RECENT MEASUREMENTS AT SLAC ON SUPERCONDUCTING NIOBIUM X-BAND CAVITIES*

P. B. Wilson, Z. D. Farkas, H. A. Hogg, E. W. Hoyt
Stanford Linear Accelerator Center, Stanford University, Stanford, California 94305

Measurements at 8.6 GHz on TM mode superconducting niobium cavities have been carried out at SLAC in an attempt to establish definitive conditions for reproducibly attaining high peak electric and magnetic fields and high residual Q's. Four cavities, processed by techniques which insure the presence of an oxide layer on the niobium surface before final high temperature outgassing, have given peak magnetic fields exceeding 1000 G and corresponding peak electric fields in excess of 56 MV/m. From this and related experience it is speculated that, in order to achieve high peak fields, carbon present on the niobium surface must be removed through the formation of volatile compounds with oxygen or fluorine during high temperature processing. Data are also presented on the effect on rf properties of exposure at room temperature to various gases. Measurements on anodized cavities are briefly discussed.

Introduction

A large number of measurements have been made at SLAC on superconducting X-band cavities in an effort to gain an increased understanding of the factors which limit the residual Q and the peak rf electric and magnetic fields at superconducting surfaces. A frequency on the order of 10 GHz is convenient because of the small cavity size and consequent economy of fabrication and ease in processing and handling. Recent effort at SLAC has been concentrated on TM mode cavities, since the electric and magnetic fields in such cavities correspond closely to the fields to be expected in a single cell of standing wave superconducting accelerator structure.

Ideally, the conditions at the superconducting surface should be characterized by LEED, Auger and other appropriate techniques simultaneously with the measurement of rf properties. In principle this could be accomplished using a rod-shaped sample which, after being processed and subjected to surface characterization studies, is lowered into place within the same vacuum envelope to become the center conductor of a TE_{011} mode coaxial cavity for rf measurement. Because of the difficulties associated with the construction of an apparatus of this complexity, we have turned to the TM mode cavity as the simplest unit in which rf properties can be measured, and for which there is reasonable control over the environment seen by the cavity surface during the various stages of processing and handling. Nevertheless it is still difficult to carry out surface characterization studies on samples which are meaningfully correlated with conditions on the inaccessible cavity interior. On the basis of limited microprobe and Auger measurements made on cavity sections following processing and rf testing, relying also on intuition, trial and error and simple phenomenological models, we have tried to develop procedures for the reproducible construction of cavities with low residual loss and high limiting fields.

Fabrication and Processing Procedures

Most of the effort has been devoted to cavities which have been processed at high temperature and assembled in nitrogen without exposure to air. Only a few anodized cavities have been measured, partly because this technique is being vigorously investigated at other laboratories,[1,2] and partly because the rf properties of clean niobium surfaces are felt to be relevant even if the surface is later anodized. Likewise, electropolishing has not been actively pursued at SLAC, in part because of the difficulty of applying the process to the convoluted interior surface of accelerator structures, and in part because good success has been attained in X-band cavities by chemical polishing alone. Further, we have not been able to detect any systematic difference in results obtained using the refrigerated solution[3] (60% of 90% HNO_3 with 40% of 40% HF at about 0° C) developed at HEPL, as opposed to a buffered room temperature chemical polishing solution (1 part 85% H_3PO_4, 1 part 70% HNO_3, 1 part 48% HF).

