SUPERCONDUCTING 500 MHz ACCELERATING COPPER CAVITIES SPUTTER-COATED WITH NIOBIUM FILMS

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SUMMARY

Thermal breakdown induced either by electron loading or by local defects of enhanced RF losses limits the accelerating field of super- conducting niobium cavities. Replacing niobium with a material of higher thermal conductivity would be highly desirable to increase the maximum field. Therefore, cavities made of OFHC copper were coated by D.C. bias sputtering with a thin niobium film (1.5 to 5 μm). Accelerating fields up to 8.6 MVm^{-1} were obtained without observing any field breakdown, the limitation being due to the available RF power. The Q values achieved at 4.2 K and low field were similar to those of niobium sheet cavities (i.e. $\sim 2 \times 10^9$), but a fast initial decrease of Q to about 10 was reproducibly experienced. Subsequent inspection of regions of enhanced RF losses revealed defects the origin of which is under study. The apparatus used for coating the cavities and the results obtained are presented and discussed.

presented by C.Benvenuti

1. INTRODUCTION

In the present plans for the Large Electron Positron Collider (LEP), the construction of which has begun at CERN, it is foreseen to use super- conducting accelerating cavities to upgrade the initial energy of 50 GeV.

Although accelerating cavities obtained by shaping bulk niobium sheet showed so far excellent behaviour [1,2] they suffer from thermal breakdown at high accelerating fields, induced either by electron loading or by enhanced heat dissipation in defects of the surface of the cavity.

Using copper as cavity material, which offers high thermal conductivity at low temperature, should greatly help to increase the stability of the cavity against breakdown by reducing the thermal gradient across cavity walls. The required superconductivity of the surface should then be provided by coating the copper with a thin layer of superconducting material, as for instance niobium. In addition, a coated copper cavity may result in a simpler and safer design of the cryostat by cooling the cavity for instance with pipes.

A feasibility study in this direction was started at CERN in 1980. This approach was also pursued in other laboratories where Nb deposition was performed by sputtering or evaporation onto a copper cover of a superconducting TE cavity at X-band with good results. For LEP application, however, it had to be shown that thin Nb films of an area of the order of a square metre could be reliably deposited inside a cavity of complex and almost closed geometry, and, furthermore, that these films could tolerate high accelerating fields without excessive power dissipation.

The present status of this study is reported here. Preliminary results have already been published [4].

2. EXPERIMENTAL APPROACH

Various Nb coating procedures were considered. These procedures are listed here, with, in brackets, the indication of the substrate temperature during coating and the energy of the impinging Nb atoms.

(a) Electrolysis from fused salts, as for instance K_2NbF_7 (700°C, < 0.1 eV).

(b) Chemical vapour deposition from $NbCl_5 + H_2$ or $NbBr_5 + H_2$ (700°C, < 0.1 eV).

(c) Evaporation (~ 300°C, ~ 0.2 eV).

(d) Sputtering (~ 100°C, ~ 10 eV).

(e) Ion implantation (< 100°C, > 100 eV).

Among these coating methods, sputtering was preferred for many reasons. The relatively low deposition temperature does not produce copper annealing and excessive crystal growth, while the energy of the impinging atoms is high enough to result in good adherence to the substrate. The feasibility of this method for Nb coatings is proven [5-8] provided that UHV technologies are rigorously applied. The uniformity of cavity coating may be guaranteed by properly shaping the cathode (see sect. 4). Sputtering may be used to deposit many other materials with a critical temperature higher than that of Nb, which therefore offer much higher potential Q values.

Among the various possible sputtering configurations, D.C. diode sputtering was initially chosen because it is inherently simple and suitable for coating the inside of cavities.

Owing to the large number of parameters involved in the coating process, the initial optimization of the Nb film was carried out on small samples in a planar cathode configuration. The characterization of samples was done by means of various techniques. The critical temperature T_c and the transition width were measured resistively and inductively. The structure of the film was studied by means of S.E.M. The chemical composition of the surface and across the film was obtained by Auger depth profiling, and the crystal characteristics by X-ray diffraction. Since the Nb film quality requirements for this application were unknown, the characteristics of bulk Nb were initially taken as quality reference. Only upon reaching values reasonably close to that of the bulk, coating was extended to cavities, initially of 3 GHz frequency (semi-spherical shape with about 9 cm diameter) and subsequently of 500 MHz (which is described in sect. 4). Quality control of cavity coatings was achieved by means of samples inserted at different locations inside cavities devoted to this purpose. To put in evidence the influence of the substrate, standard Nb sheet cavities of known performance were also coated with Nb films. The vacuum systems used were all-metal, bakeable at 200°C, pumped by sputter-ion pumps and able to reach the 10^{-10} Torr range before coating.

