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CONSTRUCTION OF LEAD GLASS TUBING MATRICES FOR APPLICATIONS IN MEDICAL PHYSICS AND HIGH ENERGY PHYSICS

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CONSTRUCTION OF LEAD GLASS TUBING MATRICES FOR APPLICATIONS IN MEDICAL PHYSICS AND HIGH ENERGY PHYSICS(*)

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Abstract

Honeycomb matrices which act both as gamma ray converter/radiator and electron drift structures have been manufactured from lead glass tubing of high density (5-6 g/cm^3). Baking the tubing in a reducing atmosphere produces a resistive metallic layer which can be used as a continuous voltage divider for drift field shaping. The application of a MultiWire Proportional Chamber/converter detector to Positron Emission Tomography is described: arrays of lead glass capillaries (< 1.0 mm inner diameter) are used as converter for the 511 keV annihilation photons. Another application is under study in High Energy Physics, a high density Projection Chamber in electromagnetic calorimetry. The various phases of the construction of these lead glass matrices for both applications are described in detail.

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1. Introduction

The detection of high energy photons in a gas filled medium is limited by the density and the absorption properties of the gas. Honeycomb arrays of lead glass tubing were originally developed at the Lawrence Berkeley Laboratory (1) to enhance the absorption and detection of photons.

In Positron Emission Tomography applications it is necessary to detect the two (511 keV, back to back) annihilation photons in two opposing detectors with high spatial resolution to reconstruct the β^+ emitter radioisotope distribution. A schematic drawing of a MWPC/converter detector is shown in fig. 1.

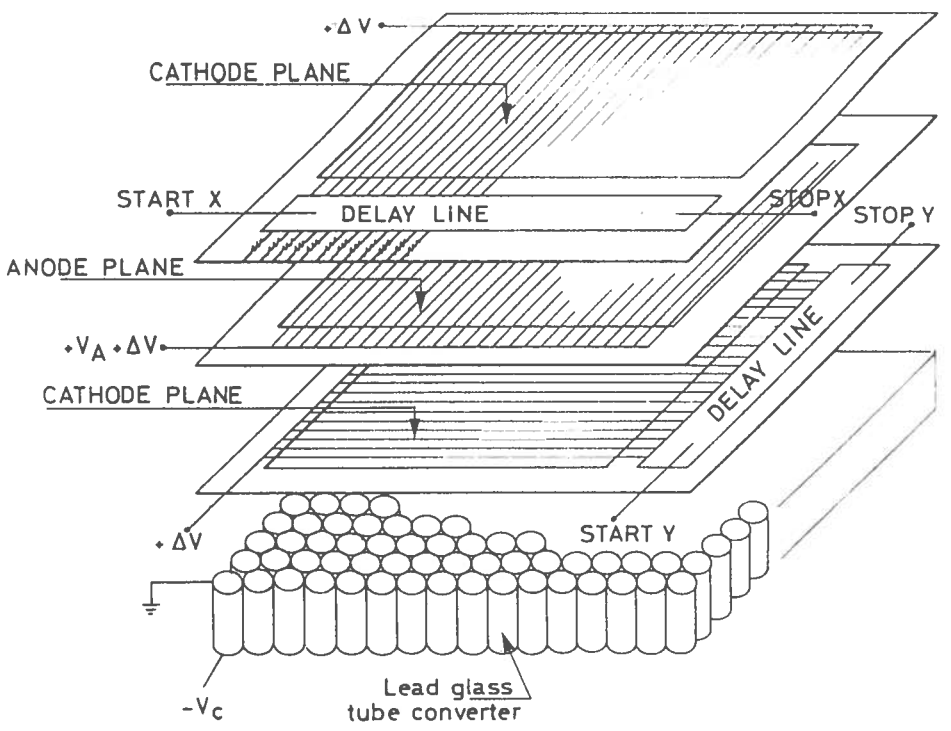


Fig. 1. Schematic drawing of a MWPC equipped with delay line readout and a single layer of lead glass tube converter.

The converter is made of lead glass capillaries fused to form honeycomb matrices. In order to have a high efficiency, a high surface to volume ratio is necessary. Thus, typical dimensions for the tubes are 0.5-2.0 mm inner diameter, 50-200 μm wall thickness, 1-2 cm drift space. The lead glass matrices are treated in a H_2 reduction process to form a uniform resistive layer on the inner walls of each tube. The Compton or photoelectron produced by the photon interacting within the converter has a finite range which depends on its energy. If it reaches the gas region within the tube, a number of primary ionization electrons are produced. A voltage difference applied between the ends of the tubes drifts these primary electrons along the electric field lines within the tube towards the chamber avalanche region.

Various size capillaries of different diameter and wall thickness have been tried. Our best results have been obtained with a matrix of lead-glass

tubing with 0.48 mm inner diameter, 0.06 mm wall thickness (80% PbO, density 6.2 g/cm^3), which gives a measured efficiency of 6.5% for a 1 cm thick converter (2). The experimental efficiency measurements for the various converter types agree very well with the Monte Carlo predictions (3).

Based on this type of MWPC/converter detector, a HIgh Spatial resolution Positron Emission Tomograph has been designed. It will consist of six modules arranged so as to form the lateral surface of a hexagonal prism. Each module of HISPET will have two MWPC and two 1 cm thick converter planes (0.48 and 0.60 mm ID and OD, respectively), see figure 2. HISPET will be capable of imaging three-dimensional distributions of a positron emitting radioisotope within a typical volume of 3 litres. It will have a volume sensitivity of ~ 100000 c/s per 0.1 μCi/ml, a signal to noise (true to accidental coincidences) ratio of 3:1 and an intrinsic spatial resolution of less than 4.5 mm (FWHM).