The cavities reported upon here have been fabricated from reactor grade, electron beam melted niobium.[4] The most successful cavities have been electron beam welded after final machining using an inside corner joint as illustrated in Fig. 1. We have not had good success at SLAC using outside full penetration welds. After welding, the cavities are typically chemically polished to remove about 10 microns of niobium, high temperature outgassed at about 2000° C for around ten hours, chemically polished a second time, and then again high temperature fired. After the second outgassing cycle, the furnace is let down to pure dry nitrogen and the rf window and valve assembled to the cavity in a glove box attached to the furnace, hopefully without the slightest exposure to air. After assembly, the cavity is pumped without baking until the pressure is on the order of 10^{-10} torr, at which time the attached all-metal valve is closed and the tubulation shown in Fig. 1 is pinched off. Good results have been obtained using both an induction furnace and a resistively heated furnace. The most important variable, in fact, seems to be the purity of the nitrogen gas used in the let down and assembly procedure. No correlation between rf results and firing temperature, over the range 1900-2100° C, has been observed.

Cavities with several different geometries have been tested, although most of the measurements reported upon here have been made with cavities as shown in Fig. 1. For this cavity geometry, the product of unloaded Q and surface resistance is 213 and the ratio of peak electric and magnetic fields is .056 (MV/m)/G.

Experimental Results : Gas Exposure Tests

The results of exposure of clean niobium cavities to various gases at room temperature will be briefly reported here. Additional measurements are planned, and a more complete account will be published later. In these experiments, the cavities, after they have been fired and tested as usual, are let down to the gas in question at a pressure of about 1/4 atmosphere for about 1 hour. The gas is then pumped from the cavity until the residual pressure is below 10^{-10} torr (without baking), the valve attached to the cavity is closed, and the cavity is retested. The results to date are summarized in Table I. In this table, each series gives the results of successive exposures for a given cavity. The Q jumping factor is the ratio of Q at low power (measured at about 1.5° K) after the application of high rf power, to the low power Q before the initial application of high power.

It is seen that the effect of various common gases on the rf properties of the surface can be profound, even though the metallurgy of the surface layer within the penetration depth must remain essentially unchanged. The results point out the importance of cleanliness in cavity assembly after high temperature firing, the importance of the kind and amount of residual gases remaining in the cavity, and the desirability of isolating a cavity from the room temperature vacuum system by means of a low temperature rf window. Recent results[5] at HEPL show that magnetic field breakdown generally occurs at that location in the cavity which is the first to cool and to form a site for trapping residual gases. This again points out the importance of cleanliness and residual gas composition. We can conclude that it is highly desirable to have an appendage attached to the cavity which is cooled during

*Supported by the U. S. Atomic Energy Commission.

TABLE I

SUMMARY OF GAS EXPOSURE TESTS

Gas	Residual Q Before	Residual Q After	Breakdown Field Before	Breakdown Field After	Q Jumping Factor
series 1					
H_2	7×10^9	6×10^9	640 G	696 G	1
Dry O_2	6×10^9	4×10^9	696	660	1
Wet O_2 (trace CO_2)	4×10^9	1.6×10^9	660	361	3
Air	1.6×10^9	3×10^8	361	343	3
series 2					
CO_2	$>5 \times 10^{10}$	1×10^9	1073	208	2-1/2
series 3					
CO	7×10^9	8×10^9	1042	684	20
CO_2	8×10^9	1×10^9	684	331	2-1/2
series 4					
CH_4	6×10^9	1.3×10^9	1000	390	1
O_3	1.3×10^9	7×10^8	390	255	2-1/2

transfer before the main cavity, and which can act as a cryopump to trap out the residual gas content well away from surfaces seen by the rf. This appears to be the situation for the cavity assembly as shown in Fig. 1.

In recent measurements at Siemens,[4] increased magnetic breakdown fields have been obtained by introducing helium gas at a low residual pressure (below the glow discharge region) during the application of rf power. Presumably, continuous helium ion bombardment "scrubs" the surface and moves any adsorbed residual gas into regions of the cavity where the rf fields are low.

