THE EFFECTS OF EXPOSING WELL PROCESSED RF SURFACES OF SUPERCONDUCTING CAVITIES TO VARIOUS MEDIA*

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Abstract

Since we started heat treatment (HT) of superconducting cavities above 1100℃ in an ultra-high vacuum furnace, followed by high power He processing, fifteen 1-cell 1.5-GHz accelerating cavities have been tested. On the average, 80% higher fields (40 MV/m) were reached with HT than with standard chemical treatment; the highest surface electric field was 53 MV/m (equivalent to an accelerating gradient of about 26 MV/m). However, field emission (FE) is still the most serious obstacle to reaching higher fields. One important aspect of our program is to determine at what stage in the surface preparation emitters are introduced onto the cavity surface. Our strategy has been to intentionally expose well processed RF cavity surfaces to various media used in cavity surface preparation. We find that chemical treatment seriously degrades well processed HT cavity surfaces by increasing FE; such degraded surfaces can not be recovered by He processing. Tests with clean water exposure show similar degradation, but these emission sources can usually be He processed, recovering the good baseline cavity performance. Exposure to dust-free (class-100) air does not introduce an abundance of new emitters. Similarly we have eliminated the high purity rinsing methanol as an abundant source of emitters. In these studies, a thermometer-based diagnostic system was used to monitor the power deposited by impacting electrons through the resulting temperature rise. Representative temperature maps are presented along with Q vs E_{pk} RF behavior. The equivalent emission enhancement factor and emissive area of the dominant emitters are also presented.

1. INTRODUCTION

Large scale application of superconducting radio frequency(SRF) cavities to electron accelerators is in progress at many laboratories around the world. With the solution of a series of problems, such as thermal breakdown and multipacting, field emission is now recognized to be the most serious obstacle to reaching accelerating fields E_{acc} higher than l0 MV/m (equivalent to a maximum surface electric field E_{pk} of about 20 MV/m). The proposed E_{acc} of 30-40 MV/m [1] for the next generation of TeV electron accelerators stresses the importance of reaching the highest possible fields. The theoretical limitation is as high as E_{acc}= 50 MV/m, set by the critical magnetic field at which superconductivity is expected to break down. A program to systematically increase E_{acc} has been in progress at Cornell University for many years using 1-cell 1.5-GHz superconducting cavities.

In order to reduce FE, we have been heat treating (HT) superconducting cavities above 1100℃ in an ultra-high vacuum furnace routinely since 1987. The results are very encouraging [2,3,4]. The main problem has been the absorption in the Nb cavity walls of residual O from the furnace vacuum. This reduces the wall RRR, indicating a lower purity that decreases the Nb thermal conductivity, tending to cause thermal breakdown. By late 1988,a technique of enclosing the cavity in a Ti box during HT [4] allowed us to raise HT temperatures to 1350℃ and heating times to eight hours without reducing the RRR.With high power He processing in addition, altogether fifteen 1-cell 1.5-GHz accelerating cavities have been tested. On the average, the accelerating fields E_{acc} reached with HT (20 MV/m, equivalent to a maximum surface electric field E_{pk}=40 MV/m) were 80% higher than with standard chemical treatment (CT); our highest Eacc was 27 MV/m (E_{pk}=54 MV/m). The details of these accomplishments have been discussed in our previous papers [2,3,4].

Although significant progress has been made through the above efforts, FE continues to block the way to higher accelerating fields. In order to reach higher peak fields repeatedly and reliably, we need to understand more fully the origin of the emitter sources in these cavities--whether they occur naturally in the bulk Nb, whether they exist in latent form in the Nb bulk and are activated by external agents, or whether they are externally introduced onto the cavity surfaces during cavity surface preparation. Such knowledge would make treatment and processing of cavities more effective and productive. Therefore, while we continue to push hard toward

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higher fields through heat treatment and processing, we have also begun an effort to determine how emitters are introduced onto or activated from the cavity surfaces.