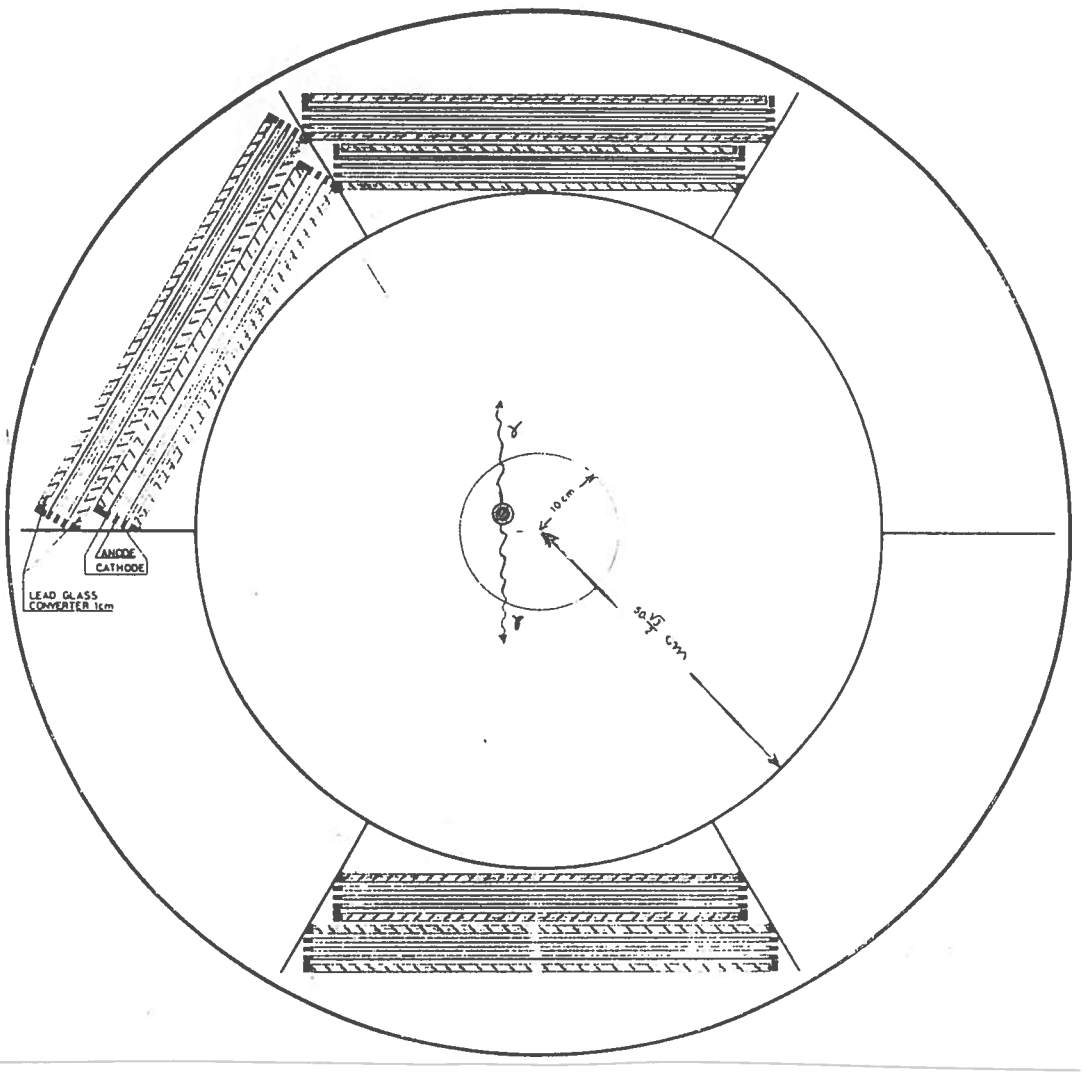


Fig. 2. Schematic drawing of HISPET: only three modules are shown.

We are also investigating the possibility of using this type of conver-

ter in High Energy Physics and particularly as an electromagnetic calorimeter. The proposed high density Projection Chamber (see fig. 3) is a method of achieving fine granularity in gas sampling which minimizes the number of readout channels. The z-coordinate (along the longitudinal direction of the shower) is directly read from the anode wire; the y-coodinate can be obtained by means of delay lines parallel to the anode wires or by charge division method. The third coordinate is given by the drift time of the electrons within the tubes to the anode plane. In this case tubes with inner and outer diameter of 5 and 7 cm, respectively, have been chosen. Experimental tests on small scale prototypes have been successfully completed. The construction of a 40 cm drift length, $\sim 20\,\chi_0$ calorimeter (CALTUB project) is now under study.