3. D.C. DIODE AND BIAS SPUTTERING

Samples initially obtained with D.C. diode sputtering never reached the T_c of bulk Nb (9.2 K). Values obtained using quartz substrates were higher (about 8.7 K) than on Cu (about 8.2 K). The film surface was rough and its structure columnar. Auger depth analysis revealed the presence of large amounts of C and O, and Cu when using Cu substrates. The films were brittle, of a dark grey aspect and exhibited good adhesion to the substrate.

It is known from literature [9-10], that both film purity and density are enhanced by bombardment with low energy ions (typically 60 to 80 eV) during film growth. This effect is usually obtained by applying to the substrate a negative bias with respect to the grounded walls of the vessel. Application of -80 V bias sputtering to the plane cathode configuration was straightforward and it gave a marked improvement. Values of T_c equal to or even higher than those of bulk Nb were obtained. The impurity content was greatly reduced and limited to the uppermost layers. No traces of Cu were observable in the film. The film surface was brilliant; its ductility and adhesion to the substrate good. The resistivity ratio (RRR) was increased by the bias approximately from 3 to 14.

Applying bias sputtering to cavities was less obvious. The problem was solved by introducing a reference electrode parallel to the cathode and biased positively with respect to the grounded cavity walls (see sect. 4). Samples obtained from inside test cavities by bias sputtering were very similar to those obtained in the plane cathode configuration.

Bias sputtering also resulted in a substantial improvement of the performance of the 3 GHz cavity which approached that of Nb sheet cavity [4]. Normal D.C. diode sputtering was therefore abandoned and only bias sputtering was used for coating the 500 MHz cavities.

4. CATHODE DESIGN AND COATING PARAMETERS

The geometry of the 500 MHz cavity and the adopted cathode structure are depicted in fig. 1. Rotating the cathode around the central axis of the cavity ensures uniformity of the film thickness on planes perpendicular to the axis of rotation. Thickness uniformity on cavity meridians may be insured by shaping the cathode as a "slice" of cavity provided that the cathode-to-wall distance is rigorously constant. Unfortunately, this requirement may not be fulfilled in practice because of the restricted access to the cavity as imposed by the entrance tubes. The restriction results in a cathode-to-wall distance of 3 cm in the end tubes and 8 cm at the equator. Consequently, the film thickness decreases progressively from the end tubes to the equator, where it is about a factor 4 lower.

In order to confine the sputtering discharge in front of the cathode, the latter is surrounded on the back and laterally, at 4 mm distance, by a shield. The favourable position of the shield makes it suitable for being used as reference electrode for bias sputtering. Best results were achieved so far with a bias voltage on the shield of +80 V with respect to cavity walls, which are at ground potential. During coating the cathode is kept at -1400 V and the argon pressure at 5×10^{-2} Torr. To speed up the film growth, three rotating cathodes are simultaneously used, as shown by fig. 2. With this cathode configuration and the chosen parameters, sputtering is carried out during 24 h, and results in a film thickness of 1.1 µm at the equator, 3.7 µm at the iris and 4.7 µm at the entrance tubes. This choice of film thickness is derived from measurements on 3 GHz cavities, but a proper thickness optimization for the 500 MHz cavities is still to be done.

5. PREPARATION OF THE CAVITIES

So far, three copper cavities have been built and coated. The two halves of each cavity are made out of OFHC copper sheet (3 mm thick, thermal conductivity 460 W m^{-1} K^{-1} at 4.2 K) by spinning and subsequently welded at the equator. The first cavity was TIG welded from the outside and large defects were visible on the inside of the weld. The two others were welded by electron beam with better results. However, since imperfections at the weld are always possible, tumbling with alumina chips was carried out for cavities 2 and 3, as a first step in the cavity preparation process. After tumbling the cavities are chemically cleaned, either with "nitric acid" (HNO_3 at 8%, H_2SO_4 at 42%, HCl at 0.2% in water, cleaning time about 2 min.) or in "persulphate" ($(NH_4)_2S_2O_8$, 30 g per litre of water, cleaning time about 5 min.).

