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ATOMIC ENERGY COMMISSION

GROWTH AND FABRICATION OF LARGE SIZE SODIUM IODIDE CRYSTAL SCINTILLATOR

by

S. C. Sabharwal, S. C. Karandikar, T. Mirza, B. Ghosh and R. Y. Deshpande
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INIS Subject Category : B12, E41

Descriptors :

SODIUM IODIDES

MONOCRYSTALS

CRYSTAL GROWTH

ACTIVATION

THALLIUM

SCINTILLATION COUNTERS

RESOLUTION

BRIDGMAN METHOD

CRYSTAL DOPING

DOPED MATERIALS

NUCLEATION

FURNACES

ABSTRACT

The growth of 80 - 135 mm dia. Sodium iodide crystals activated with thallium is described in the present report. The growth is effected in a glazed porcelain crucible in a protective ambient of dry nitrogen. The technical details of the equipment developed have been fully described. The results of our measurements on the rate of growth of crystal and the optimization of different growth parameters are reported. The dependence of various factors upon the performance characteristics of the scintillator detectors made using these crystals is also discussed. The energy resolution obtained for a typical detector of dimensions 76 mm dia. x 76 mm ht. is 10 percent.

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INTRODUCTION

Sodium Iodide crystals activated with thallium occupy an important place in gamma ray spectroscopy. The effectiveness of a scintillator depends upon its ability to absorb the incoming ionizing radiation and convert the absorbed energy into light pulses. In the case of NaI type scintillator the energy from the incident gamma or X-ray is first absorbed by the host lattice and subsequently the activator ions are excited to the upper states[2]. The deexcitation of the latter gives the light flash. To get better response from a scintillator detector it is necessary to have isotropic optical transparency of the scintillator with optimum concentration of the activator distributed as uniformly as possible. This is mostly achieved with a single crystal scintillator. In this communication we report the growthof large size NaI(Tl) crystal and fabrication of the detector.

NaI(Tl) crystals upto 135 mm dia have been grown in a protective ambient of dry nitrogen by Bridgeman-Stockbarger technique[2]. While crystals upto 50 mm dia x 50 mm ht. are usually grown in vacuum sealed quartz crucibles[3], the large size crystals have been grown in glazed porcelain crucibles. Taking into account, the specific heat, the latent heat, the thermal conductivity etc.. of the material the processing parameters like the growth rate, the nature of solid/melt interface and the temperature distribution and stability have been optimized for growing high quality large size crystals. The effects of several processing parameters on the quality of the crystal are mentioned. The typical

resolution of a fabricated detector is quoted and its dependence on factors like polishing and canning is discussed.

2. THEORY OF CRYSTAL GROWTH

In Bridgman Stockbarger technique, crystal growth is effected in a suitable container by lowering the melt through the melting point isotherm along a gradient in the furnace. The whole material in the crucible is initially in the molten state and gets supercooled when lowered through the melting point isotherm. In absence of any seed crystal, the super-cooled melt does not solidify until the degree of supercooling[1-6] is sufficient such that the change in the free energy of system due to the appearance of the solid phase in the melt, is equal or greater than a critical value called the 'activation energy'. The process which gives rise to the first appearance of the solid phase is known as 'Nucleation' and the minimum size of the solid phase which is capable of existing independently in contact with the melt is called 'critical nucleus'. Once the melt nucleates, the remaining mass is capable of undergoing a phase change and this process is known as 'crystal growth', where the overall change in the free energy of the system is quite small compared to the nucleation process. In order to get a single crystal it is imperative that only one nucleus propagates throughout the melt. For this reason, the two processes, i.e., nucleation and crystal growth must be separated. After the nucleation has occured the growth of the critical nucleus is maintained so slow that the possibility of any further nucleation is very small for all practical purposes. However, nucleation being an uncontrolled

process usually gives rise to a number of nuclei in the melt. For this reason the tip of the crucible is made conical so that, initially only a very small volume of the melt is supercooled. Thus, only a small number of nuclei appear and as the solidification proceeds a faster growing nucleus usually dominates the growing interface.

Once the nucleation takes place, more and more material undergoing phase transformation helps in the growth of the nucleus into a tiny crystal at the tip of the crucible in equilibrium with the melt. For this crystal to grow into a large single crystal, a precise control of those factors influencing the heat transport at the solid/melt interface is the most important. The nature of the interface is preferred to be convex[7,8] with respect to the solid as it helps in eliminating nucleation on the crucible wall. In practice this is achieved by properly positioning the freezing plane in the furnace and forcing the heat of solidification to flow out along the axis of the already grown crystal. The freezing plane is maintained in the upper zone where the convex radial temperature gradient exists. For the proper heat transport it is necessary that the thermal gradient should be sharp at the interface. In case of the radial heat transport dominating over the axial heat transport the crystal grows with middle portion foggy as shown in Fig.1. Besides this, the sharp gradient at the interface helps in separating the nucleation from the growth process.