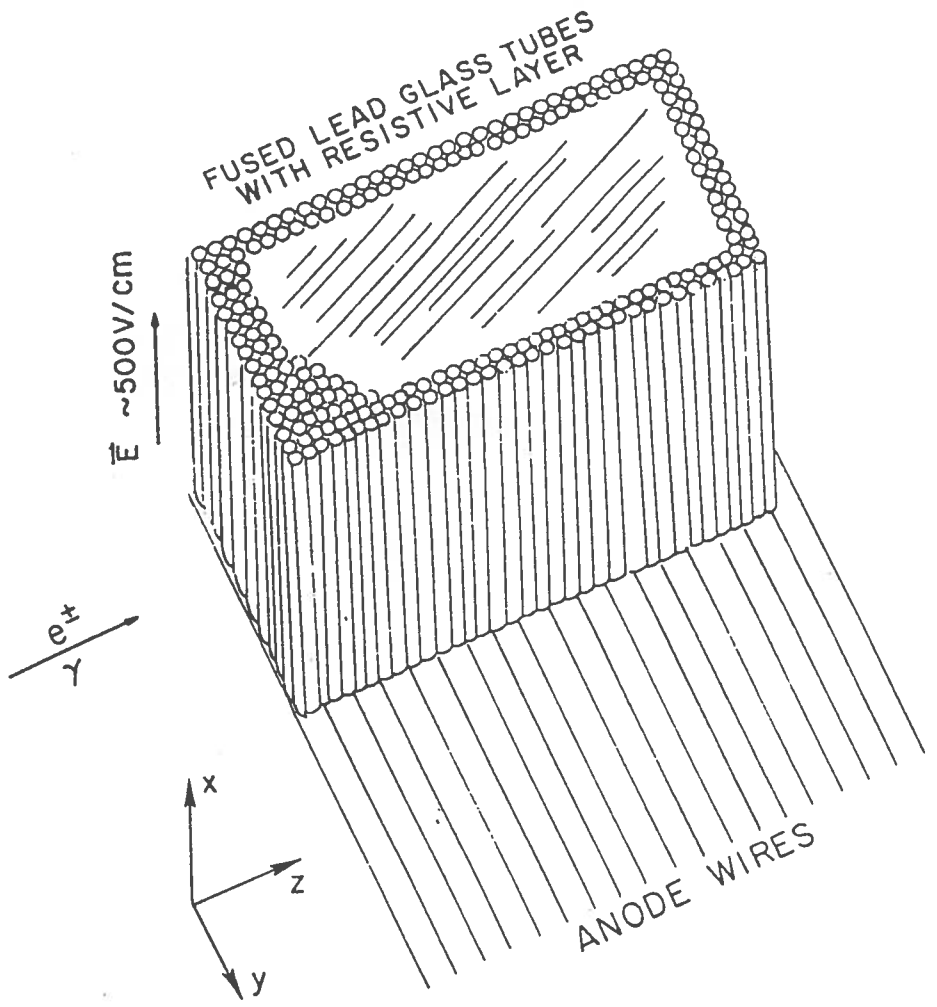


Fig. 3. Schematic drawing of the drift collection calorimeter with lead glass tubes (CALTUB project).

The HISPET and CALTUB projects are both dependent on the use of fused, reduced lead-glass tubes for their success. There are essentially six phases to the construction of the finished project. These are: 1) the cutting of the tubes and their stacking in a mold, 2) the fusing of the glass, 3) slicing and polishing of the fused glass, 4) cleaning of glass and HCl

treatment, 5) hydrogen treatment, 6) production of a conductive layer on the ends of the tubes. This report describes the progress made on each of these phases at the laboratories of ENEA, Frascati, during 1984.

2. Preparation of Glass Tubes for Fusing

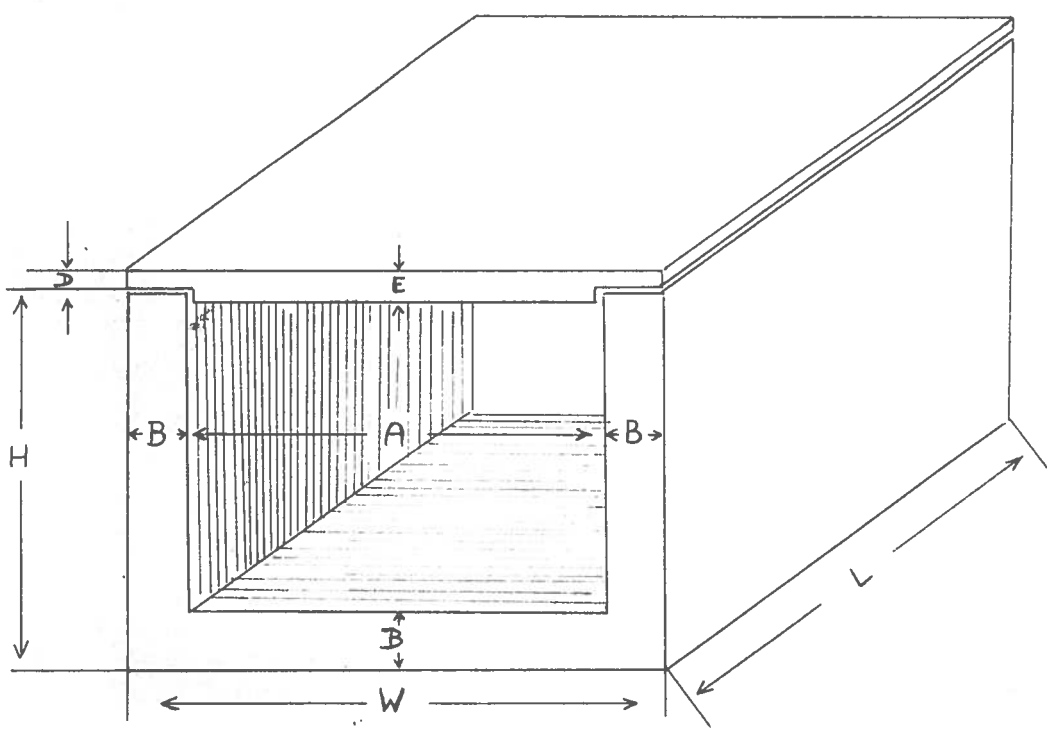
For the HISPET project we use the same methods of cutting the tubes that had been previously used at LBL (1). That is, the tubes (of small diameter, ranging between 0.5 and 1 mm, and wall thickness 50 to 100 μm) were lined up side by side, set flush at one end; lengths of tubing were marked off with masking tape, the tubes were nicked with a carborundum blade and the cut lengths were places in a mold.

