughing line
④ gas inlet system
⑤ stainless steel bellows
⑥ external springs
⑦ hydraulic press
⑧ sorption pump
⑨ rotary pump
⑩ trap
(VM) vacuum gauges

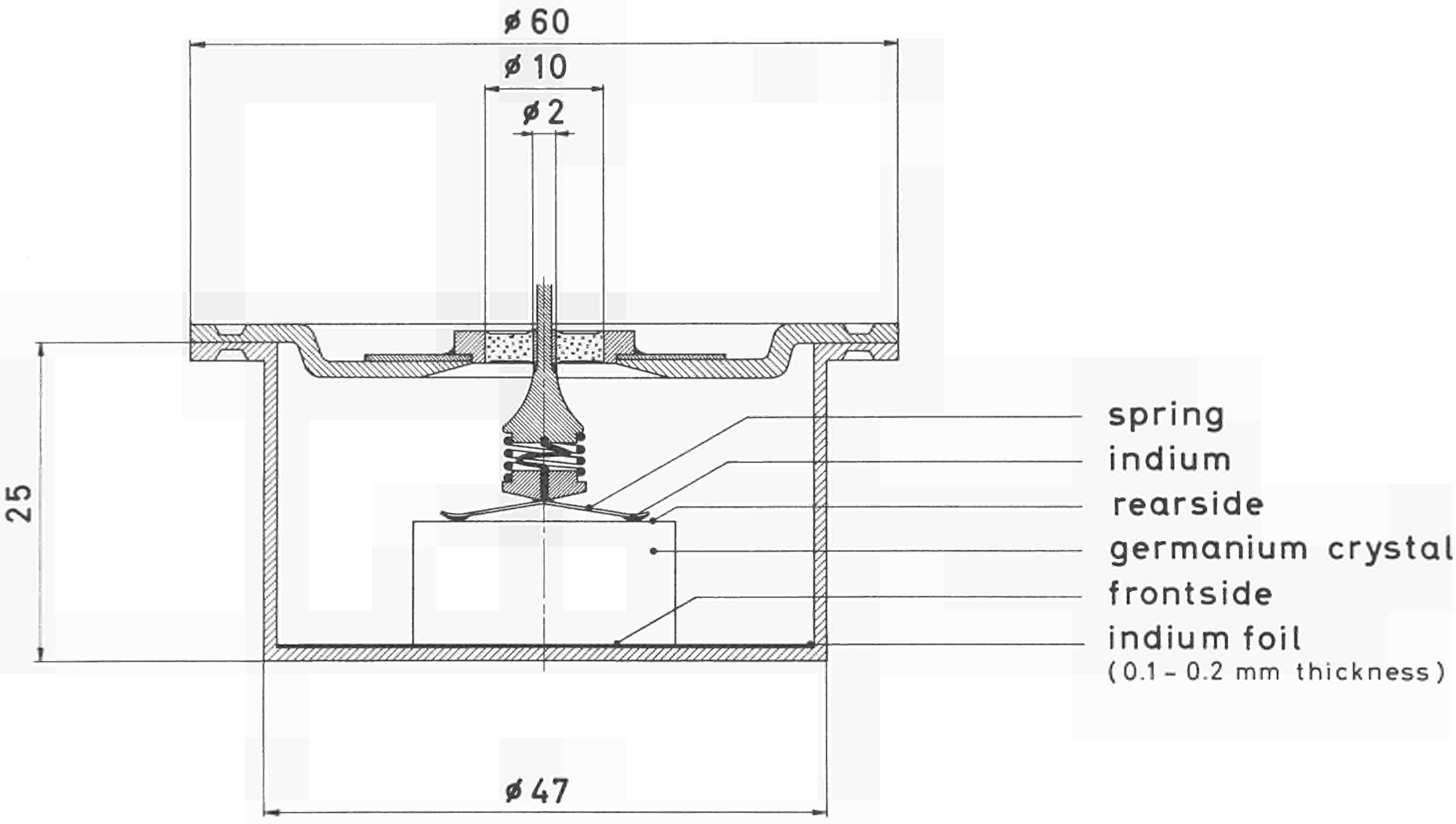

Fig.7 Encapsulated planar detector.