Some of our earlier studies have already shown that condensed gases play a significant role in activating field emission [5]. For instance, on introduction of O_2 into a cold cavity the dissipated power increases and the Q of the cavity decreases, both by an order of magnitude. It is possible for a resonant tunneling process through a thin overlying insulating layer on a field emitting pure metal surface to increase emission. In view of the role of condensed gases in aggravating emission it will be important to explore the possible benefits of cooling cavities in a better vacuum, as well as to attempt better outgassing of the RF surface before cooling down.

In this paper, we report initial results of a second program aimed at determining the stage in the cavity surface preparation at which the emission sites themselves are introduced onto the cavity surface. Our strategy was to intentionally expose well processed and characterized RF cavity "reference" surfaces to various media used in cavity surface preparation. After exposure tests we carefully studied both the new emitters which were introduced on the cavity surface and the resulting RF performance degradation caused by these emitters. The different media involved in our cavity preparation are chemical etching agents, clean water, high purity rinsing methanol, and dust-free (class- 100) air. In presenting our results, we make two kinds of comparisons for each cavity before and after treatment:

1. Overall RF performance, Q vs E_{pk}.

2. Cavity wall temperature maps as a function of E_{pk}. These maps are taken with a thermometer-based diagnostic system [6]; they show the power deposited by impacting FE electrons.

2. REFERENCE CAVITY PREPARATION

The ideal reference state of a cavity intended for exposure tests is one that fulfills two requirements. First, the cavity should be capable of reaching the highest field levels of interest. The effects of exposure to a particular medium may be striking only at relatively high fields and could be missed by a test on a cavity limited (e.g., by defects) to lower fields. Second, the cavity should give no significant field emission throughout its range of fields. Any emission sites and non-ideal overall RF performance observed after subsequent exposure could then be cleanly attributed to the exposure medium.

Unfortunately, there is presently no known way to produce such an ideal reference state. In this study, therefore, each cavity was prepared in the best possible reference state in the following manner: First, a heat treatment is carried out, since an HT cavity can reach a higher field in general than a standard CT cavity [4]. Second, the cavity is He processed, as this increases the maximum attainable fields (in most cases giving E_{pk} above 40 MV/m). Normally the cavity is also room temperature cycled and retested to ensure that its good performance was retained. (Room temperature cycling here means a special procedure in which a cold-tested cavity is warmed to room temperature and then recooled to superfluid liquid-He temperature without mechanical disturbance.)

The inner surface of the chosen cavity in its reference state is then carefully exposed to one of the media used in surface preparation. Special care is taken during exposure to avoid unintended contact of thc cavity's inner surface with other media. (Inevitably, any exposure test also involves warming to room temperature and exposure to clean air. We show in sections 6 and 7 below that these processes have no significant effects on a cavity.)

3. EXPOSURE TO CHEMICAL ETCHING AGENTS

As noted above, we have found that heat treatment of an initially chemically treated superconducting cavity will improve its RF performance by reducing field emission. The inverse question is whether the performance of a fired cavity will be degraded by a subsequent chemical treatment--i.e., whether chemical etching agents produce emission sources on cavity surfaces. To answer this, we chose two well He- processed HT cavities as references and then subjected each to our standard etching process. This process uses an etching solution of equal volumes of HNO_3, HF and H_3PO_4, and consists of the following: .

1. Ultrasonically (US) degreasing cavity in acetone for 15 minutes.
2. Storage in clean de-ionized (DI) water.
3. Exposure to etching solution for two minutes.

4. Rinsing in clean DI water three times, using fresh water each time.
5. US cleaning in 5% H_2O_2 for one hour.
6. US cleaning in DI water at 40-50℃ for one hour.
7. Rinsing with high purity methanol in clean room.
8. Drying cavity horizontally on clean bench.
9. Placing cavity in clean bag with clean caps covering both ends of cavity beam tube.
10. Removing cavity to cryostat and installing under class 10-100 dust-free conditions, provided by a clean air laminar blower and a home-made tent covering the assembly area.