The glass used for CALTUB presented a different problem. Since the tubes had 1 mm thick walls they could not simply be scratched and broken cleanly. To solve this we have been using a lathe, cutting each tube individually. A blade (actually a sharpened piece of Tungsten-Carbide) was set in place, and as the glass rotated in the lathe it cut itself; when a deep enough cut was made the tube was broken off. These tubes were also placed in a mold for the fusing.

The choice of material for the mold presented some problems. Several materials were tested for suitability as a mold: aluminum, asbestos, stumatite, iron painted with aquadag. None of these worked well; the glass attached to all (it came off easily from the iron, but took aquadag with it). The asbestos left an imprint on the glass as well as several threads. The glass simply stuck to the stumatite and aluminum. We also tested brass as a suitable surface. This worked well enough to merit production of two brass-lined molds (the bottom is covar, the sides are iron, and there are brass sheets lining the inside), one 2.5x2.5 cm^2, the other 5x5 cm^2. However, unless perfectly cleaned and polished, brass attaches to the tubes, causing lateral cracks in the CALTUB glass, and attachment to the side walls by the HISPET glass. We decided that graphite was the best material. Subsequently, three graphite molds were built: the dimensions are presented in figure 4. The large mold is used for construction of CALTUB modules, the small mold for testing; the middle mold is used for testing and for HISPET. We did have some problems with the graphite, too. With molds constructed of "poor-quality" graphite, after some fusing cycles we began to see some imprinting on the tubes and experienced difficulty in removing the fused glass from the mold. We have yet to see this problem with the high purity graphite (type P2239) we are now using.

3. Fusing of the Glass

For the fusion of the glass tubes we essentially followed the same prescription that was used at LBL (1). That is, we slowly raised the temperature on the first day to a point roughly 40°C below the softening point



(Dimensions in mm)	Large mold	Middle mold	Small mold
A	140	50	25
B	20	20	10
D	5	5	5
E	10	10	10
W	180	90	45
L	450	150	150
H	128	75	40

Fig. 4. Dimensions of the graphite molds.

of the glass. It was left to equilibrate overnight, then raised the following morning 10°C/hr, until the softening point of the glass was reached. (The softening point of the glass is defined as that temperature at which the glass deforms under its own weight.) The oven was periodically opened to check whether the mold had closed: a space was left between the mold and its lid to allow for compression of the tubes. A closed lid indicated that the glass had sagged and, therefore, fused. When the mold had closed, the oven was opened to allow the temperature to drop roughly 30°C, ending the fusing process. The oven was then set to the annealing temperature of the glass, which was then allowed to anneal for eight hours, after which the oven was turned off and allowed to cool. Naturally, this procedure was carried according to the properties of the different glass used. A list of the essential physical properties of the two types of glass we used is given in table 1.

TABLE 1

Physical properties of the glass used

	Nuclear Pacific Hi-D(★)	Schott RS-520
PbO Composition (by weight)	79%	71%
Density	6.2 g/cm^3	5.18 g/cm^3
Annealing temperature	397°C	423°C
Thermal expansion coefficient	9.5x10^{-6}/°C	8.1x10^{-6}/°C

(★) This glass was originally used at Lawrence Berkeley Laboratory (1).

Several 2.5x2.5 cm^2 and 5x5 cm^2 boules of Schott RS-520 glass have been successfully made. The softening temperature has always been roughly 480-490°C. We have also fused several boules of HISPET(with 0.6 mm and 1.0 mm OD) glass,type Nuclear Pacific Hi-D.

Recently,we have been using a large furnace in which we expect to fuse the 10x14x40 cm^3 tubes for the CALTUB modules. This oven is programmable, in that there is a brass cut-out which rotates at a rate of 1 rotation/36 h. In this way the fusing of the glass has been fully automated except for the decision ofwhen to stop the process. In fact, one failure of the system used to this point has been the need to open the oven at its highest temperature in order to view the position of the lid of the mold as it sagged. To this end we have developed a sensor that announces the closure of the mold.

The sensor was made as follows: a hole was drilled into two diagonally opposed corners of the base of the mold, into which was placed a cylinder of ceramic (for electrical insulation). Into the ceramic was placed a stainless-steel bolt, with a wide, thin, flat head. A second hole was bored into the side to provide the electrical lead (again insulated with alumina). A hole was drilled into the lid of the mold and a stainless-streel screw was inserted upside-down. It was held in place (and its height adjusted)by two stainless-steel nuts which also serve to hold the electrical lead. The heads of the screw and the bolt make contact when the lid of the mold closes. This completes an electrical circuit, ringing a bell, lighting a lamp, and alerting us that the lid has closed. This system has worked perfectly each time it has been used. We have fused several packs for HISPET and three packs of 10x14x45 cm^3 for CALTUB. A photograph of the mold with its sensor is shown in figure 5.

