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**STATUS REPORT ON CERN ACTIVITIES AIMING AT THE PRODUCTION OF  
SPUTTER-COATED COPPER SUPERCONDUCTING RF CAVITIES FOR LEP**

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**ABSTRACT**

To upgrade LEP energy above 55 GeV, the first step will consist in installing 32 SC cavities of 352 MHz frequency at Point 2 of the machine. This operation will be carried out in steps and should be completed by the end of 1991. It has been decided that 8 of the 32 cavities will be Nb coated copper cavities, the crucial part of which (i.e. the cavity proper) will be manufactured and coated at CERN. For the time being, 4 of these 8 cavities have been prepared. They present  $Q_0$  values at low field of about  $10^{10}$ , while at the specified operating field of 5 MV/m their  $Q_0$  range between 5 and  $7 \times 10^9$ . In order to carry out assembly, coating and rinsing of cavities in better (i.e. cleaner) conditions, an experimental hall is being prepared, which will become operational after summer 1989, such as to be used for the manufacturing of the second batch of 4 coated cavities. In parallel with this main activity, some work is also being devoted to the study of coatings of higher  $T_c$  materials, namely NbTiN. Due to the higher  $T_c$ , these new coatings should present a lower BCS RF resistivity, a necessary condition to obtain higher  $Q_0$  values. The first cavity coated so far with NbTiN (a single cell cavity of 500 MHz frequency) gave encouraging results, which however are not better than what was obtained with a Nb film.

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## 1. Introduction

The energy upgrading of LEP (Large Electron Positron Collider) presently under construction at CERN, from the initial 55 GeV to about 90 GeV will require the installation on the machine of about 200 4-cell superconducting cavities (SC) of 352 MHz frequency<sup>1)</sup>. Each of these cavities should provide an accelerating field of 5 to 7 MV/m and a  $Q_0$  value not lower than  $3 \times 10^9$  at 5 MV/m and 4.2 K.

Traditionally, SC's are made of high purity Nb sheets by lathe spinning and welding. A development program for cavities prepared in this way was started at CERN in 1979<sup>2)</sup>; their feasibility has been shown and prototypes have been produced from industry<sup>3)</sup>. In spite of being produced using high purity Nb sheets, this type of cavity suffers from the relatively poor thermal conductivity of Nb at liquid helium temperatures. This may result in thermomagnetic breakdown of the cavity, because it permits the inner cavity surface to exceed the critical temperature whenever a local surface defect results in a large RF power absorption.

To minimize this risk, the purity of Nb has been increased and its thermal conductivity enhanced from 10 to about  $80 \text{ Wm}^{-1} \text{ K}^{-1}$  with beneficial effects on cavity stability.

Another possible solution to this problem consists in reducing the thickness of the superconducting Nb by coating with a Nb thin film a cavity made of high purity copper (OFHC, with thermal conductivity at 4.2 K of about  $400 \text{ Wm}^{-1} \text{ K}^{-1}$ ). Due to the Meissner effect which limits the RF field penetration in a superconductor to a very thin superficial layer, a Nb film 1  $\mu\text{m}$  thick is sufficient to shield completely the more resistant underlying copper substrate. In addition to the higher thermal stability, the Nb/Cu solution brings about other advantages, namely a surface free from macroscopic resistive inclusions and a saving on the cost of Nb. Furthermore, as more recently discovered<sup>4)</sup>, Nb coated cavities are insensitive to the presence of external static magnetic fields. This feature renders superfluous the often complicated magnetic shielding. For these reasons, a development program was undertaken at CERN in 1980, aiming at producing SC's by coating copper cavities with Nb films<sup>5)</sup>.

Coating is at present carried out by means of a cylindrical magnetron sputtering configuration. This configuration makes use of a cylindrical cathode concentric to the cavity axis and containing some electromagnets (fig. 1). The magnetic field produced by the solenoids traps the electrons on closed orbits, resulting in higher ionization efficiency and, consequently, in enhanced sputtering rate. The solenoids are kept at fixed position and their distribution, shown in fig. 1, allows coating of all parts of a LEP cavity and also of the tubes of the coupling ports. Figure 1 also shows 2 magnets placed outside the cavity which are used to produce a gettering film for impurities prior to cavity coating.