Chemical cleaning is followed by rinsing with demineralized water and then with ethylic alcohol of analysis grade. Drying and the insertion of the cathodes are carried out in a dust-free room, then the cavity is baked at 200°C while pumping with a turbomolecular pumping station for 24 h. After cooling, a sputter-ion pump of 200 ls^{-1} pumping speed ensures an ultimate pressure of about 10^{-10} Torr. After coating, the cathodes are removed, the film visually inspected, the cavity rinsed again with demineralized water, dried in a dust-free room, installed in the cryostat, pumped and cooled.

To save on cavity manufacturing, after RF measurements the deposited Nb film is dissolved and the cavities are reused. After each coating the cathodes are disassembled and the shields chemically cleaned.

6. RESULTS

The characteristics of the Nb coating at different positions of the cavity are given in fig. 3. Only in the equatorial region is T_c = 9.2 K, while at the iris and at the entrance tubes T_c = 9 K. The average grain size is about 200 Å and RRR ~ 14. The higher T_c at the equator may be due to a more effective role of the bias at this place and/or to trapping of external impurities in the entrance tubes.

On the whole, 9 coatings were carried out (Table 1), one on a Nb cavity and the others on 3 copper cavities.

The results obtained for the coatings 2.2, 2.4 and 2.5 were good, as shown in fig. 4. In experiment 2.2 after reaching about 6 MV/m an irreversible deterioration occurred and the subsequent measurements resulted in consistently lower Q values, although a higher field was reached (8.6 MV/m). For all three experiments (fig. 4) the initial Q value was comparable to that obtained with Nb sheet cavities (about 2×10^9), but Q degraded rapidly by a factor 2, reaching about 10^9 at 3 MV/m. The maximum accelerating field always exceeded 7 MV/m without field breakdown. Higher values could not be reached because of the high power losses (about 200 W) and the limitation of the RF power supply. Temperature mapping was routinely applied and allowed to correlate "hot spots" on the map to defects on the cavity (fig. 5). However, the temperature signals were broader than for Nb sheet cavities (FWHM ≈ 200 mm). In spite of the good RF performance, in all three experiments a few defects of different type (blisters, protrusions, black spots, bare copper areas) and size (up to 1 mm diameter) were noticed. The majority of these defects were located on the lower flat part of the cavity, where a contamination consequent to chemical cleaning or particles falling from the upper part of the system are more likely to remain. These particles/contaminants may spoil the thermal contact between Nb and Cu (blisters) or decompose and result in a different film colour ("halo") and composition. In some cases they may fall off after coating and leave a hole in the niobium film. However, bare copper regions of about 0.3 mm size in the flat area did not produce any noticeable effect, both on the initial Q value and on temperature mapping. The magnetic field value at these places and at maximum accelerating field was about 20 mT.

The first copper cavity had a very bad equatorial weld and the coating was carried out mainly to finalize details of the cleaning and coating processes. After RF measurements, the cavity was cut for direct inspection of the film, which had good adherence and was since kept in normal air for about 1 year without any visible deterioration.

In two cases (2.3 and 3.1) the experiment was spoiled by an accidental air inlet during coating, consequent to a large leak which developed on the bellows of the rotating feedthrough. In another case (2.1) the film peeled-off, while for coating 3.2 the measured Q corresponding defect (blister 0.7 mm) value was very low (about 10^7) for reasons not understood.

7. CONCLUSIONS

The available results confirm, according to expectation, that a copper substrate greatly improves the behaviour of a superconducting cavity in terms of quench stability. This feature is particularly important when a string of cavities is powered by the same klystron, because in this case the lowest breakdown field would limit the accelerating field of all the cavities of the string. Niobium films of reasonable quality may be deposited on the inside of copper cavities of large dimensions, leading to high accelerating fields.

Defects of large size (up to about 1 mm diameter), although resulting in high local power dissipation, did not produce field breakdown. The large size of the tolerable defects renders possible to perform quality control on cavities by optical inspection.