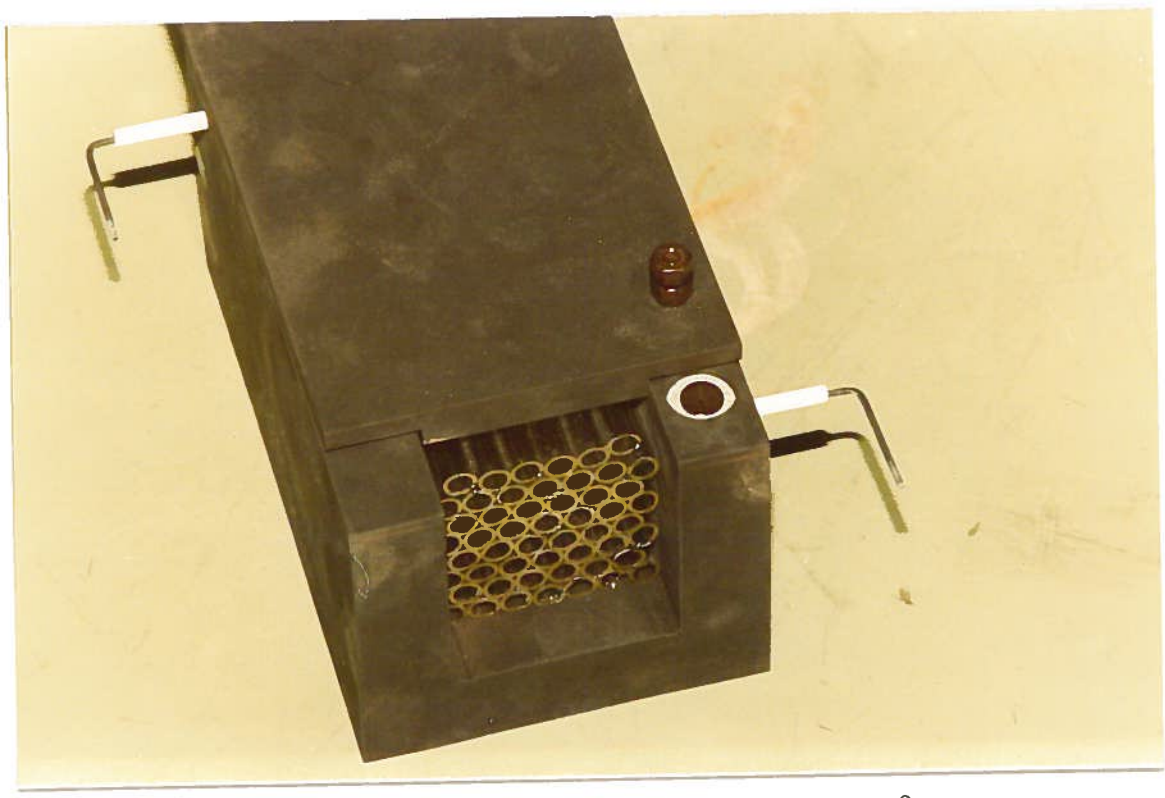


Fig. 5. Graphite mold with its sensors:a 5x5 cm^2 CALTUB prototype is inside the mold.

4. Slicing and Polishing of the Fused Glass

The tubes were first cut with a diamond saw (1.5 mm thick, rotating at 1000 rpm, external diameter 245 mm, with a grit of $\sim$ 200 μm). We cut (for CALTUB) two 4.5 cm and one 8.5 cm pieces. These were 5x5 cm^2. For HISPET we cut 5x1 cm thick slices of 2.5x2.5 cm^2 (1 mm OD) glass. In addition,four 1 cm thick slices and two 2 cm thick slices of 5x5 cm^2 (0.6 mm OD) glass were cut.

For the polishing of the sliced glass we tried several methods: use of a rotating wheel, upon which we put wet grit to polish the glass; wet

grit on a flat plane of glass, using various grit sizes. We tried alumina, cerium oxide, etc.. The smaller tubes seemed to nearly always clog; we broke off some of the tubes from the larger boule. We also tried using sandpaper - fine grit and wet. In the end it has decided that the best method of polishing the tubes was simply to use dry sandpaper. No dust was carried up the tubes, and they were left fairly unclogged.

5. Cleaning of Glass and HCl Treatment

The cleaning process is unchanged from that used at LBL (1). The glass is placed in an ultrasonic cleaner filled with the deionization water, and left for about five minutes. The cleaner is drained, then filled with acetone, into which the glass is placed for another five minutes. The acetone is drained from the cleaner, which is refilled with deionized water; the glass is bathed again.

As far as the HCl treatment is concerned, for HISPET we followed this procedure: 2 minutes soaking in a 0.1 molar HCl solution. This is for both the 1 mm OD and the 0.6 mm OD tubes. For the CALTUB glass, that is, for the 7 mm OD glass, we immersed the larger tubing in HCl for 5 minutes.

When the resistivity of the reduced glass was researched (the Schott 71% PbO glass had not been used before) and a minimum (with respect to temperature) found, we observed that it was higher than one would expect, given the data from Blodgett (5) for 60% PbO glass and our previous experience with 80% PbO glass (1). This led us to examine the effect of the acid treatment on the final resistence of the glass. As we know, the acid leaches out some PbO from the surface of the glass, leaving a very thin layer of SiO_2. This silica layer serves to protect the glass from water vapor. Since we had changed the time of the treatment, the creation of a thicker silica layer could in some way decrease the effective conductivity of the glass.

We decide to treat sample tubes (10 cm length, 5 and 7 mm ID and OD, respectively) in HCl for various times, up to 300 seconds, and measure the resistivity of the tubes before and after the hydrogen treatment. From the results, presented in Table 2, there is evidence that the resistivity tends to saturate after 60 s in HCl. However, as a safer precaution, we continued to treat the glass in acid for 5 minutes.