Critical free energy for nucleation ΔF_{crit} is given

by expression[5].

$$\Delta F_{crit} \propto \frac{T_M^2}{L^2 (\Delta T)^2} \qquad \ldots\ldots (1)$$

Therefore for large supercooling, $\Delta T = T_m - T$ (where T_m is the melting temperature and T to which melt is cooled) the change in free energy for nucleation required is small and therefore the probability of nucleation increases. With a relatively small gradient a large amount of the melt will supercool before nucleation initiates. Under the circumstances once the nucleation is there, growth through the remaining melt is quite fast and the final product is always of poor quality. But under the conditions of the sharp gradient the nucleation is quickerand is limited to a very small amount of the melt.

Large growth rates disturb the interface stability[9] which is also affected by growth rate variation. Moreover for a two component system like NaI(Tl), the thallium distribution in the crystal would be uneven for varying growth rates. The two necessary conditions of (1) sharp temperature gradient required for nucleation and conduction along the crystal axis and (2) small growth rate for crystal growth and interface stability are quite compatiable as can be seen from the following expression for the continuity of heat fluxes.

$$K_s G_s - K_m G_m = \rho_s . \Delta H . R \qquad \ldots\ldots (2)$$

where R - rate of growth, ρ_s the density of the solid, ΔH the heat of fusion, K_s, K_m are the thermal conductivities of the solid and melt and G_s and G_m are the thermal gradient in the solid and melt phase. Restricting the growth rate to small values the equation admits large

temperature gradients for judicial choice of Ga and Gm. The variation in the growth rate for the fixed temperature profile and the lowering rate are mainly due to the thermal oscillation(6) in the bulk of the liquid phase. This necessitates the need for a finer control of the melt temperature.

3. CRYSTAL GROWING EQUIPMENT:

The experimental setup and the necessary instrumentation for the growth of NaI(Tl) crystal by Bridgman-Stockbarger technique consist of the following: a suitable crystal growing furnace capable of generating the desired temperature gradient and temperature profile with built-in facility for supporting and lowering the crucible at a specific rate, a suitable annealing furnace, and the electrical power supply and control units.

Growing Furnace:

The crystal growing furnace is made up of two zones thermally isolated from each other with a gradient between them. The upper zone is always at a higher temperature than the lower zone. Therefore, in order to obtain steep gradient at the boundary, the radiations received by the cold zone from the hot are cut off using a baffle. The size of the baffle is selected in a fashion to permit free crucible movement. Baffle made of syndanic has been found very effective for this purpose. The length of the furnace depends upon the size of the crystal which is to be grown. However, for growing crystals by this technique it is required that the whole molten charge should be at a uniform temperature above the melting point isotherm. Therefore the length of the hot zone just above the gradient region

should be sufficiently larger than the length of the melt in the crucible. Considering the end heat losses and the necessary length of the hot zone, the length of the upper zone is decided to be roughly twice the length of the hot zone. The lower zone helps in producing the suitable gradient and in maintaining the temperature of the grown crystal above some minimum temperature. Taking care of the end heat losses, the total length of the lower zone should be roughly twice the crystal height.

Two refractory tubes of appropriate lengths and diameter are taken and resistance wire is wound on them. The details of the lengths, diameter, heating element and winding are given in Table.I. As shown in Fig.3 the two tubes are arranged one above the other inside the aluminum can. The two tubes are isolated from each other by a syndanic disc having internal diameter slightly smaller than the tube diameter. The step formed by the syndanic disc in the furnace is used as the baffle support. The annular space between the refractory tubes (muffles) and the aluminium can is filled with insulating asbestos magnesia to minimize the power losses. The heat losses are further minimised by providing a static air gap between the furnace and the outer aluminium cylinder as shown in the figure.

The crucible inside the furnace is rested on a metal seat of conical shape, which is attached to a rod projecting out of the furnace from the bottom side. The metallic seat helps in the conduction of heat from the crystal along the axis. The use of a ceramic seat in place of the metallic is not advisable as under these conditions the growing interface tends to become concave. By the help of rack and pinion arrangement the support rod could be lowered by a motor

coupled with appropriate gears. The lowering employed in this case is of the order of 10 mm / day.

Annealing Furnace

The annealing furnace consists of a single muffle, having a single winding to provide a uniform temperature zone of about 25 cms. The relevant details of the furnace shown in Fig.4 are given in Table .2.