Together with these encouraging facts, problems still exist. The reasons of the initial Q degradation (shown by fig. 4), of the sporadic film peel-off and of the poor Q value observed in experiment 3.2, are not yet well understood. Furthermore, the nature and the origin of the Nb film defects, as well as the importance of the copper surface conditions are not yet clear.

These facts must be understood and cured before a large scale production of cavities of this type may be envisaged for LEP. Furthermore, the feasibility of a coated multicell structure must be demonstrated. Less necessary but very attractive for LEP applicaton would be the development of a sputtering configuration more suitable for industrial production and the enhancement of Q by means of coatings with a critical temperature higher than that of niobium.

Acknowledgements

The use of copper as the cavity wall material was suggested to us by P. Bernard and E. Picasso. The authors are indebted to M. Wurgel for advice on cathode design, J. Genest for mechanical calculations and drawings, A. Boulmier and R. Kopp for surface cleaning, A. Grillot and A. Mathewson for Auger analysis, J. Adam and J-P. Bacher for S.E.M. analysis, M. Blin for T_c and G. Cavallari for cavity measurements. X-ray diffraction and RRR measurements were carried out by Ø. Fischer's group at the University of Geneva. Useful discussions with P. Bernard, H. Lengeler and H. Padamsee are greatfully acknowledged.

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TABLE 1

Summary of cavity preparation and performance

Cavity (weld)	Coating	Preparation of		$Q(10^9)$ at 4.2 K			E_{max} (MV/m)	Remarks	Relevant defects
		Cavity	Film	.05(MV/m)	$E_{max}/2$	E_{max}			
Nb/Nb 1 (EB)	1.1	CP	S	1.4	.8	.7	.9	Quench, Q-switch	Open blister at quench location
Nb/Cu 1 (TIG)	1.1	N	S	1.0	.2	.05	1.5	Q-switch	Blister on weld
Nb/Cu 2 (EB)	2.1	N	S	-	-	-	-	No RF measurement	Film peel off
	2.2	N	S	2.5	.7	.3	8.6	e^-, Q-degradation	Protrusion with halo, black spots
	2.3	-	A	1.5	.5	.1	2.4	Q-switch	Blister at equator
	2.4	N	S	1.7	.9	.5	7.9	Cu protrusion as e^- emitter	Black spots
	2.5	P	S	2.5	.9	.4	7.1	Q-switch	Blisters
Nb/CU 3 (EB)	3.1	D	A	.02	.01	.01	.8	Film feel off on large area	
	3.2	P	S	.01	.005	.005	.14	Poor performance not yet understood	

Legend: CP = Nb chemical polishing, N = "nitric acid" polishing, P = "persulphate" polishing, D = chemical degreasing, S = standard deposition parameters of the film, A = accidental air inlet during coating, Q-switch = step-like Q change consequent to localized phase transition.

FIGURE CAPTIONS

Fig. 1	Schematic view of the 500 MHz cavity with cathode.
Fig. 2	Cathode assembly - the three cathodes are inserted individually in the cavity and subsequently assembled together by means of the central rod.
Fig. 3	Structure of the Nb films obtained on samples placed at different locations inside the cavity.
Fig. 4	Variation of Q as a function of the accelerating field for the coatings 2.2 (+), 2.4 (□), 2.5 (o).
Fig. 5	T-map at 2.5 MV/m with "hot spot" and corresponding defect (blister Ø 0.7 mm).