6. The Hydrogen Reduction Treatment

The basic procedure was as follows: glass to be treated was placed at the bottom of the oven, which was sealed and then evacuated. The temperature of the oven was slowly raised until the desired temperature was reached. The following morning hydrogen was introduced into the oven. As soon as the pressure reached atmospheric the outlet of the oven was opened, allowing

TABLE 2

Dependence of the resistivity upon duration of the HCl treatment

t in HCl (s)	R after H_2 reduction (Ω/tube)
0	130×10^9
30	212×10^9
60	270×10^9
120	275×10^9
300	307×10^9

the hydrogen to escape. This gas was lit, and, therefore, the escaping hydrogen was simply burned off. After the prescribed time of the treatment, the outlet was closed, as was the inlet; the oven was evacuated and then turned off. The glass was allowed to cool under vacuum. The H_2 gas was generally allowed to flow at a rate of 5-8 l/minute.

The first hydrogen treatments were performed on 10 cm long tubes of 7 mm OD. We searched for the minimum resistence of the glass vs. temperature of treatment. We found it at T=360°C; the resistance was $3.15 \times 10^{11} \Omega$ for a 10 cm tube. A graph of the measured resistence of a 10 cm long tube vs. temperature of the hydrogen treatment is presented in figure 6. All values are for six hour treatments.

One measurement that we tried to obtain was the resistence of the glass vs. time in the hydrogen atmosphere at the constant temperature. We have performed treatments for durations of from two to six hours as well as one of nine hours. The results are presented in figure 7. These measurements were all taken for treatment at 360°C.

For the HISPET project, the glass needed to be supported in a way that would ensure that none, or as little as possible, of the glass would be blocked; that is, that the hydrogen would be able to reach all points of the glass. A photograph of the support is shown in figure 8. In the photo, a one cm thick wafer rests in the support. The support was designed to hold 6 one cm thick slices; bars could be removed so that 2 cm thick slices could be treated. It was designed so that the pieces sit at an angle, exposing the maximum area of converter.

7. Production of Conductive Layer at the Ends of Tubes

We decided that the best method for producing this conductive layer for HISPET was vacuum deposition of a metal onto the surface of the converters. However, little work has been done: we have only seen that it is possible to do this work at ENEA, and we are trying to obtain a reproducible "system". We will be using nickel instead of molybdenum, as was originally used, since

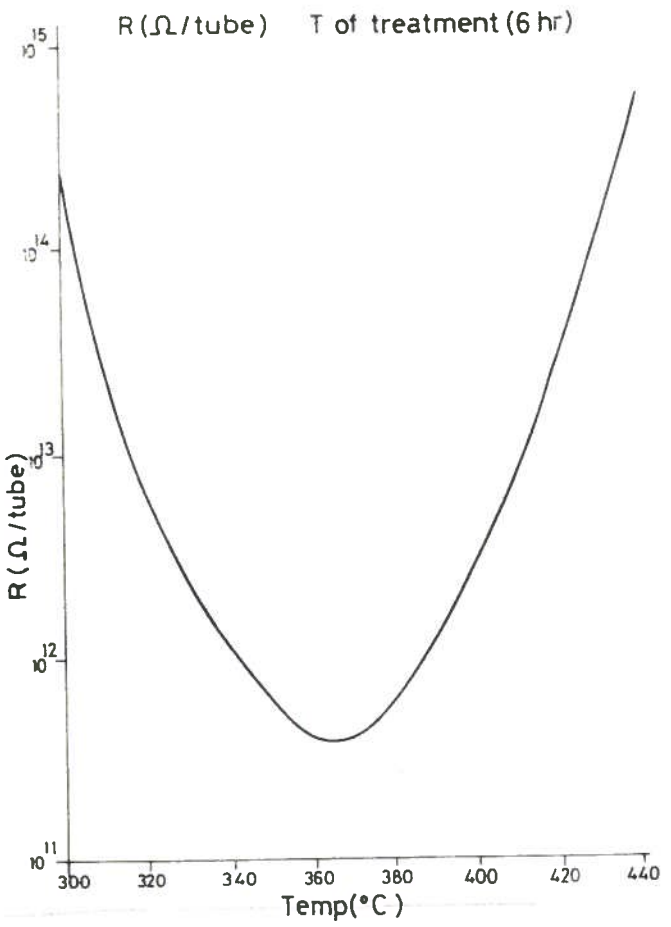


Fig. 6. Resistence of a 10 cm (5 cm ID) tube vs. temperature of the H_2 treatment

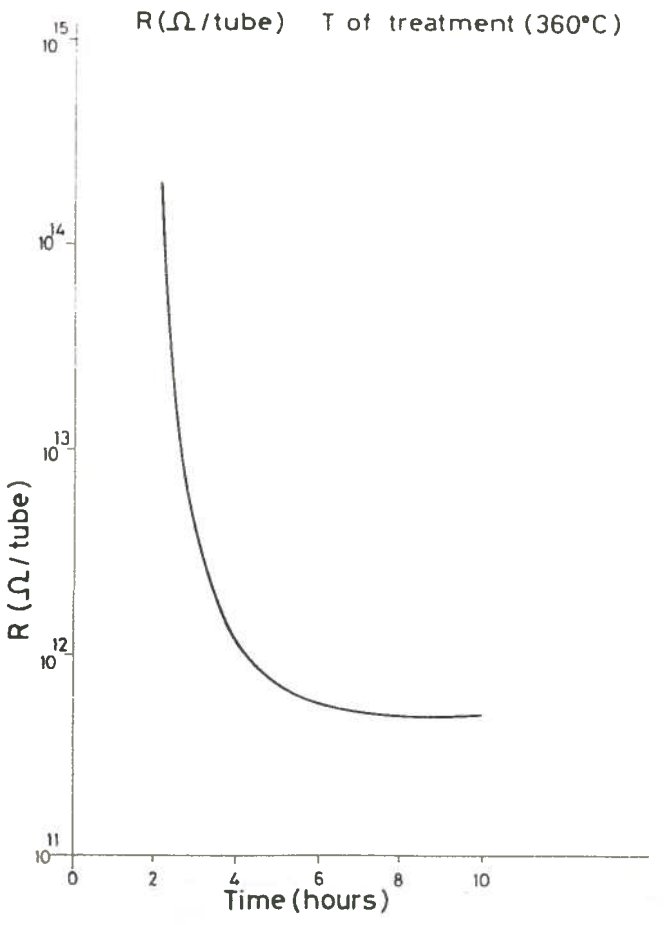


Fig. 7. Resistence of a 10 cm (5cm ID) tube vs. time of the H_2 treatment.