Experimental Results : The Role of Surface Carbon

At one point in the cavity measurement program at SLAC, the results were extremely discouraging. Every effort was made to improve cleanliness at each step of cavity processing and assembly, but in spite of all such efforts cavities continued to be limited by breakdown fields in the 200-300 G range. There seemed, in fact, to be a negative correlation between cleanliness and breakdown field. In analyzing the steps that led to the production of the few relatively good cavities measured up to that time, it was found that in each case the cavity had been exposed to air for a substantial length of time after final chemical polishing. Presumably, a layer of oxide played an important role during final high temperature processing. It seemed a reasonable assumption that oxygen is necessary to remove carbon at or near the surface during firing.

The presence of carbon on a fired niobium cavity surface has been seen in electron microprobe studies[6] at SLAC. The carbon tends to occur as granules with dimensions on the order of 10 microns, frequently located at crystal boundaries. Similar carbon inclusions have been seen[7] at Cornell on samples studied with a scanning electron microscope. These carbon clumps can presumably become regions of enhanced local heating leading to magnetic field breakdown at fields well below the critical field expected for a homogeneous surface.

Three techniques have been used at SLAC to facilitate surface carbon removal. The simplest is a double final firing in which the furnace is let down to oxygen for several hours between firings. A second technique is to anodize the cavity before final firing. Finally, two cavities have been processed for an hour or so in the induction furnace in an oxygen atmosphere at a pressure of about 7×10^{-6} torr and a temperature of 1900° C. After this step, the oxygen was then pumped out and the temperature raised to 2050° C for several hours to outgas dissolved oxygen from the metal. In these two tests the residual Q's were 1.3×10^9 and 6×10^8, and the respective rf magnetic breakdown fields were 830 G and 613 G. The high power behavior of one of the cavities (XTM 12-3) is shown in Fig. 2. The second cavity, with the lower breakdown field and residual Q, had a very unusual, sharp resonance peak in the curve of surface resistance vs. field level. At a field of 12 G the surface resistance increased abruptly by about a factor of two, falling back more gradually as the field was further increased. The width of the peak at half maximum was about 5 G. The resonance, and the relatively low residual Q, is probably associated with a high concentration of solute oxygen remaining within a penetration depth of the surface. The results of Giordano et al.,[8] indicate that a high solute oxygen concentration has an adverse effect on both residual Q and breakdown field.

The interpretation of the role of oxygen as an agent to expedite the removal of surface carbon is further clarified by observations made using a residual gas analyzer during high temperature outgassing. Heavy evolution of CO_2 and CO is seen during the initial stage of firing. These peaks diminish in intensity as firing progresses. In fact, the reduction of CO partial pressure to below a defined level has been the major criterion which we have used in our work to determine that the firing time has been adequate.

As the temperature is raised at the start of a firing cycle, a strong burst of carbon monofluoride is typically seen at around 1000° C, giving a further indication of carbon removal during high temperature processing. Higher fluorides (CF_2, CF_3 and CF_4) have also been observed. The presence of the fluorine is probably due to residual niobium fluoride complexes on the surface not removed by solvents following chemical polishing. One can therefore speculate that the production of a niobium fluoride layer with subsequent firing could also be an effective way to achieve the removal of carbon on and near the surface.

If the preceeding model has some validity, it would indicate that an overly-long firing period without the presence of adequate oxygen or fluorine could be detrimental. During firing, dissolved carbon in the bulk of the metal will be continuously diffusing toward crystal boundaries. If the crystals are in fact growing, the moving boundaries can act as carbon sweeps. The carbon might be expected to diffuse along the boundaries toward the surface, or to gather into discrete clumps at defects or boundary confluences. Subsequent chemical polishing could expose such clumps at interior grain boundaries.

Experimental Results : Anodizing

Several tests have been made on anodized X-band cavities, with mixed results. A TE_{011} mode cavity was anodized at 50 V and stripped six successive times in an effort to achieve surface smoothing. It was then anodized at 120 V in a 20% NH_4OH solution. A residual Q of 1.7×10^{10} ($R_s = 4 \times 10^{-8}$ ohms) and a magnetic breakdown field of 630 G were measured. After storage under vacuum for one month, the cavity was exposed to air for 4 hours and then remeasured. The residual Q had degraded to 1.0×10^9, and the breakdown field to 580 G. Two TM mode X-band cavities were anodized in a 25% NH_4OH solution at 110 V. In both cases the residual Q was 3×10^8. The breakdown fields were 275 and 580 G. Dielectric losses in the relatively thick oxide layer can explain the low residual Q.