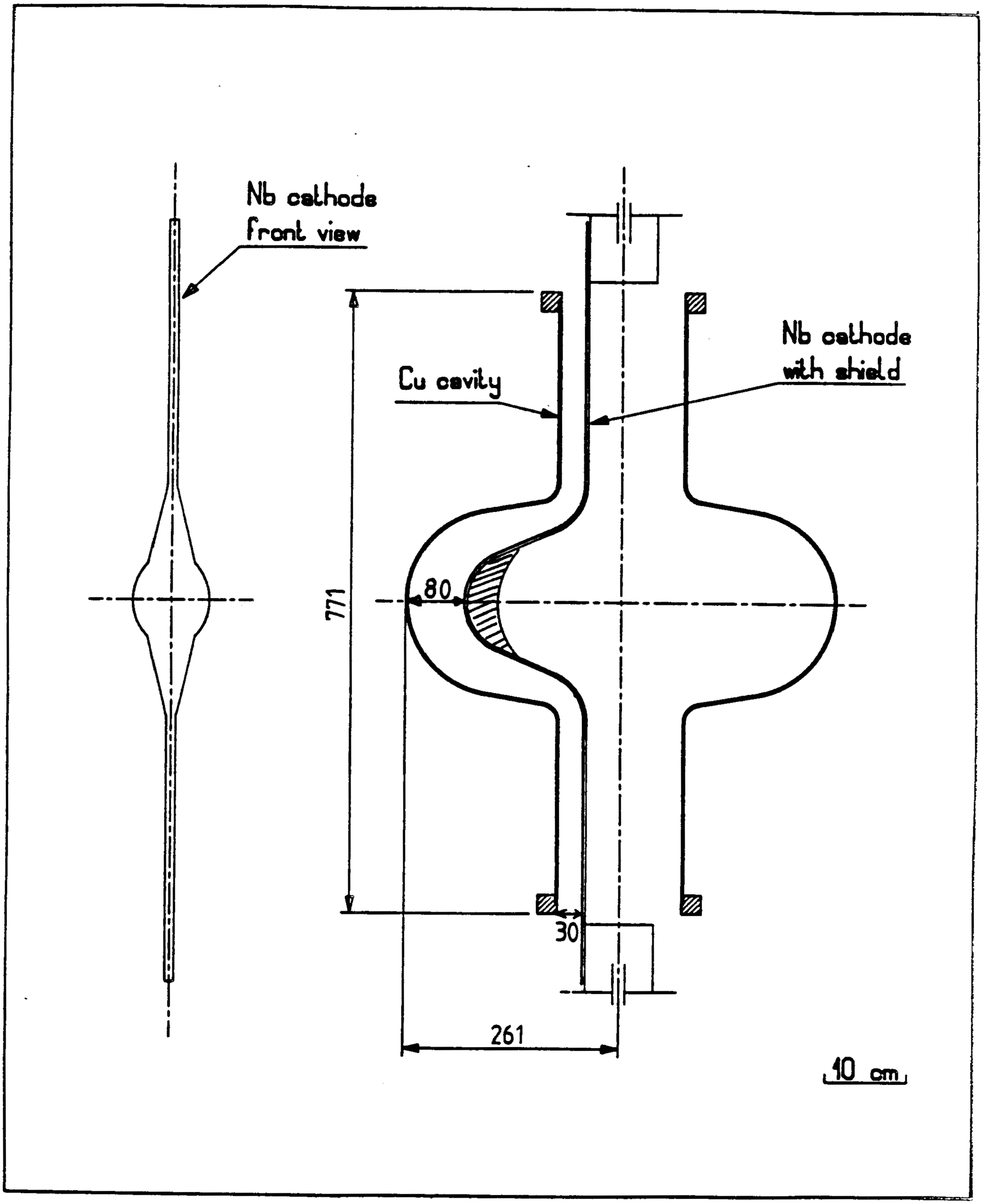


Fig 1 : Schematic view of the 500 MHz cavity with cathode

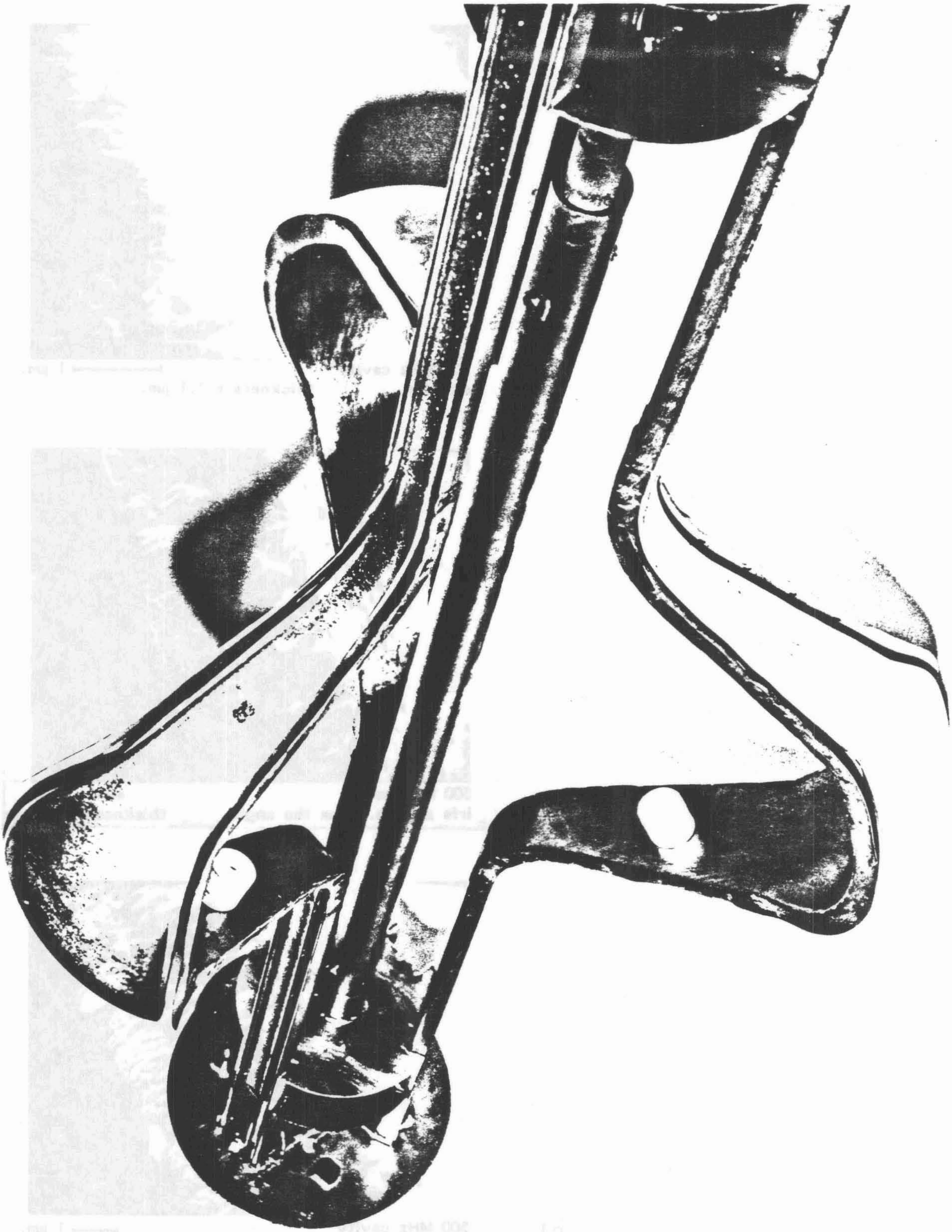
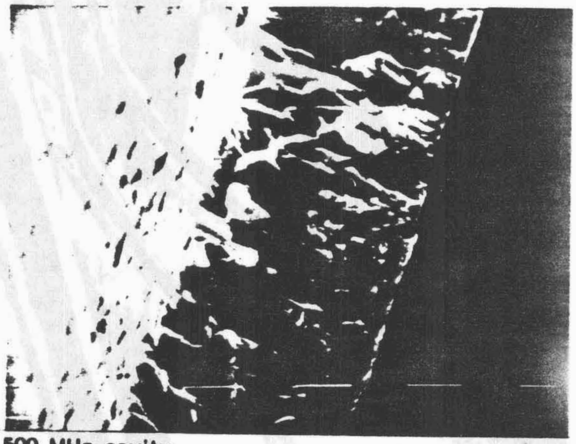
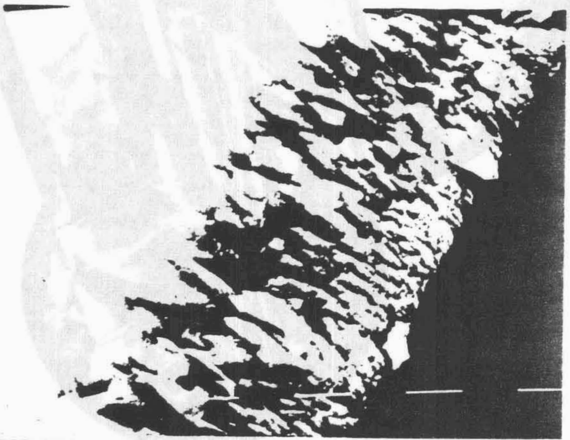


Fig 2 : Cathode assembly - the three cathodes are inserted individually in the cavity and subsequently assembled together by means of the central rod.

a) 500 MHz cavity equator thickness ≃ 1.1 µm.



b) 500 MHz cavity iris ≃ 6 cm. from the angle thickness ≃ 3.7



c) 500 MHz cavity tube thickness ≃ 4.7 µm.