Fig. 8. Holder of the HISPET matrices for the H_2 treatment.

the high melting and boiling points of molybdenum make it difficult to use without burning out the system.

For CALTUB, this method would allow too much metal on the inside walls of the tubes, so we searched for another method. We first used silver print, a conductive paint used in circuitry, to achieve our uniform conductive layer. This did not work too well since the silver paint was too runny: in fact, the liquid entered the tubes even when brushed on upside-down. We settled on conductive silver epoxy, as previously used at LBL. The method of applying the epoxy, however, was much more ingenious that dabbing it on with rubber stoppers. A method not unlike that of a printing press was employed, in which we used a rubber roller to apply the epoxy. The layer on the roller was rolled very thin (the second surface was a flat piece of rubber) and then transferred to the glass (see figure 9). We were able to produce what looked to be a very uniform layer and the resistivity was less than 50 Ohms along the entire surface (5x5 cm^2).

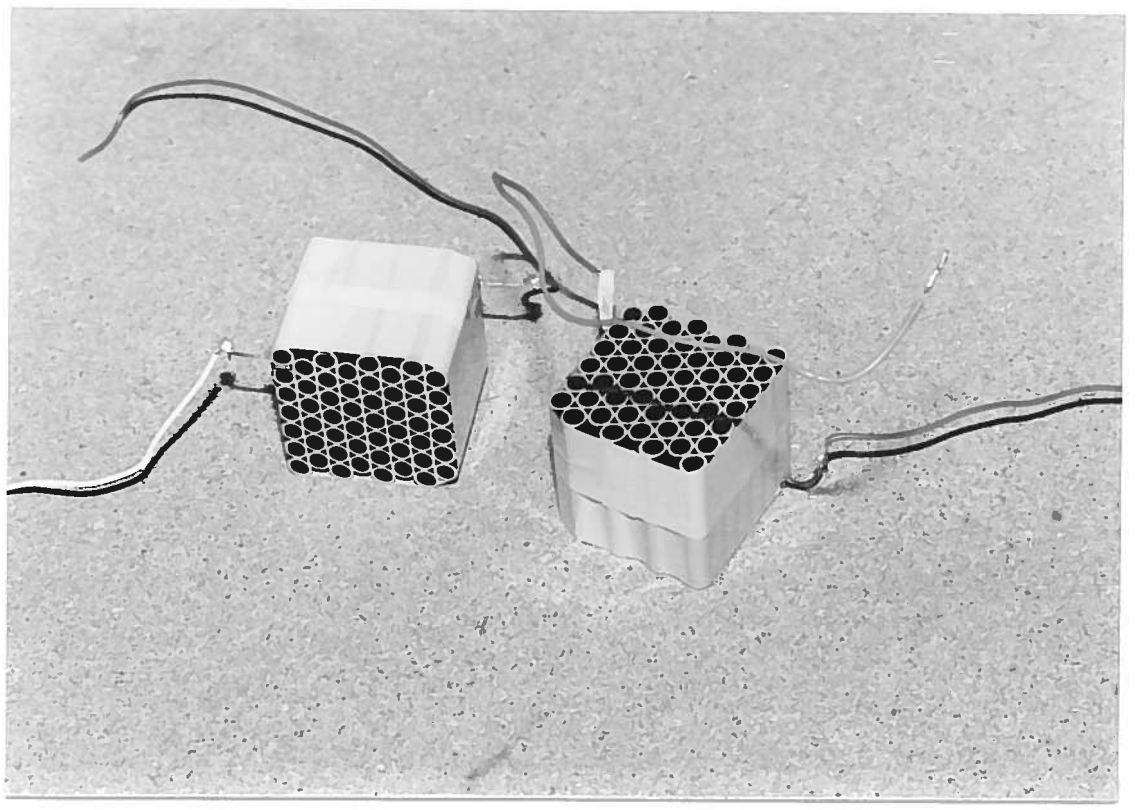
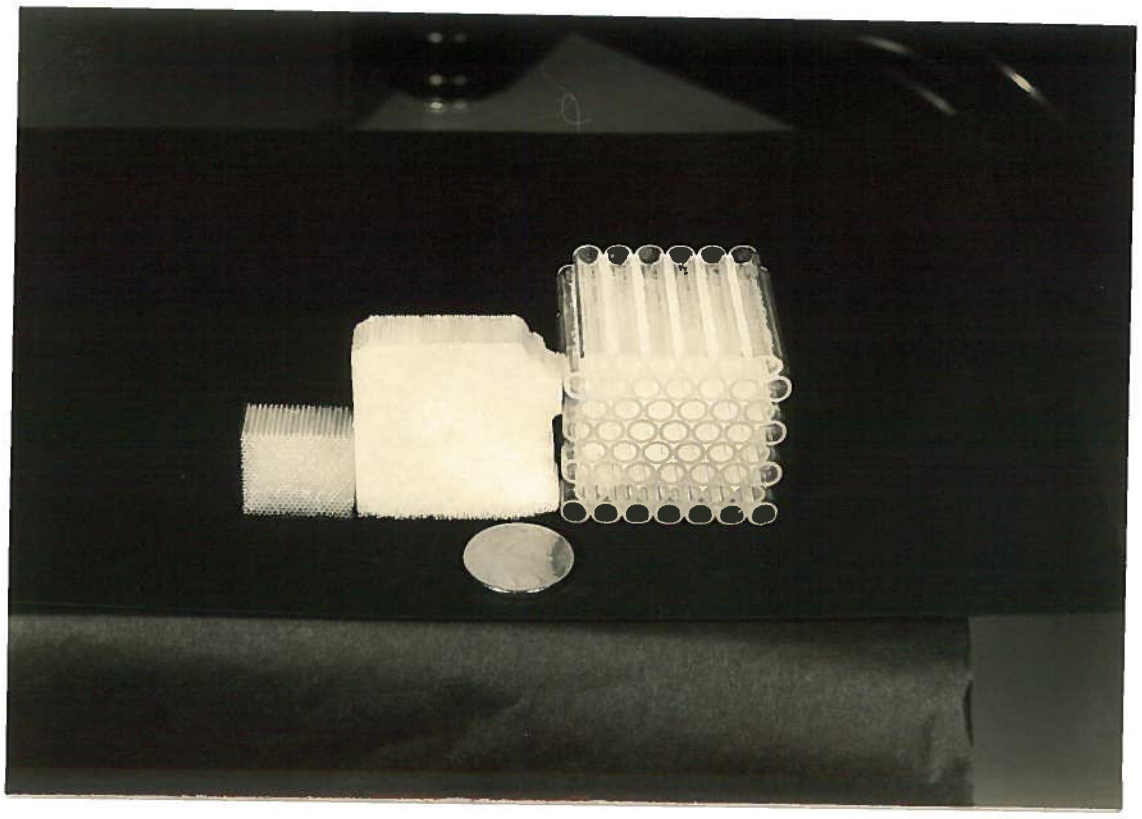