3.1. Overall RF Performance

The reference condition of the cavity in Fig. l(a) resulted from Ti box firing at 1350℃ for 4 hours. As shown by the circle points, its highest E_{pk} was 53 MV/rn with $Q = 1.5\times10^9$. The flat region of Q vs E_{pk} extended up tn about 40 MV/m at a Q of about 4×10^9. After exposure to the chemical etching agents the cavity performance was seriously degraded. The highest E_{pk} was only about 31 MV/m with a Q of 3×10^8 before He processing. After He processing the highest E_{pk} was 37 MV/m with a Q of 4×10^8, and the flat region only extended to about 20 MV/m. Clearly FE starts at much lower fields and is much stronger after exposure.

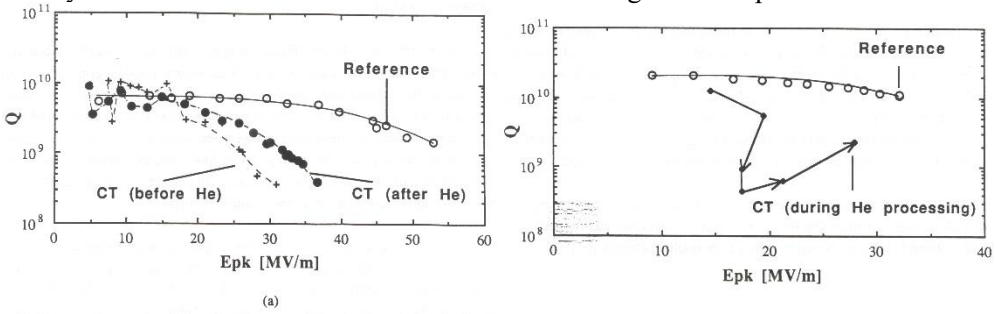

Fig.1. Overall RF performance of two cavities before(reference)and after exposure to chemical etching agents(CT).The lines connecting data points show the measurement and RF-processing sequence.

Figure l(b) shows the results of the other chemical exposure test. The cavity in the reference condition reached 32 MV/m with a Q of about 1×10^{10}. After exposure the Q was seriously reduced and the highest field attainable was only 28 MV/m with a Q of abot 2×10^9.

3.2. Temperature Maps

Our thermometer-based diagnostic system [6] has 36 boards placed at 10° intervals around the cavity, each carrying 19 carbon resistance thermometers. Each resistor senses the local temperature rise of the cavity wall due to energy deposited by impacting FE electrons. The magnitude of the temperature rise is a function of emitter strength, and the number of temperature changed areas indicates the density of emitters. A computerized data acquisition system scans all 684 resistors, allowing us to record a temperature map of the entire surface of a cavity in only 15 {?} seconds. This in turn allows us to study dynamic processes and transient FE states.

Figure 2(a) shows a comparison of the temperature maps before and after CT for the same cavity represented in Fig. 1 (a). The four maps in the left column were taken in the reference srarted at fields E_{pk} from 19 to 49 MV/m. The maps in the center column represent the cavity exposed to chemical agents but before He processing. The maps in the right column are from the exposed cavity after He processing. The maps in each row were taken at about the same E_{pk}.

From Fig. 2(a) we can conclude the following: (1) In the reference condition there is no significant emission until E_{pk}=49 MV/m. (2) After chemical exposure but before He processing, significant emission started at E_{pk}=19 MV/m. Thc emitters are strong and local temperature rises of about 1500 mk are seen. The cavity was quenched by the large FE loading. (3) After subsequent He processing, emission was reduced greatly, but emission becomes very strong again at E_{pk}=37 MV/m. Further properties of the emitters in the three conditions of this cavity are discussed below in section 8.

Figure 2(b) shows similar results for the cavity of Fig. l(b). Again there is a very strong emitter after chemical exposure. Although the emission was reduced with RF processing, enough remained to stop the cavity from

teaching a higher field.