High Field Results

Data on the processing techniques together with rf results for five high field cavities are given in Table II. It is seen that all three techniques for processing with oxygen have

TABLE II

PROCESSING TREATMENT AND RESULTS FOR HIGH FIELD CAVITIES

Cavity and Test	Treatment	Residual Q (Low Power)	Breakdown Field H_b	Q at H_b	Radiation at H_b
XTM 6-14	Anodized at 115 V, fired	9×10^9	1003 Oe	4.3×10^9	0 mR/hr
XTM 6-15	After one month on shelf	6×10^9	1000	3.5×10^9	0
XTM 10-5	Anodized and stripped, fired, exposed to O_2, fired	2.5×10^9	1035	2.0×10^9	0
XTM 10-7	Anodized at 110 V, fired	7×10^9	1042	1.2×10^9	210
XTM 11-1	Prefired, final machined, welded etched, fired, etched, exposed to O_2, fired	$>5 \times 10^{10}$	1060	1.6×10^{10}	0
XTM 12-3	Fired in O_2 (7×10^{-6} torr) at 1900° for 2 hrs, outgassed at 2050° C for 5 hrs.	1.3×10^9	830	1.1×10^9	30

given good results. The behavior of the surface resistance of these cavities as a function of field level is given in Fig. 2. In general, the surface resistance is seen to decrease at moderate field levels, rising again at the highest fields. This general behavior is similar to that observed by Kneisel et al.[9] for anodized TM_{010} mode cavities at S-band. A rise at high field levels in the effective surface resistance would normally be expected as the result of simple thermal heating, although in two of the cases shown in Fig. 2 a considerably steeper rise was observed, associated with field emission loading. For cavity XTM 10-7, a Fowler-Nordheim plot was made using the observed radiation level outside the dewar, following a relation due to Schopper et al.,[10] which takes into account absorbtion by the cavity and dewar walls. The dose rate divided by E_m^6 is plotted on a semi-log scale as a function of $1/E_m$, where E_m is the maximum electric field in the cavity in MV/m. The enhancement factor is equal to -2.2×10^4 divided by the slope of the resulting straight (in theory) line. For XTM 10-7 an enhancement factor of 80 was calculated in this manner.

The variation in effective surface resistance as a function of field level for one of the cavities (XTM 6-15) is shown in Fig. 3 for several different temperatures. The behavior is unusual in that the breakdown field clearly does not vary as $\left[1 - (T/T_c)^2\right]$. During initial processing, this cavity first showed classical magnetic field breakdown at the 700 G field level. After a period of processing the breakdown field suddenly increased to the 980 G level shown for 1.77° K. A relatively slight increase in temperature to 2.06°K caused the breakdown field to drop to 686 G. We can speculate that a small region on the cavity surface went normal at about 700 G. At the lower temperature thermal conduction was just adequate to remove the heat generated at the defect so that thermal runaway did not occur at that field level. An increase in temperature to 2.06° K changed the thermal balance sufficiently to allow thermal runaway, causing the breakdown field to drop abruptly back to the 700 G level.

Concluding Remarks

During the period reported upon here, about a dozen tests were made in an attempt to obtain good cavities using one of the three methods of processing with oxygen, and in which there was not at the same time an obvious source of known degradation such as an inadvertant exposure to air. The breakdown fields for these tests ranged from 313 G to 1060 G, and the residual Q's from 0.5 to 50×10^9 (median values for the breakdown field and residual Q were 630 G and 3×10^9). Thus it appears that the presence of sufficient oxygen during high temperature processing may be a necessary condition for obtaining good rf properties, but it is obviously not a sufficient condition. Particularly toward the end of the measurement period, contamination of the nitrogen used for let-down and assembly was suspected as a factor in a series of poor results. The gas exposure tests have led to a better understanding of these results, and have pointed the way to the necessary steps for improvement.