Power Supply & Control Units

For setting up the furnace temperature at any desired value, it needs well stabilized power supply, temperature sensors and temperature controllers.

The power to each zone is provided from separate stabilized mains supply of 240 Volts. In order to obtain the stabilised power supply either saturated core or servo type voltage stabilizers are used. The voltage stabilizer output is connected to the furnace terminals through a 15 amp variac to get variable power input. The power requirement for the special case are indicated in Table.I. To control the furnace temperature at a desired value, the input power is controlled by means of ON-OFF type temperature controllers with time-proportional band. The changes in the temperature from a set value are sensed by Chromel-Alumel thermocouples, positioned in the upper and lower zones, and connected to the temperature controllers. Depending upon the deviation in temperature from the set values, the power is controlled through electrical contactors. Temperature stabilization better than $\pm$ 3°C is achieved at the operating temperatures.

Growth Procedure:

In effecting the crystal growth by Bridgman-Stockbarger

technique the thermocouples controlling the temperatures of the different zones in the furnace are usually kept outside the crucible near the furnace wall as shown in Fig.2. However, in most of the cases the crystals grown were foggy in the middle. This was thought to be due to the uncontrolled movement of the freezing plane during the process of the crystal growth consequent to the poor control of the melt temperature. In order to overcome this difficulty the technique was modified as shown schematically in Fig.3. The thermocouple which is to be used to control the melt temperature was inserted inside the melt. To avoid the direct contact of the thermocouple with the melt, it is contained in a thin walled quartz tube closed at one end. This helped in reducing the temperature fluctuations inside the melt and in stabilizing the freezing plane. To begin with the thermocouple tip resting on the crucible tip is maintained at a temperature 10 to 15°C above the melting point, the upper layers of the melt being at temperatures 20 to 30°C above the melting point instead of 50 to 80°C above the melting point maintained in the conventional procedure. The tip temperature being close to the melting point helps in initiating nucleation before much of the crucible is lowered. Thus the amount of the material undergoing supercooling before nucleation is minimum and the number of nuclei, as desired, is restricted.

Several experiments were carried out to standardize the processing parameters for the growth of 80 - 135 mm dia NaI(Tl) crystals. From time to time many modifications were introduced to simplify the growing arrangement while having better control on the process of crystal growth. Attempts were made to grow crystals initially in a furnace consisting of five separate resistance windings as used by Stepanov and Vasilera[10] for the growth of alkali halide crystals. Proper

protective ambients were provided by placing the crucible inside a gas tight chamber with appropriate cooling coils and arrangements for lowering the crucible. However, subsequently the multicoil furnace was discarded in favour of a two coil furnace. The advantages with a two coil design are the easily reproducible temperature profile and the minimization of the temperature fluctuations along the length of the melt in the upper zone.

It was further realized that the use of gas tight chamber is not advantageous as it provides poor-thermal contact between the melt and furnace. This gives rise to large temperature fluctuations in the melt. Moreover the large end losses, continuous gas and water circulation led us to modify the whole arrangement. The improved arrangement is shown in Fig.3. Here absence of the cylinder minimized the temperature fluctuations in the melt and allowed the crucible to sense the set temperature profile. We have maintained a flow of purified nitrogen gas inside the crucible for protective atmosphere. For this purpose the crucible was specially designed as shown in Fig.5. The crucible with the charge is sealed with an Inconel cap by means of recrystallized sodium chloride. Concentrated solution of NaCl in water is prepared and poured at the sealing joint of the crucible where the metallic cap rests. Sodium chloride left after evaporation of the water seals the cap and crucible together. Purified nitrogen is circulated through the long metal tube joined to the cap.

For the growth of a 80 mm dia crystal of sodium iodide, about 3.5 Kg of material is taken in a clean crucible. Thallous iodide roughly 0.3% by weight is added to the material. For proper distribution of the dopant in the melt, it is divided into parts and is mixed with the

sodium iodide while charging the crucible. The metallic cap is then sealed to the crucible.

The crucible with the thermocouple is positioned in the furnace with its tip at 406 mm from the top of the furnace, at 100°C. The purified nitrogen gas is then circulated and the charge is dehydrated at temperatures of 100°, 150°, 200° and 250°C. After dehydrating the material properly the temperature of the furnace is raised to 690°C in the upper zone as sensed by a thermocouple outside the crucible(T1). The temperature of the lower zone (T2) is fixed at 550°C. As the material gets melted, the quartz tube slowly goes inside the crucible and rests at the tip of the cone. The thermocouple outside the crucible is then disconnected from the temperature controller which is now connected to the thermocouple kept inside the melt. After sometime when the temperature of both the thermocouples have got stabilized the lowering motor is switched on. A lowering rate of 19 mm/day is employed in this case.