4. EXPOSURE TO CLEAN DE-IONIZED WATER

Two well He-processed HT cavities were chosen as reference states to expose to clean DI water (p=18 MΩ -cm). Special care was taken to prevent the inner cavity surfaces from contamination by agents other than clean DI water. The exposure procedure is as follows:

1. Dismount thermometer boards.
2. Wipe off remaining grease.
3. US clean outer surface of cavity for 30 min, maintaining vacuum in cavity on cryostat, using an acetone bath.
4. Repeat with Freon bath.
5. Repeat with methanol bath.
6. Disconnect cavity from cryostat; cap, bag, and carry to clean room.
7. Rinse cavity inside only with clean DI water and dry in clean hood in horizontal position.
8. Cap, bag, and carry cavity to cryostat; install as before under dust-free conditions.

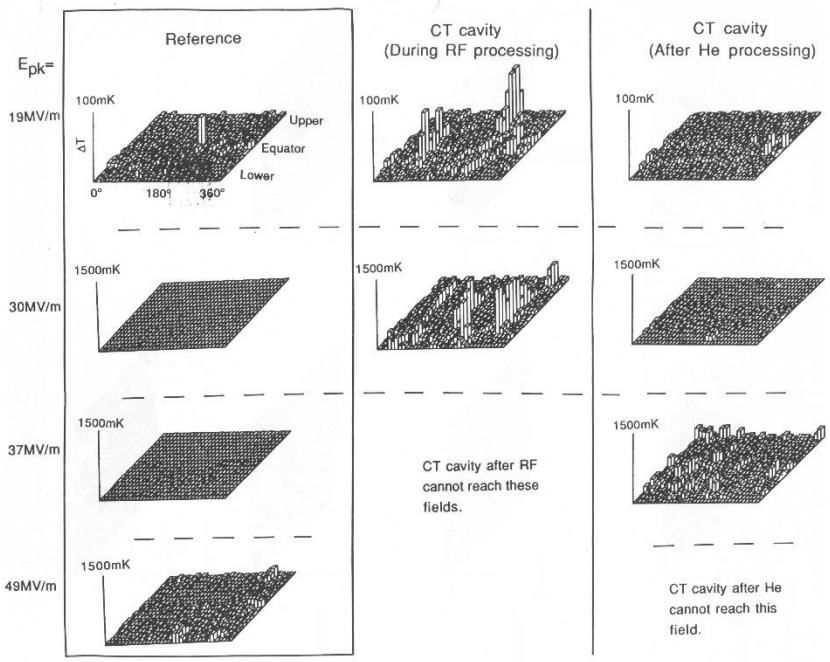

Fig.2(a). Comparison of temperature maps before(reference) and after exposure to chemical etching agents(CT), corresponding to the cavity and treatments of Fig.1(a).After exposure the cavity has many more and stronger emitters.

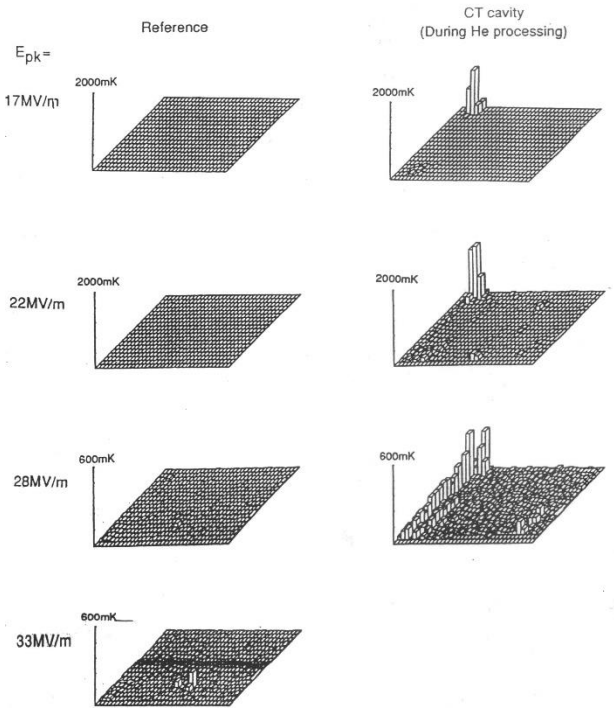

Fig.2(b). Comparison of temperature maps before(reference) and after exposure to chemical etching agents(CT), corresponding to the cavity and treatments of Fig.1(b).The emission site apparent at 22 MV/m after CT has been RF-processed a way before reaching 28 MV/m but new stronger emitters still occur.