Acknowledgements

The authors wish to thank Hank Deruyter, Howard L. Martin, and Robert W. Silvers for their help in processing and assembling the cavities and in making the microwave measurements.

References

1. H. Diepers, H. Martens and O. Schmidt, Proceedings of the 1973 Particle Accelerator Conference. To be published in IEEE Trans. Nucl. Sci. (1973).

2. P. Kneisel, O. Stoltz, and J. Halbritter, Proceedings of the 1973 Particle Accelerator Conference. To be published in IEEE Trans. Nucl. Sci. (1973).

3. J. P. Turneaure, IEEE Trans. Nucl. Sci. NS-18, No. 3 166 (1971).

4. Supplied by Wah-Chang Albany Corporation.

5. C. Lyneis, M. McAshan and Nguyen Tuong Viet, Proceedings of the 1972 Proton Linear Accelerator Conference (Report LA-5115, Los Alamos Scientific Laboratory, 1972); p. 98.

6. Carried out with the cooperation of the Center for Materials Research, Stanford University.

7. R. Sundelin, Laboratory of Nuclear Studies, Cornell University, private communication.

8. S. Giordano, H. Hahn, H. Halama and C. Varmazis, "Influence of solute oxygen and nitrogen on superconducting niobium cavities." To be published in J. Appl. Phys. (1973).

9. P. Kneisel, O. Stoltz and J. Halbritter, Proceedings of the 1972 Applied Superconductivity Conference (IEEE Pub. 72CH0682-5-TABSC, 1972); p. 657.

10. H. Schopper, H. Strube and L. Szecsi, "An investigation of field emission in superconducting rf cavities by dose rate measurements," (external report 3/68-6, Karlsruhe, 1968).

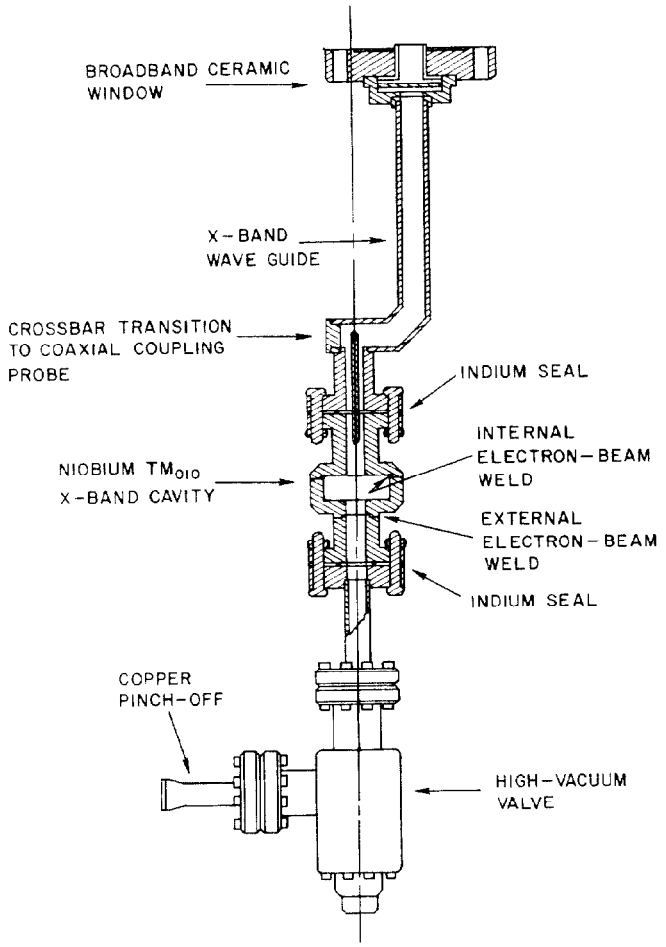

FIG. 1--TM mode X-band cavity assembly.