Last year (summer 1988), some full size LEP cavities were successfully coated<sup>6</sup>). The results obtained at that moment, summarized in fig. 2, indicate that Nb coated LEP cavities are feasible.

However, the reliability of the coating process, which is an essential requirement to envisage industrial production of cavities, remained to be demonstrated. Furthermore, no experience was available with coated cavities in real machine operating conditions.

Therefore, it has been decided to produce some of these cavities at CERN and to install them in LEP as soon as possible. Producing a significant number of coated cavities would also serve the purpose of gaining experience on the reproducibility of the coating process and of optimizing the various steps of the cavity manufacturing sequence.

## 2. Manufacturing sequence

The main steps of cavity manufacturing are the following:

- (i) production of half-cells (by lathe spinning) and of the cut-off tubes;
- (ii) chemical treatment of the cavity components before welding;
- (iii) welding of components;
- (iv) chemical cleaning of the complete cavity;
- (v) coating;
- (vi) final rinsing.

For each step, a "standard recipe" has been established which leads to good performance if strictly applied. However, in view of possible cost reduction for series production it is convenient to simplify as much as possible the manufacturing operations. For this reason, some simplifications have been introduced in the preparation of some cavities. The standard recipes and the tentative simplifications are given below.

## 2.1 Production of components

Presently, half-cells are produced by lathe spinning and the cut-off tubes by rolling and welding a metal sheet according to the procedure already applied for the manufacturing of Nb cavities. As an alternative for cell manufacturing, hydroforming has been envisaged and studied. At this stage, complete 5-cell cavities of 1.3 GHz have been produced by hydroforming at CERN<sup>7)</sup>, and the tooling for hydroforming LEP cavities has been designed but not yet fabricated.

As far as the cut-off tubes are concerned, an obvious simplification would consist in using prefabricated tubes without welds, but this may only be envisaged if a relevant quantity of tubes is required, as will be the case for series production of LEP cavities.

## 2.2 Welding of components

All welds are presently carried out by means of an Electron Beam (EB) gun. Problems were experienced, particularly at the equatorial weld, when an external beam-gun had been used. Now all cavity welds (except for those of the coupling ports) are carried out from the inside using an internal EB gun<sup>8)</sup>. Since this procedure was adopted, 6 cavities have been produced without any problems.

## 2.3 Chemical treatment of components

This treatment is necessary to obtain good welds. The standard recipe consists of degreasing the components in perchlorethylene vapours, cleaning in a sulphonitric solution and passivating in a sulphochromic solution. Electropolishing has also been tried with good results.

However, the chemical treatment at this stage may also deserve the purpose of removing from cavity components the surface layer which is damaged by mechanical forming. This removal may be carried out after completion of the cavity (as described in sect. 2.4), but it would be easier on components, particularly if electropolishing is envisaged. This modification will be tried out in the near future.

#### 2.4 Final chemical cleaning

This operation serves the purpose of removing the copper damaged surface layer and of providing a smooth surface. Metallographic inspection of the copper surface after cavity formation indicates that mechanical damage extends over a thickness of about 50  $\mu\text{m}$ . Usually, about 80  $\mu\text{m}$  are removed by the final chemical cleaning. The chemical solution consists of a mixture of sulphamic acid, hydrogen peroxyde, ammonium citrate and n-butanol<sup>9</sup>).

The resulting surface roughness  $R_A$  is of the order of 0.1  $\mu\text{m}$ . This treatment is followed by rinsing at first with demineralized water (final stage high purity, dust-free water with resistivity higher than 18 MOhm cm) and then with analytic grade ethanol.

The rinsing with alcohol prevents the formation of drying stains which are likely to appear if the cavity is dried in air after water rinsing. Drying stains may result in poor adhesion of the Nb coating and in the formation of "blisters" and/or film peel-off.

An interesting alternative to the described procedure consists in eliminating the alcohol rinsing and drying the cavity by evacuating it with an oil-free pumping system.

To improve on reproducibility, the chemical treatment has been completely automatized.