4.1. Overall RF Performance

Figures 3(a) and 3(b) present the overall changes of RF performance of the two cavities after exposure to clean DI water. In Fig. 3(a), Q in the reference state is flat at 4×10^9 to about 40 MV/m, and the surface field E_{pk} reached 53 MV/m with a Q of 1.5×10^9. After exposure of the cavity to DI water, E_{pk} could only reach 38 MV/m with Q less than 1×10^9. However, He processing returned Q to about the same value as before exposure, but the maximum surface field was not recovered. In the case of Fig. 3(b), the reference state went to 40 MV/m with $Q = 7\times10^9$ and quenched due to defect thermal breakdown. After exposure it could reach as high a field as before, but with a Q of only 9×10^8. After He processing, both the highest field and the Q recovered.

4.2. Temperature Maps

Figure 4(a) shows maps for the same cavity as Fig.3(a) taken at the highest field, 38 MV/m, attainable after exposure.The reference state shows no emitter at this field.After exposure, the cavity has one strong and several weaker emitters. After He processing the dominant emitter has disappeared, but some of the same weaker emitters are still present.

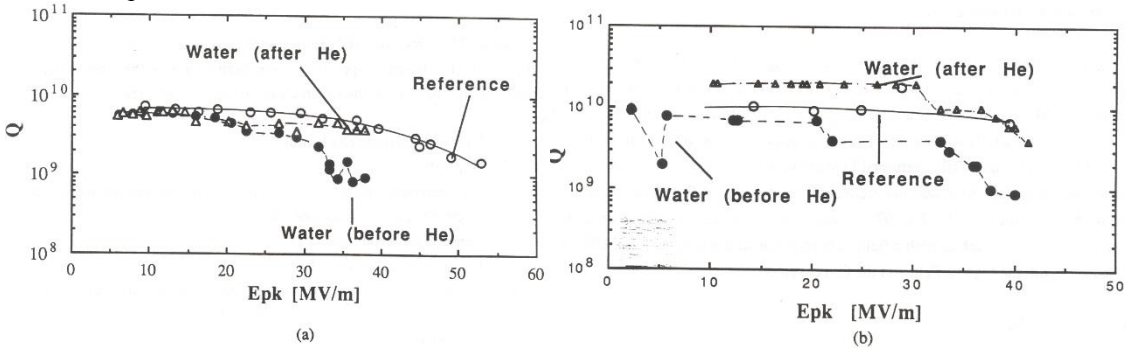

Fig.3.Overall RF performance of two cavities before(reference)and after exposure to de-ionized water.