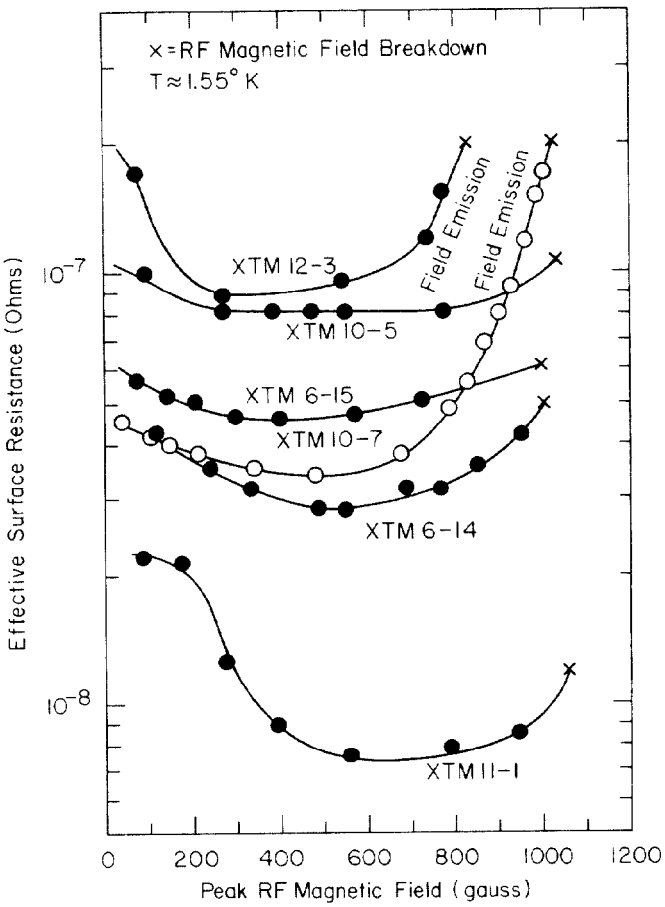

FIG. 2--Effective surface resistance as a function of peak rf magnetic field for several high field cavities.

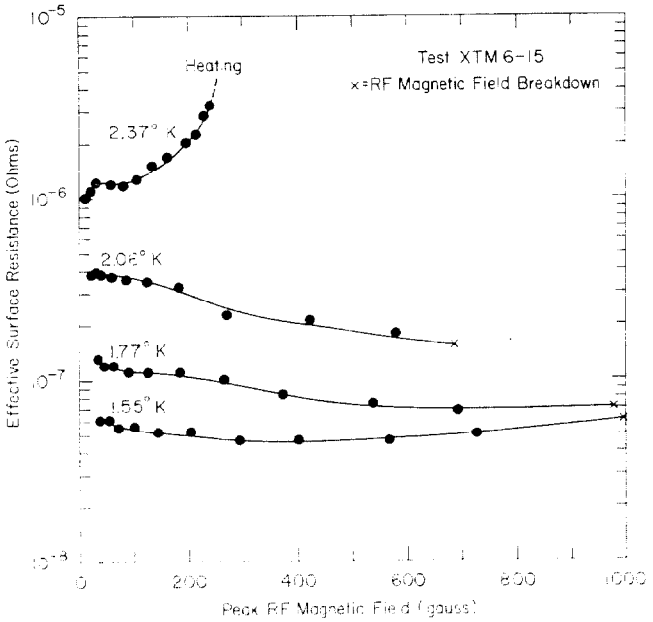

FIG. 3--Effective surface resistance of XTM 6-15 as a function of peak rf magnetic field at several temperatures.