## 2.5 Coating

Coating of a LEP cavity is carried out in several steps, namely:

- i) Upper and lower manifolds (in sequence) to produce a gettering action and trap gas impurities outside the cavity (pressure  $5 \times 10^{-4}$  Torr, cathode voltage -700 V, cathode current 2 A, duration of the sputtering 4 min).
- ii) Extremities of the cut-off tubes (in sequence) ( $5 \times 10^{-4}$  Torr, -400 V, 7 A, 7 min).
- iii) Cut-off tubes in front of the connecting ports (in sequence) ( $8 \times 10^{-4}$  Torr, -400 V, 15.6 A, 27 min).
- iv) Outer cells (in sequence) ( $5 \times 10^{-4}$  Torr, -400 V, 15.6 A, 50 min).
- v) Central cells (in sequence) ( $5 \times 10^{-4}$  Torr, -400 V, 15.6 A, 75 min).

The cathode voltage is kept constant while the cathode current is stabilized by varying the magnetic field produced by the solenoid. During coating, the cavity is kept at 200°C. As previously reported<sup>6)</sup>, heating during coating helps in reducing the slope of the cavity Q(E) curve.

Besides the coating parameters, the absence of any solid particle on cavity walls is of vital importance for a successful coating. Many disappointing results<sup>6)</sup> may be attributed to the presence of foreign particles which, buried under the Nb film, spoil its cooling and result in "hot spots" or "blisters". To avoid dust pick-up during storage, the cathode is kept in a class 100 clean room. Unfortunately, our existing clean room is not large enough to house the operation of inserting the cathode into the cavity. An improvement of cavity performance, on a statistical basis, may be expected when a larger class 100 clean room will become operational after summer 1989.

### 3. Results

Since June 1988, nine coatings have been carried out on four different cavities.

Some of these coatings have been executed on cavities prepared according to "standard" recipes, as discussed in sect. 2. The purpose of these coatings was to gain information on the reproducibility of the process.

The other coatings served the purpose of ascertaining if any one of the standard recipes could be made simpler, in view of industrialization of the coating process.

On this general philosophy the additional constraint was imposed of producing three good cavities to be installed on LEP at the beginning of 1990 together with the coated cavity which was already available<sup>6</sup>).

The results, discussed in more detail in the following paragraphs, showed that good coatings could only be obtained by strictly following the standardized procedures. None of the others was acceptable for LEP.

#### 3.1 Cavities produced following "standard" procedures

The results obtained are shown in fig. 3. The best of these cavities (curve 5 in fig. 3) presents a  $Q_0$  value at 4.2 K of  $1.2 \times 10^{10}$  at low field and about  $7 \times 10^9$  at the specified field of 5 MV/m. The residual resistivity of this cavity is lower than  $2 \text{ n}\Omega$  and its  $Q_0$ , scaled according to the inverse square of the frequency ratio, closely corresponds to the  $Q_0$  of the best single-cell cavities of 500 MHz frequency coated so far<sup>6</sup>). For these reasons cavity 4 may be assumed to provide the best performance which may be obtained with cavities of this type, unless the present slope of the  $Q_0$  (E) curve is improved.

The two other cavities present  $Q_0$  values of about  $5 \times 10^9$  at 5 MeV/m. For one of them  $Q_0$  exceeds  $10^{10}$  at low field but it decreases rapidly with increasing field due to some small defects. The other one did not show any hot spot on temperature map nor sudden decrease on the  $Q_0$  (E) curve, but its  $Q_0$  at low field is somewhat lower ( $8.5 \times 10^9$ ).

As far as the maximum accelerating fields are concerned, cavity 5 reached 9.4 MV/m with RF processing only (10 h); cavity 4 reached 8.2 MV/m after 5 h of helium processing and cavity 3 required 17 h of helium processing to reach 6.8 MV/m.

It is worth noting that a fourth cavity prepared in the standard way was limited at an accelerating field of 2 MV/m because of a large Nb film peel-off consequent to the presence of a piece of stainless steel on the cavity wall. This piece was tightly incrustated in the copper and it has been ground off. After this operation and chemical removal of the Nb film, this cavity has been coated again. The subsequent coating gave the results shown by curve 4 of fig. 3.