The temperature profile for the bare furnace with the upper thermocouple at 650°C and the lower one at 550°C is reproduced in Fig.6. In this particular case a temperature gradientof 8 deg/cm roughly exists at the freezing plane. It is found that for smaller gradients than this, the crystal grows with the central portion foggy as shown in Fig.1. The variation of temperature as recorded by two thermocouples fixed at 3 cms above and below the baffle during the growth of crystal are shown in Fig.7. As the crystal starts growing the temperature of the upper thermocouple falls below its initial value and then increases as the

the growth progresses. Since the thermocouple initially is resting at the tip of the crucible and therefore it senses the conduction losses which take place through the supporting rod. However, on lowering the crucible, the appearance of the solid phase acts as heat insulator between the controlling thermocouple and the metalic seat. This reduces the conduction losses and in order to maintain the controlling thermocouple to the set temperature, the wall temperature decreases. The subsequent increase in the wall temperature results from the fact that when the thermocouple passes out of the conical portion into the cylindrical portion the large amount of melt in the surrounding provides insulation to the thermocouple. And consequently to maintain the set temperature at the middle of the melt the furnace temperature increases as shown by the variation of T1. The attainment of the steady temperature indicates the completion of the crystal growth.

In the case of lower thermocouple the temperature falls due to the fact that lowering the crucible cuts off the direct radiation from the upper zone. On the contrary as shown in Fig.8 there is no variation in the temperature of the controlling thermocouple(Tm) throughout the growth period.

After completion of the growth, the temperature controller for the upper zone is again connected with the thermocouple T1. The seal between crucible and the cap is broken and the latter is pulled out of the furnace. The crucible is raised and is fixed between the clamp. Holding by the clamp, the crucible is taken out of the furnace and is inverted and put into the annealing furnace which is then covered by a lid. The arrangement in the annealing furnace is shown schematically in Fig.4.

The crucible in the inverted position rests on a stand. The temperature in the annealing furnace is set about 80°C higher than the melting point. As a result, the sides of the crystal in contact with the crucible melt and consequently the crystal slips down. At this stage there is no contact of the crystal with the crucible walls due to the tapering of the crucible. The slipped crystal is arrested on a seat which is attached to a rod coming out of the furnace. The rod rests on a spring so that when the crystal falls on the seat the mechanical jerk on the spring indicates the detachment. The furnace lid is then removed and the empty crucible is taken out. The lid is slowly replaced and the temperature in the annealing furnace is stabilized at 520°C.

The crystal is maintained at 520°C for about 24 hours before lowering the temperature at a rate of 20 deg./day. Two to three days after bringing the furnace to the room temperature, the crystal is taken out and is put in the oil. The crystal is kept in the oil bath for a day to ensure that the whole crystal has attained the room temperature.

The crystal is machined to a desired diameter on a lathe. During the machining process, oil is continuously poured to dissipate the frictional heat. The crystal after machining is cut to the required size and is transferred into a glove box where the humidity is maintained equal to or better than 15%. Both the sides of the crystal are then polished to the required smoothness. One face of the crystal which is to be optically coupled with glass plate is given the finish with tissue paper soaked in alcohol.

RESULTS AND DISCUSSIONS

The rate of growth of crystal for a given size is mainly controlled by the operating temperature and the rate of crucible lowering. From the measurement carried out for the growth rate at different operating temperatures, the temperature for proper growth was found to be near 655°C. Higher operating temperatures were found unsuitable as the freezing plane in such cases has a tendency of shifting down towards the lower zone with crucible lowering. This results in a concave solid/melt interface and the crystal grown normally had a defective central core as shown in Fig.1.

For a pulling rate of 1 mm/hour the movement of the solid/melt interface with respect to the crucible lowering was studied maintaining the operating temperature at 656 ± 2°C. The results obtained are shown in Fig.9. The growth rate increases from an initial lower value of 0.56 mm to 1.7 mm/mm lowering as the crucible is being lowered. This increase in the growth rate results from the increased conduction losses taking place in the lower zone as the crucible is lowered. The crystal grown under such conditions was found transparent only about 20 to 30 mm above the cone portion and the remaining cylindrical portion of the crystal has defective growth. The problem could be overcome in different ways such as, by suitable programming of the crucible lowering rate or programming the temperature of the controlling thermocouple in the upper zone, in such a way that a single growth rate is achieved irrespective of the crucible position in the furnace. The programming of the melt temperature is rather difficult to achieve due to the practical difficulties involved.