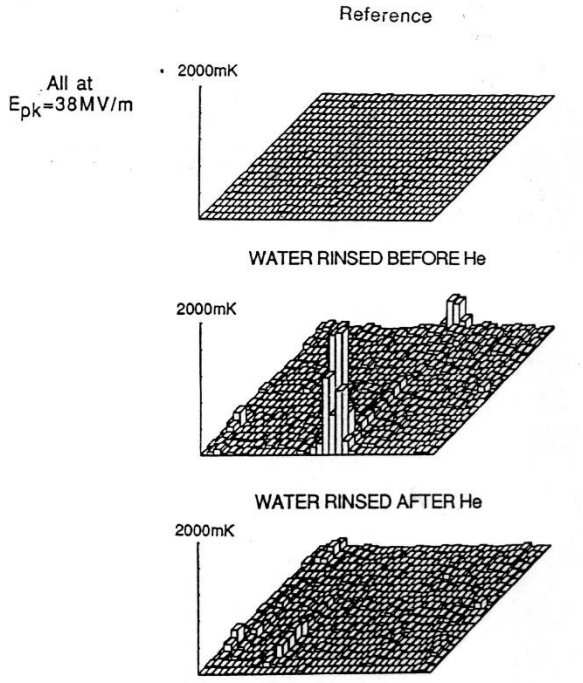

Fig.4(a).Comparison of temperature maps before(reference) and after exposure to de-ionized water,corresponding to the cavity and treatments of Fig.3(a).The emission site visible in the center and absent from the lower map has been He-processed away.

Figure 4(b) shows maps for the same cavity as Fig. 3(b) taken in increasing fields.The maps in the left column show that this cavity in its reference state has no emitter until E_{pk} = 39 MV/m. After exposure to DI water (center column), it starts emission at E_{pk} = 15 MV/m. Although it is able to reach as high a field as before exposure, the emission becomes very strong with the appearance of many new emitters. Thus the Q of the exposed cavity is lower than before exposure. After He processing (right column), no significant emitter appears until cavity breakdown.

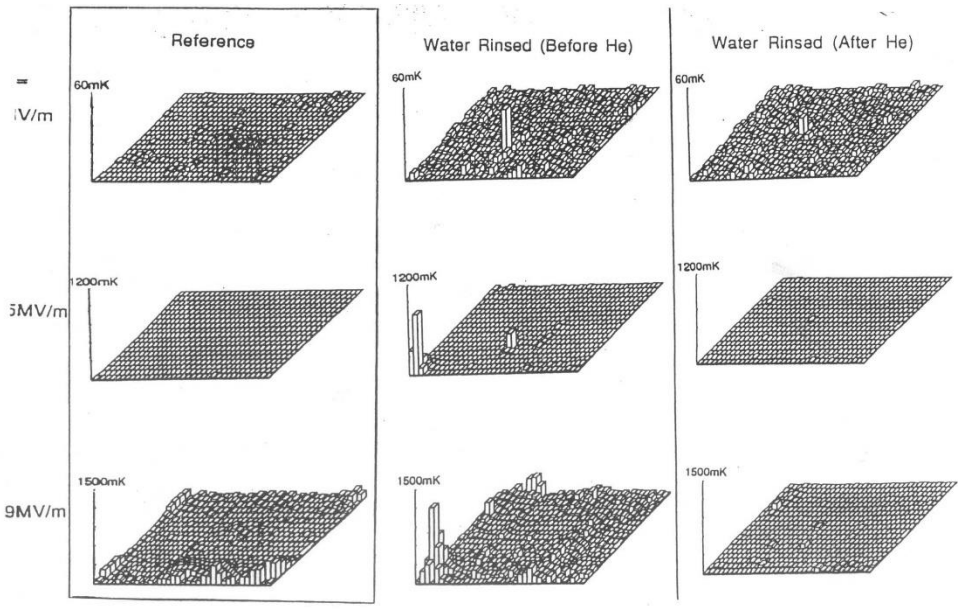

Fig.4(b).Comparison of temperature maps before (reference) and after exposure to de-ionized water,corresponding to the cavity and treatments of Fig.3(b).The two emission sites apparent at 25MV/m in the center column were He-processed away;the lower left peak at 39 MV/m occurs at a significantly different location.

5. EXPOSURE TO HIGH PURITY METHANOL

Since rinsing with high purity methanol is the final step of our cavity surface preparation, it is important to know whether methanol will introduce or activate emission sources on cavity surfaces. To investigate this, two HT cavities were well He-processed as references and then their inner surfaces were exposed to high purity methanol following the same procedure as with exposure to water.