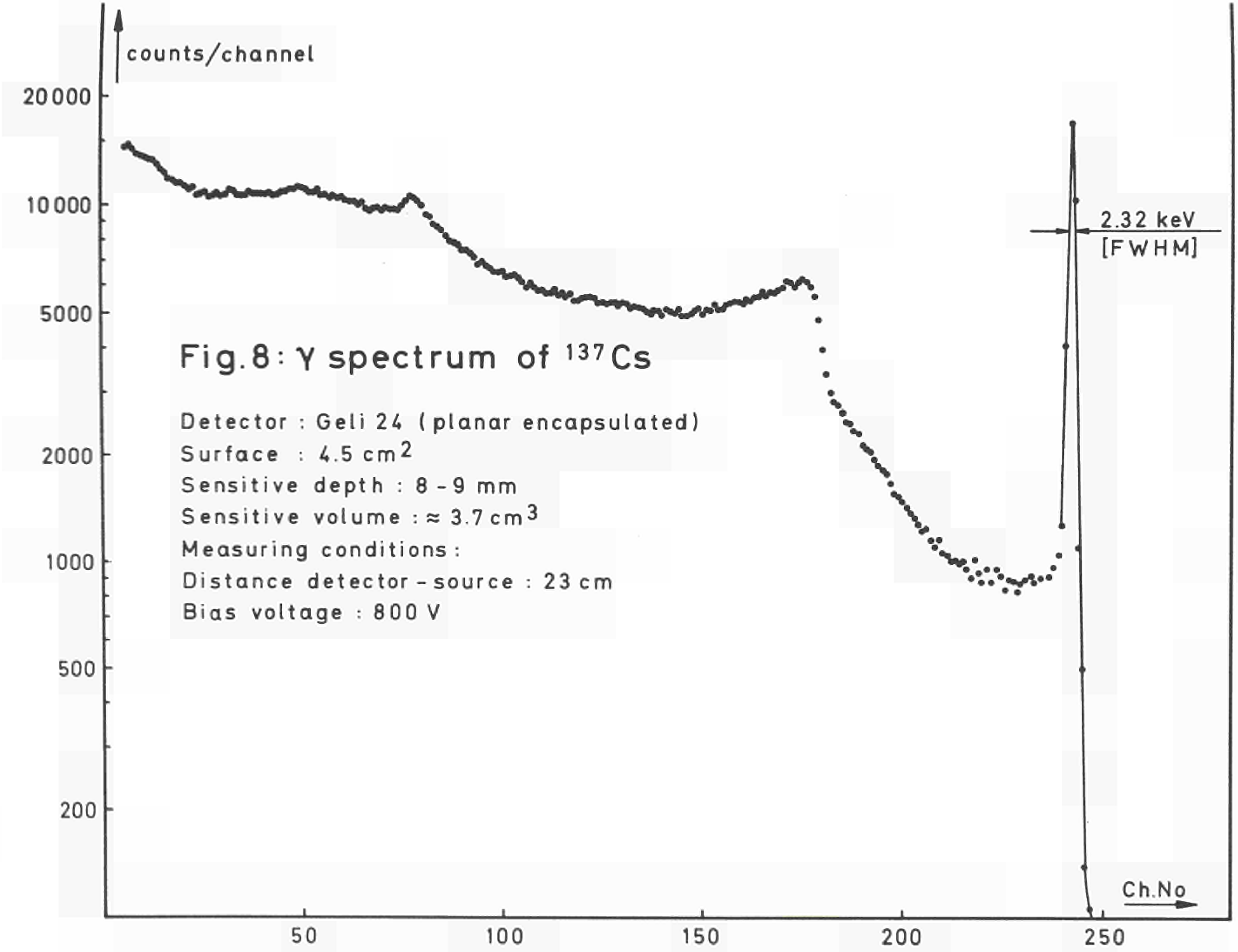

Fig. 8: γ spectrum of ^{137}Cs

Detector : Geli 24 (planar encapsulated)
Surface : 4.5 cm^2
Sensitive depth : 8 - 9 mm
Sensitive volume : ≈ 3.7 cm^3
Measuring conditions :
Distance detector - source : 23 cm
Bias voltage : 800 V

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Alfred Nobel

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