However, programming of the crucible lowering is simple and a desired growth rate could be maintained throughout the growth process. The results of the programmed lowering have been shown in Fig.10. In case of sodium iodide single crystal growth, a growth rate of 0.85 to 1.2 mm for every mm lowering of the crucible is conducive with an initial lag between the lowering of the crucible and the length of crystal grown as shown in figure.

A photograph of the typical detector fabricated from the crystal is shown in Fig.11. The energy resolution of the NaI(Tl) detector was studied using a photomultiplier tube RCA 8054. The energy spectrum of Cs^{137} gamma source obtained for a typical detector is shown in Fig.12. The energy resolution obtained from the plot is 10%. The energy resolution obtained depends upon several factors which could be classified as intrinsic and extrinsic. The intrinsic factors which affect the resolution are the material purity and crystal quality. The extrinsic factors include the effects arising due to the polishing of the different crystal surfaces and its canning[11]. The resolution of the detector improves in case the surfaces other than one which is coupled to the glass plate are made diffuse rather than highly polished. A partial explanation of this is due to Schuraliff and Jones[12] given in a description of trapping of light in symmetrical bodies of high index of refraction. The diffuse surfaces act to destroy the symmetry and prevent trapping. The use of aluminium or magnesium oxide powder surrounding the crystal as reflector is also useful for the same reason. Further, contrary to the observation

reported[13] elsewhere the annealing of the grown crystal near its melting point is seen to have hardly any significant effect on its resolution. The annealing, however, does improve the pulse height of the detector. The other factors which effect the pulse height are the presence of certain impurity ions[14], concentration of the activator[15] ions etc.. Further improvement in the characteristics of the crystals would require some detailed investigation.

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TABLE 1

GROWING FURNACE DETAILS

1.	Length of the muffle	508 mm (Upper zone)	330 mm(Lower zone)
2.	Diameter of the muffle	225 mm I.D,	250 mm O.D.
3.	Baffle	170 mm I.D.	
4.	Heating element	Nichrome (SWG - 16)	
5.	Thermocouple position	Tm-406 mm, Tr-482 mm, T_2-538 mm	
6.	Power requirement (for Sodium Iodide)	2.5 KW(Upper zone)	1.0 KW(Lower Zone)
7.	Insulation thickness	60 mm	
8.	Static air gap	50 mm	
9.	Lowering rate	19 mm/day.	
	i) Motor speed	950 RPM	
	ii) No. of gears & ratio	Three, Two of 400 : 1 and third 60:1	

TABLE - 2

ANNEALING FURNACE DETAILS

1.	Length of the muffle	500 mm
2.	Diameter of the muffle	245 mm I.D., 265 mm O.D.
3.	Insulator thickness	110 mm
4.	Heating element	Nichrome (SWG-16)
5.	Power requirement	2.0 KW