5.1. Overall RF Performance

Figures 5(a) and 5(b) show the Q vs E_{pk} behavior of the two cavities. In that of Fig. 5(a), both the Q and the highest E_{pk} after exposure to high purity methanol not only did not degrade, but also slightly improved even before He processing. After He processing the highest E_{pk} increased to 53 MV/m with Q above $1\times10^9$and the flat Q region increased its extension to 40 MV/m. For the cavity of Fig. 5(b), the highest field in the reference state was 41 MV/m with Q of 7×10^8; Q is flat to 27 MV/m with a value of about 1×10^{10}. After exposure, but before He processing, the highest E_{pk} decreased to 34 MV/m and the Q in the flat region was slightly lower than before exposure. After He processing both Q and the highest E_{pk} recovered and even slightly improved to 42 MV/m andQ of 2×10^9.

5.2. Temperature Maps

The maps in Fig. 6(a) (same cavity as in Fig. 5(a)) were taken at E_{pk} = 45 MV/m. Several significant emitters at this field are apparent in the reference state. After exposure, but before He processing, the spatial distribution of emitters changed, and the emission level significantly decreased. After He processing, the number and strength of emitters are both seen to be further reduced. The maps in Fig. 6(b) (same cavity as in Fig. 5(b)) were taken at 34 MV/m and about 42 MWm. At 34 MV/m, there is no significant emission in the reference state, but three significant emitters are apparent in the exposed cavity before He processing. At 40 Mv/m, about six significant emitters can be seen in the reference state with somewhat fewer and weaker emitters in the exposed cavity after He processing. A detailed analysis of these emitters will be presented in a separate paper.

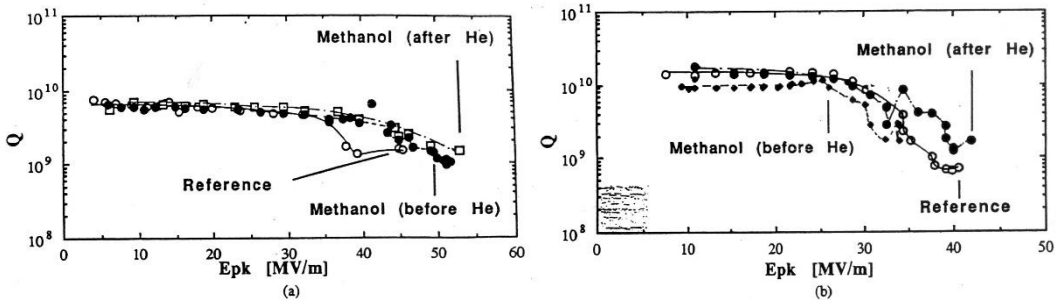

Fig.5. Overall RF performance of two cavities before(reference) and after exposure to high-purity methanol.

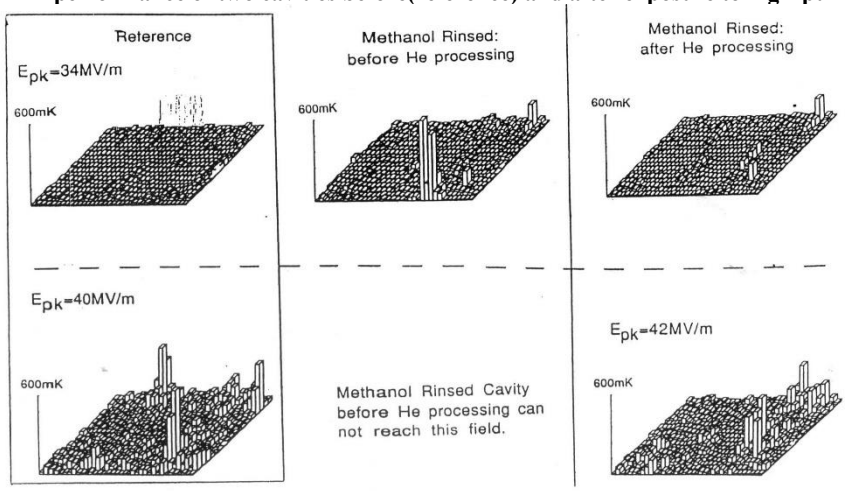

Fig.6(a).Comparison of temperature maps before(reference)and after exposure to high-purity methanol,corresponding to the cavity and treatments of Fig.5(a).