### 3.2 Simplified production procedure

Four of the five other unsuccessful coatings have been a consequence of improper/simplified rinsing. These failures have been originated by stains (particularly in the upper cut-off tube) produced by insufficient rinsing of the upper flange of the cavity and aggravated by the concomitant suppression of alcohol rinsing in the final chemical treatment prior to coating.

This type of failure has been completely suppressed by opening the upper flange before water rinsing and adding a final rinsing with ethanol.

In the fifth case, the final chemical treatment did not result in a shiny surface as it does usually. Instead of repeating the treatment, a rinsing with sulphamic acid has been applied, but apparently this mild cleaning was insufficient.

## 4. Other development work on Nb coatings

A smaller cylindrical magnetron sputtering configuration has been developed to coat 1.3 GHz cavities for Saclay. At distinction with the previous work, permanent magnets instead of electromagnets have been used to produce the discharge magnetic field.

The results are encouraging but a complete evaluation of the performance of these cavities to be carried out at Saclay is still missing.



## 5. Coatings of NbTiN

Reactive sputtering is a well established procedure to produce niobium nitride on carbonitride films starting from a Nb cathode. This method consists in adding, to the argon which produces the sputtering discharge, a certain amount of  $N_2$  (and  $CH_4$  for the carbonitride). These compounds, having a higher  $T_c$  (values up to 17 K are reported in the literature<sup>10</sup>), et 11), should result in a lower RF surface resistivity (BCS) at 4.2 K and hence in higher Q values.

The main difficulty presented by this approach is that to obtain high  $T_c$ 's the cavity should be heated at 500-700°C, which is not feasible in our case for mechanical reasons. Coatings carried out inside a cavity heated at 200°C, i.e. the maximum permissible temperature, did not result in  $T_c$  values higher than 13 K. Furthermore, the discharge was usually unstable and the results not very reproducible.

It has been found that most of these inconveniences could be removed by using a cathode made out of a Nb-Ti alloy<sup>12</sup>). Starting from a cathode of nominal composition Nb 54%, Ti 46% (weight percentages), critical temperatures up to 15 K have been obtained on samples placed at the equator of a cavity kept at 200°C during coating.

Following these results on samples, a 500 MHz single-cell cavity has been coated in this way. The results are given in fig. 4. An  $R_{BCS}$  at 4.2 K of about 20 n $\Omega$  has been obtained, which is about a factor 2 lower than that measured on the best Nb coatings. However, this low BCS resistivity is spoiled by a high residual resistivity (about 40 n $\Omega$ ) such as to result in a  $Q_0$  value at low field and at 4.2 K marginally lower than for the best Nb coatings ( $4 \times 10^9$  instead of  $6 \times 10^9$ ). Unfortunately  $Q_0$  deteriorates very rapidly with increasing field, and it is much lower than specified at the nominal LEP field value of 5 MV/m.

The results of fig. 4 have been obtained at the first attempt. Therefore, it is easy to predict that some improvement will be achieved by varying the deposition parameters and/or the composition of the cathode. It is known<sup>12</sup>) that a Nb 55%, Ti 45% (atomic percentage) cathode composition should provide  $T_c$  values up to 17 K.

However, the values of the residual resistivity, and its dependence on accelerating field are unpredictable. Furthermore, the practical difficulties presented by these coatings are much more relevant than for Nb coatings. For instance, a variation of 5% on N<sub>2</sub> pressure or Nb-Ti deposition rate may result in a T<sub>c</sub> variation of about 2 K.

Any performance improvement along this line may only be obtained at the price of a much more careful control of the deposition process.

## 6. Conclusions

Although some of the equipment foreseen to guarantee full reproducibility of the coating process is still missing, the results obtained are encouraging in this respect.

Simplifications of the cavity cleaning did not work so far but the small statistics does not provide conclusive proof.

The four cavities which should be installed in LEP at the beginning of 1990 are ready and in the process of being mounted in their cryostat.

Coating has been extended to cavities of 1.3 GHz frequency.