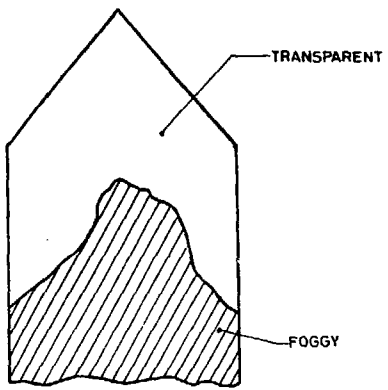

FIG.1 : SCHEMATIC OF A CRYSTAL GROWN UNDER IMPROPER CONDITIONS

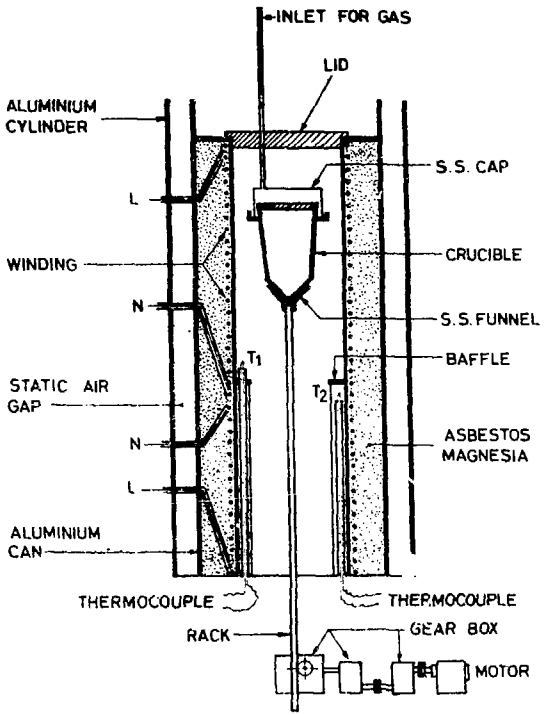

FIG.2 : SCHEMATIC OF BRIDGMAN-STOCKBARGER CRYSTAL GROWING FURNACE

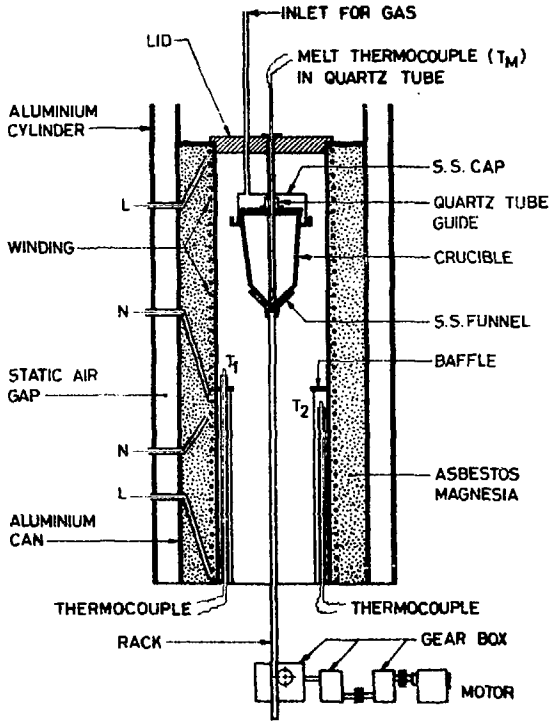

FIG. 3 : SCHEMATIC OF MODIFIED CRYSTAL GROWING FURNACE

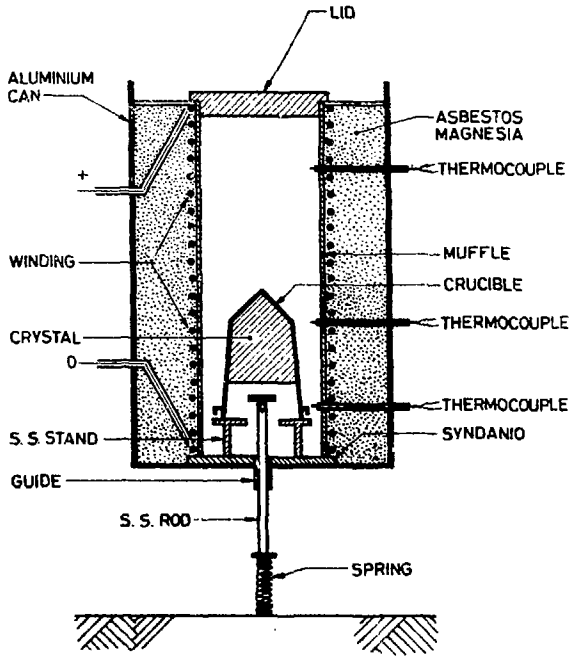

FIG. 4 : SCHEMATIC OF ANNEALING FURNACE