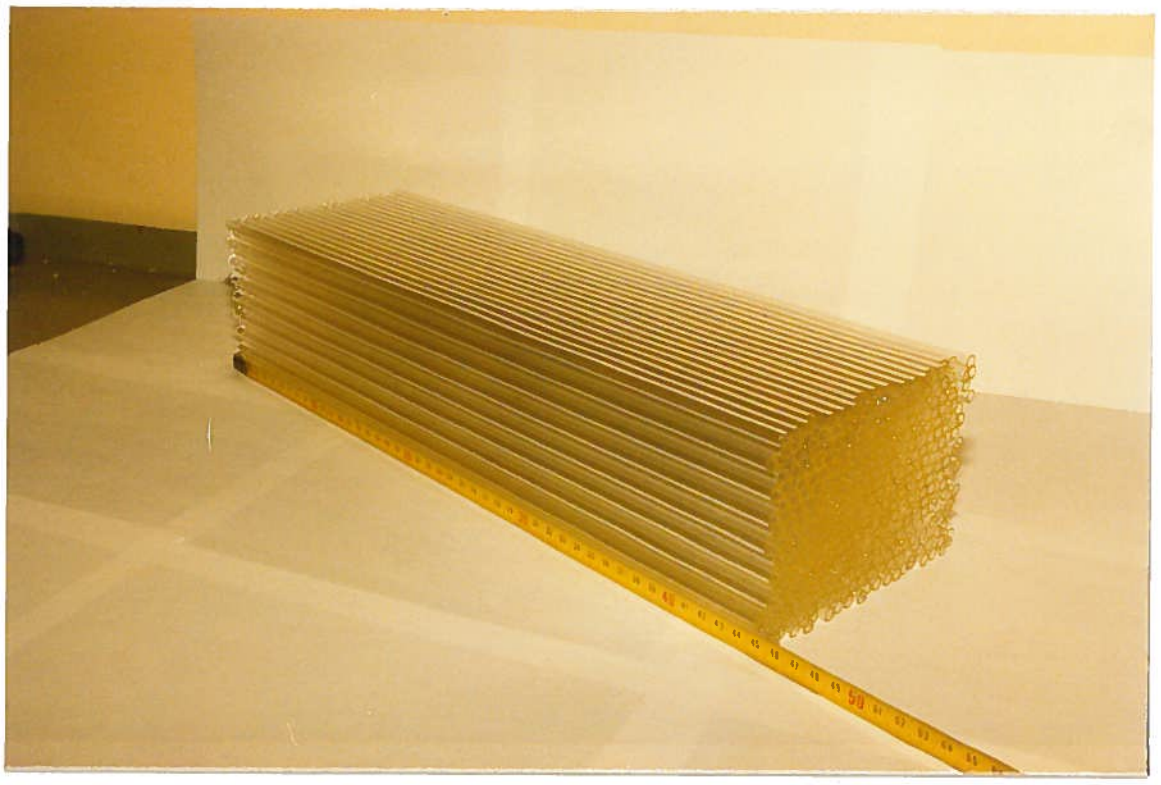
Fig. 9. CALTUB prototypes after being treated with silver epoxy.

8. Conclusions

We have made several working prototypes of lead glass matrices both for HISPET and for CALTUB projects (see fig. 10 a). The entire construction and assembly line has been established and fully tested. The two projects have now started and some final modules have already been completed (fig. 10 b).



(a)



(b)

Fig. 10. (a) Prototypes of lead glass matrices for HISPET and CALTUB projects; (b) a final module (10x14x40 cm^3) of the electromagnetic calorimeter, before being polished.

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