Fig 3 : Structure of the Nb films obtained on samples placed at different locations inside the cavity : a) equator , b) flat part , c) tube.

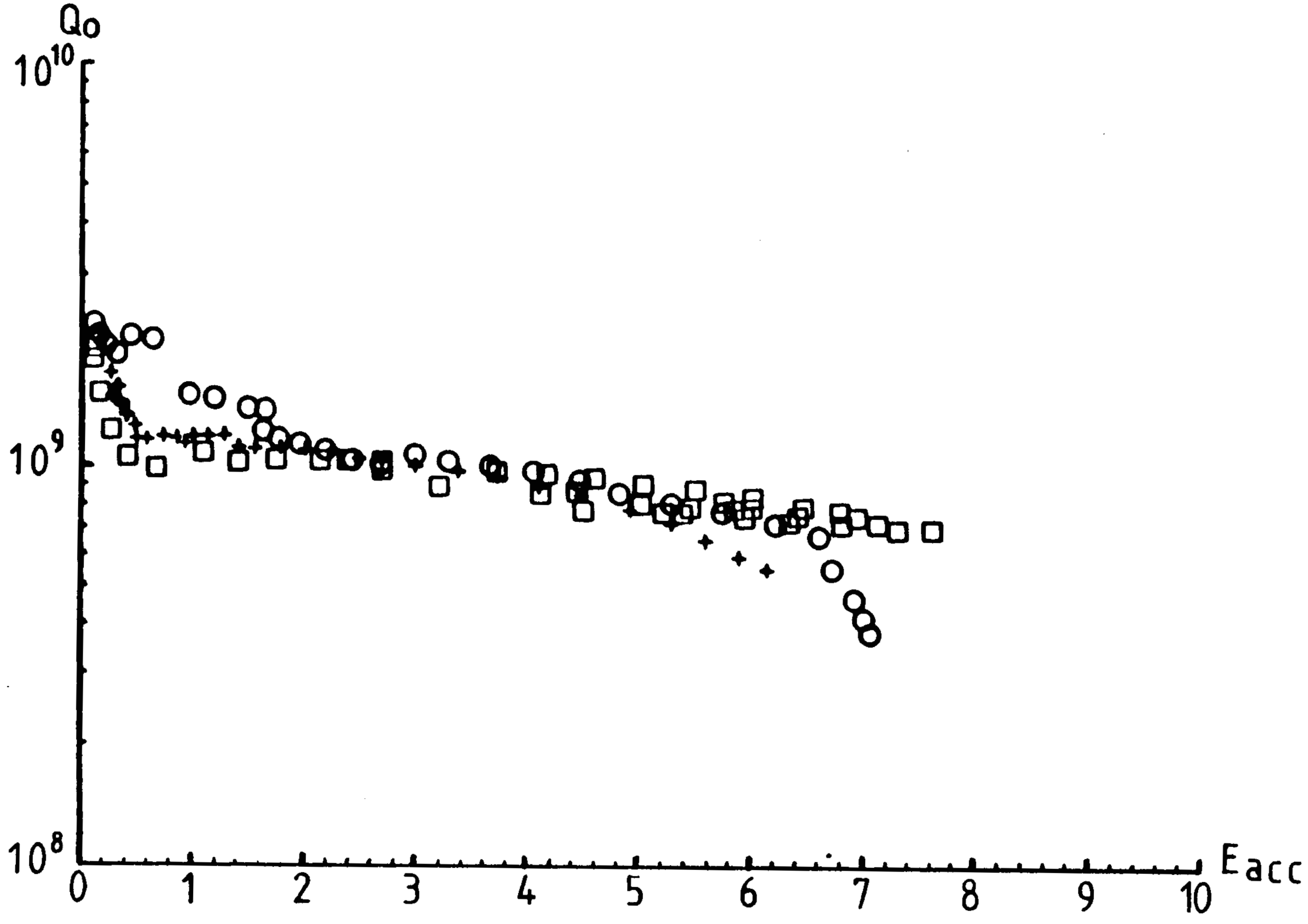


Fig 4 : Variation of Q as function of the accelerating field for the coatings 2.2 (+) , 2.4 (□) , 2.5 (o).