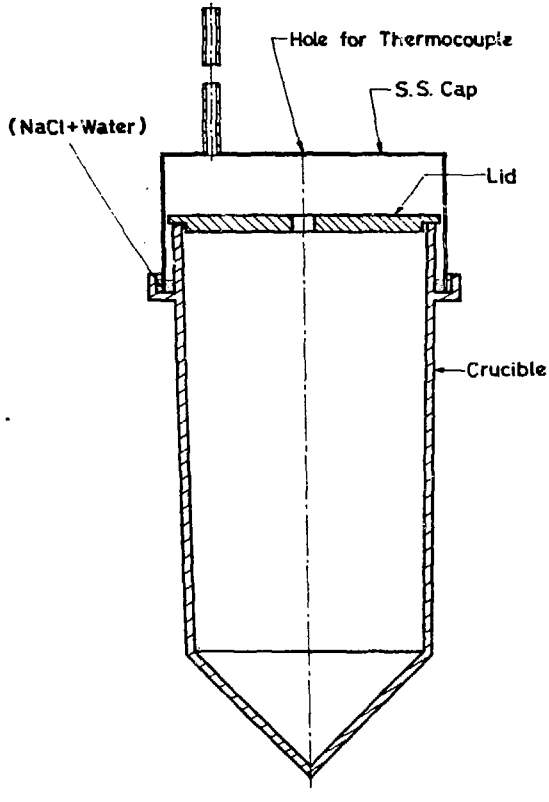

FIG. 5 : CRUCIBLE AND S.S. CAP ARRANGEMENT

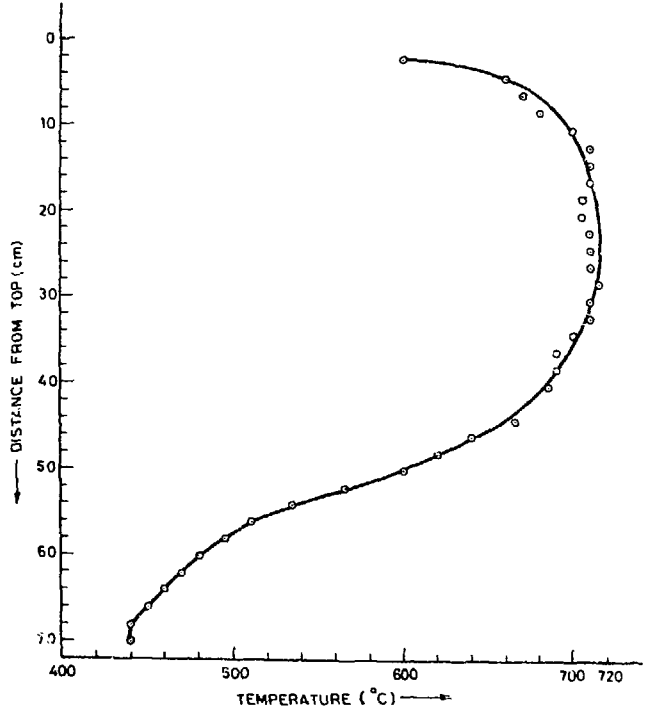

FIG. 6 : TEMPERATURE PROFILE

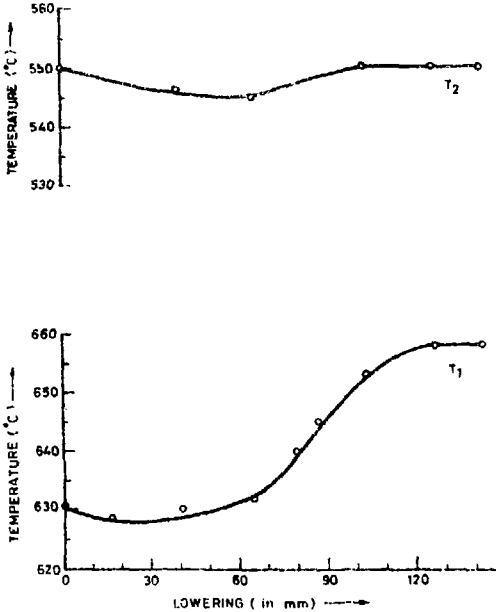

FIG 7 . TEMPERATURE RECORD OF THE THERMOCOUPLES PLACED 3 cms ABOVE (T_1) AND BELOW (T_2) THE BAFFLE

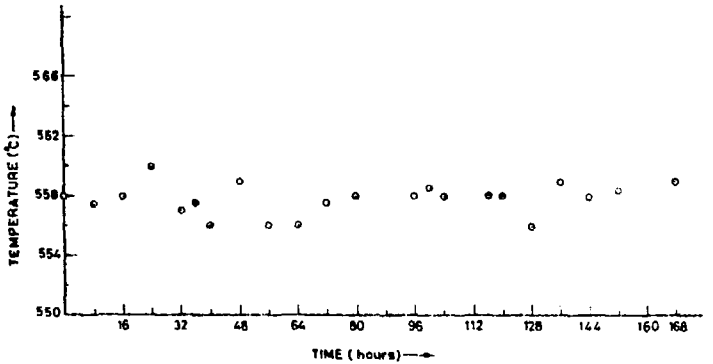

FIG. 8 . A TYPICAL TEMPERATURE RECORD OF THE CONTROLLING THERMOCOUPLE INSIDE THE MELT