Niobium-titanium nitride seems to be a good candidate to provide higher Q<sub>0</sub> values at low field. However, some doubts remain concerning its behaviour at higher fields. Furthermore, the setting of the deposition parameters is very crucial in this case.

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## Figure Captions

- Fig. 1 Schematic view of a LEP cavity with cathode for magnetron sputtering.
- Fig. 2  $Q_0(E)$  curves of the first LEP Nb coated copper cavities (curves 1 and 2) compared to the best Nb cavity produced at CERN.
- Fig. 3  $Q_0(E)$  curves for the 3 LEP Nb coated copper cavities produced this year.
- Fig. 4  $Q_0(E)$  curves for a 500 MHz single-cell copper cavity coated with NbTiN. Upper curve, measurements taken at 2.6 K; lower curve, measurements taken at 4.2 K.

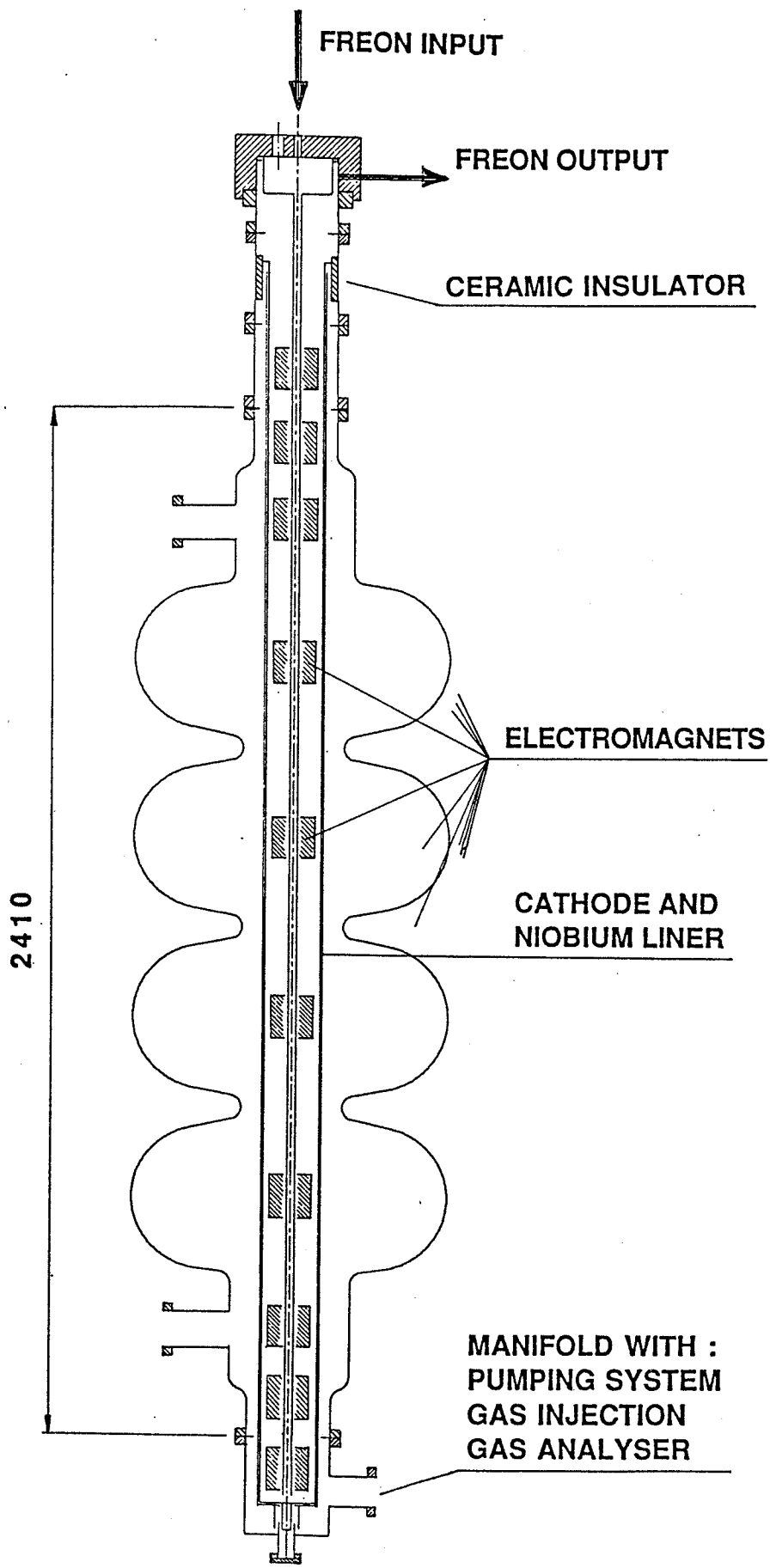


Fig. 1

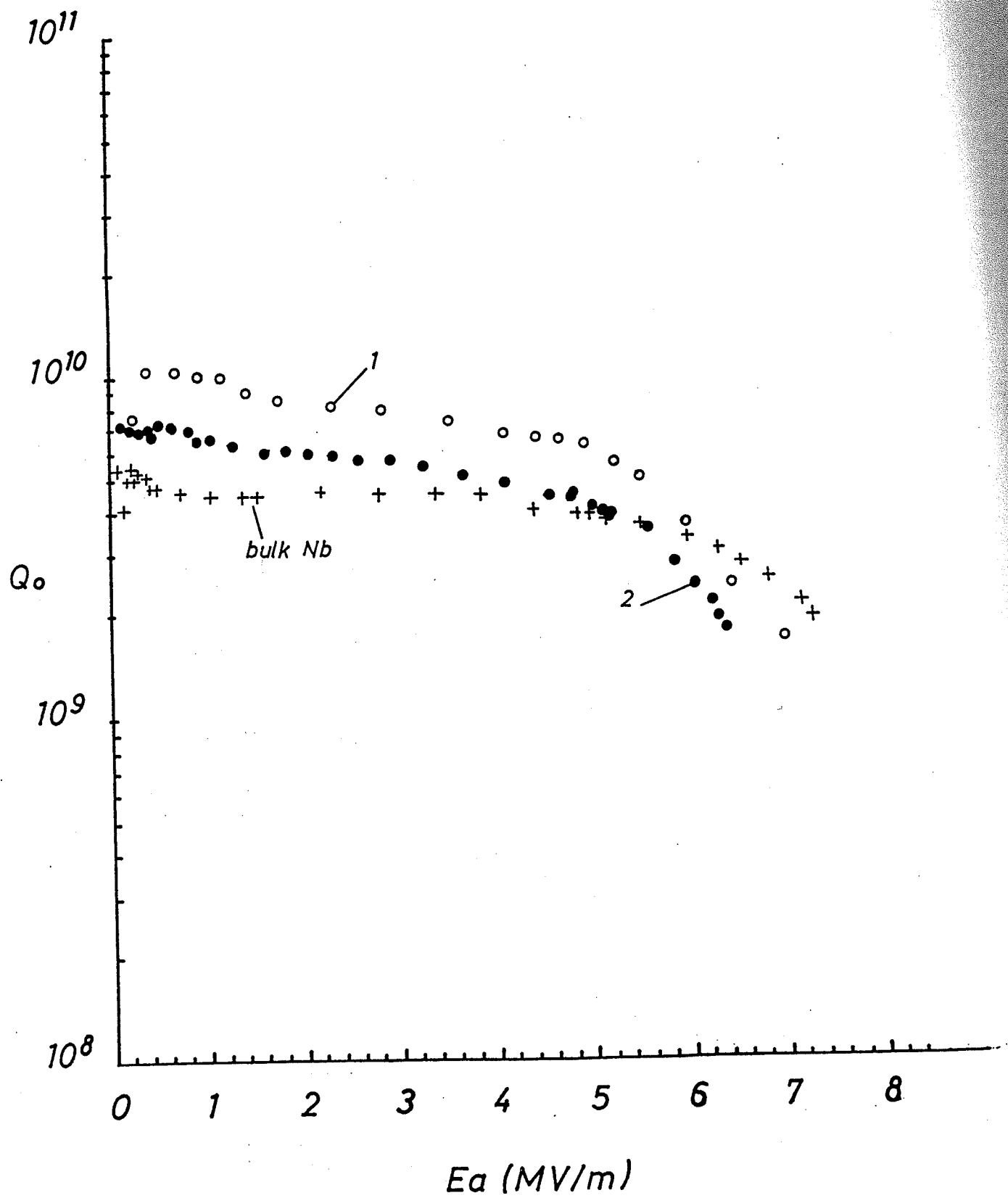


Fig. 2

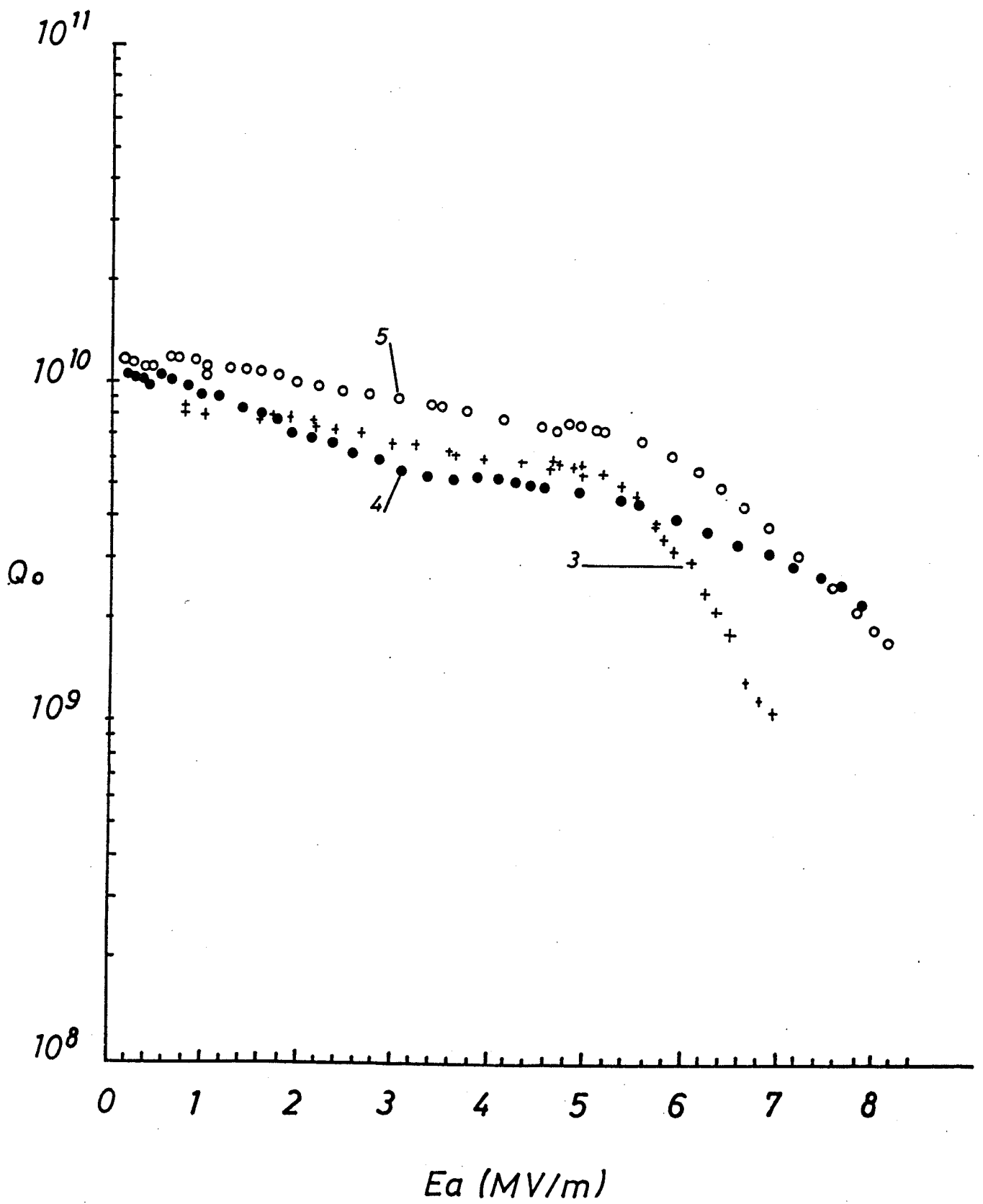


Fig. 3

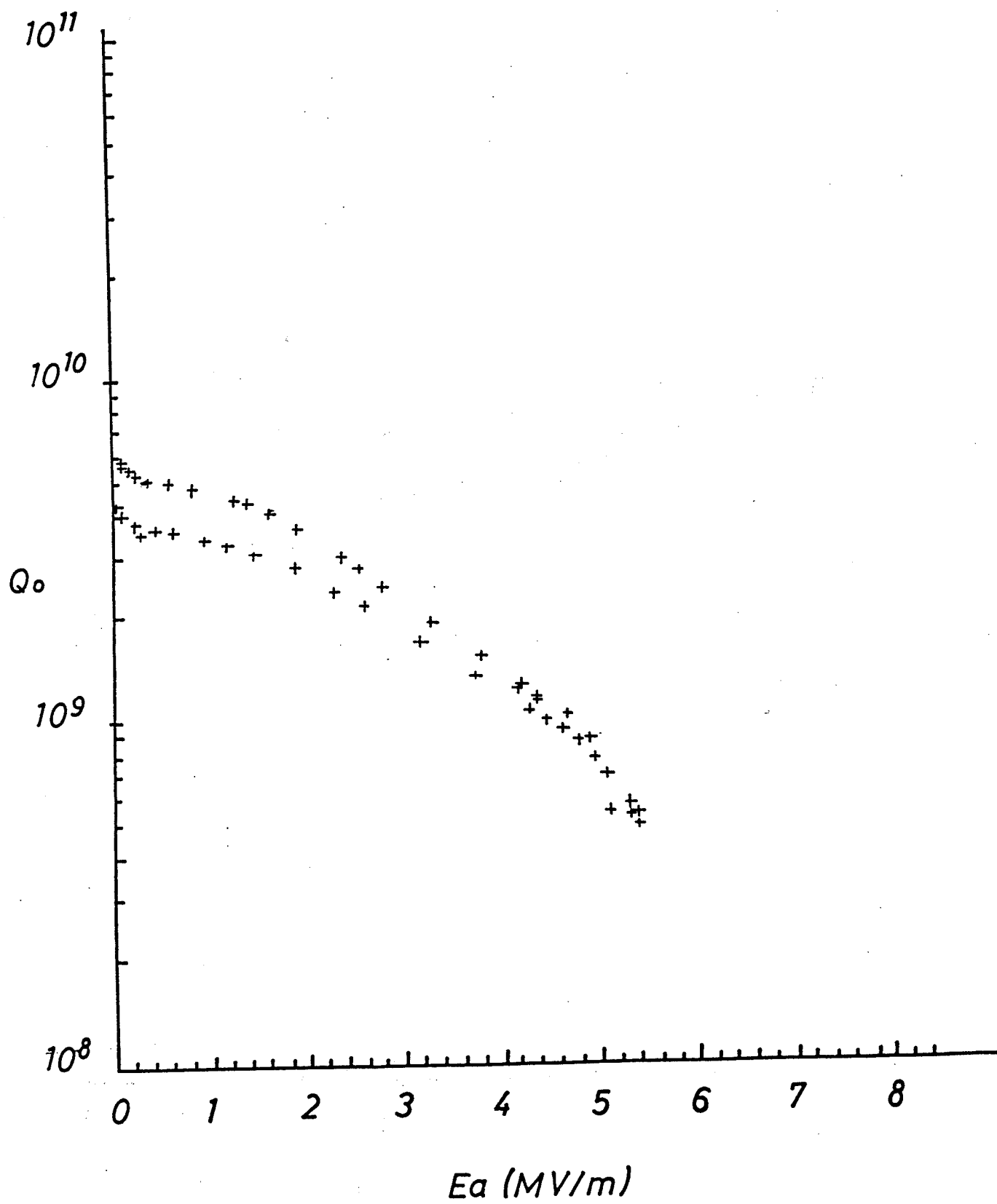


Fig. 4