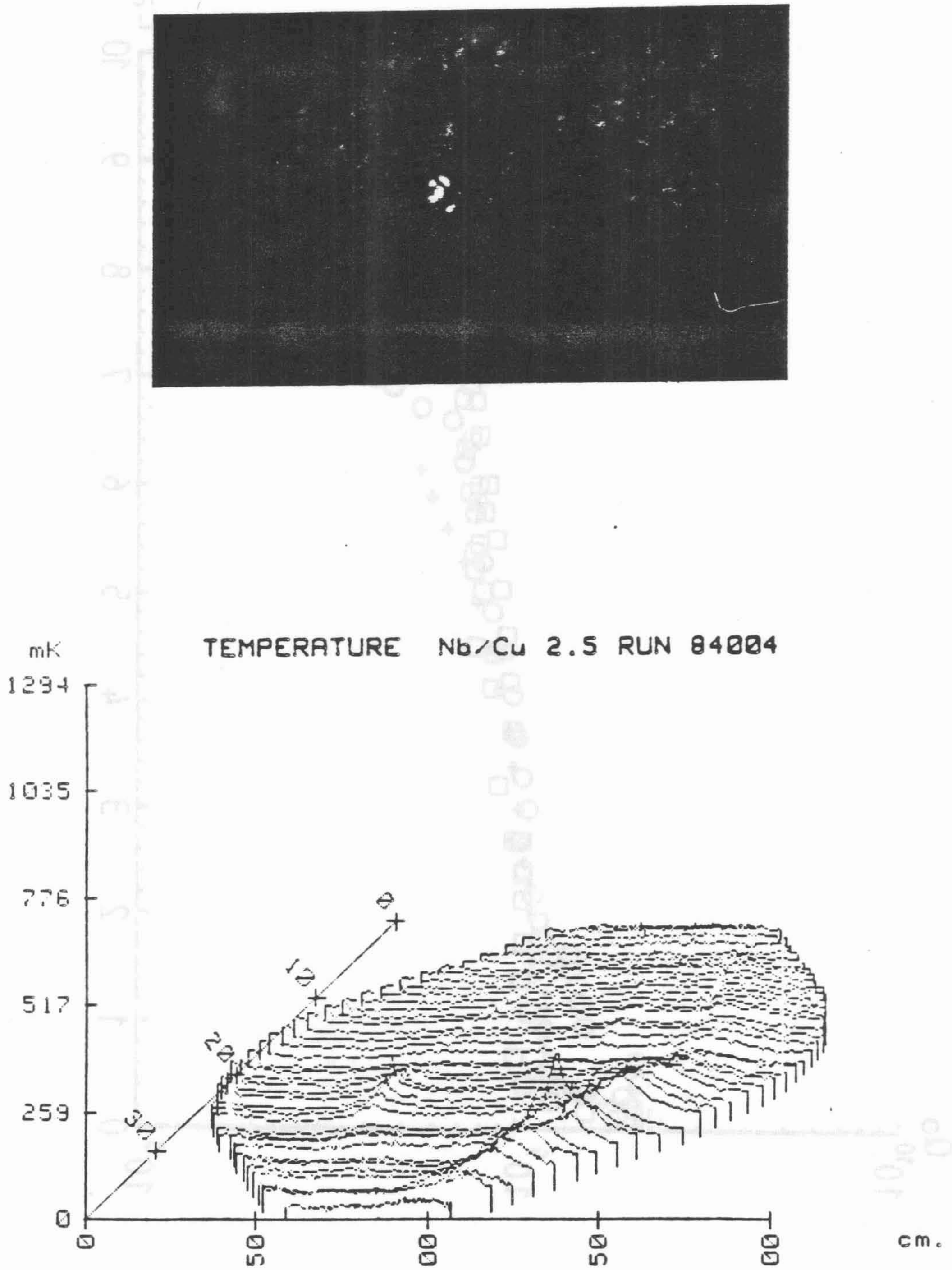


Fig 5 : T-map at 2.5 MV/m with "hot spot" and corresponding defect (blister ø 0.7 mm).