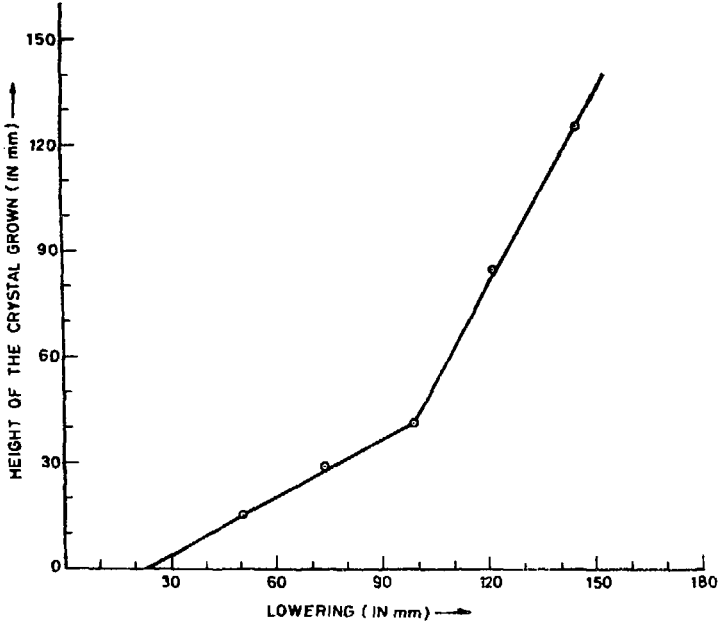

FIG. 9 : THE MOVEMENT OF THE SOLID / MELT INTERFACE FOR CONTINUOUS CRUCIBLE LOWERING

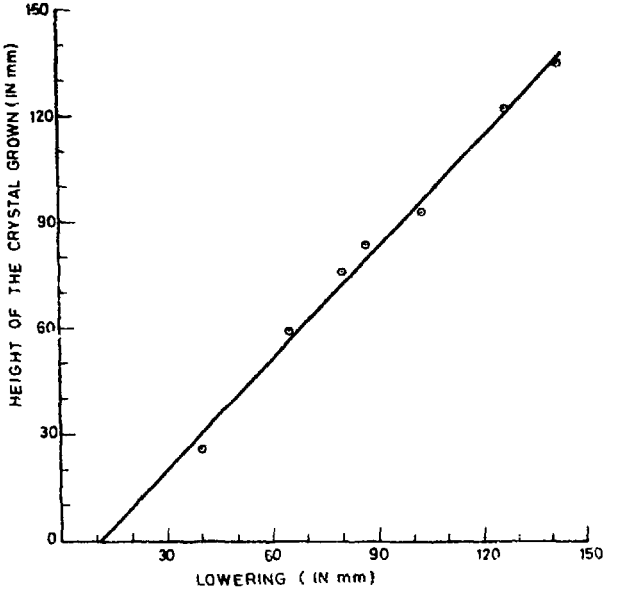

FIG. 10 : THE MOVEMENT OF THE SOLID / MELT INTERFACE FOR PROGRAMMED CRUCIBLE LOWERING

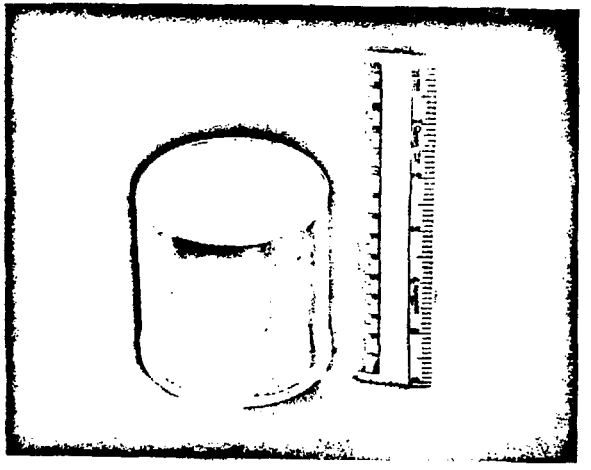

Fig. 11 Photograph of the detector

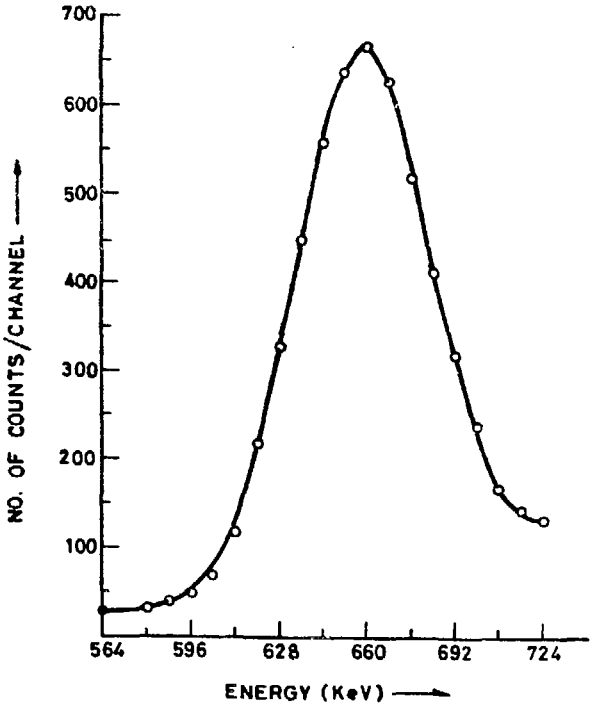

FIG. 12 : ENERGY SPECTRUM OF Cs^{137} Y SOURCE