All maps at E_{pk}=45MV/m

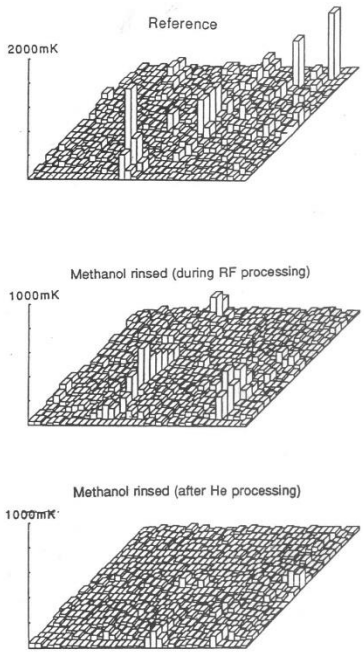

Fig.6(b).Comparison of temperature maps before(reference)and after exposure to high-purity methanol, corresponding to the cavity and treatments of Fig.5(b)

6. EXPOSURE TO CLEAN AIR

Clean air is one of the most important media to be tested since cavities are always necessarily exposed to clean air in the process of carrying out other exposure tests. A knowledge of the effects of air exposure allows us to separate out the influences of other media being tested. To investigate clean air effects, a well processed cavity was left in the cryostat after warming up to room temperature. The cavity was then filled to one atmosphere with class-100 dust-free air, taken from a laminar blower, through the backfilling line of the vacuum system. After letting the air stay in cavity for a few minutes, we then evacuated the cavity and cooled it down to test again.

6.1. Overall RF Performance

Figure 7(a) is a comparison of Q vs E_{pk} between the reference state of the cavity and the cavity after exposure to clean air. Both curves nearly overlap, showing that exposure to clean air did not significantly change the overall RF performance of the cavity.

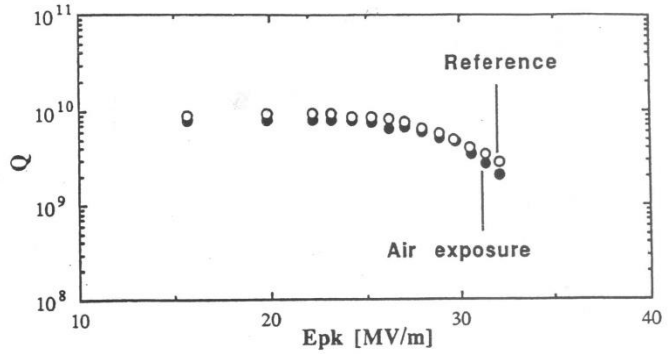

Fig.7(a). Overall RF performance of a cavity before(reference)and after exposure to dust-free air.

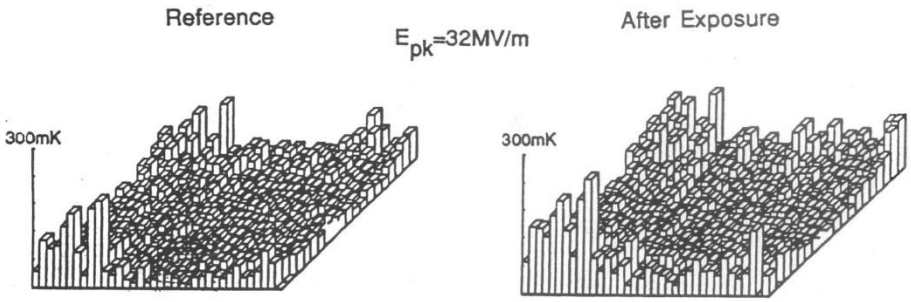

Fig.7(b). Comparison of temperature maps before (reference) and after exposure to dust-free air,corresponding to the cavity and treatments of Fig.7(a)

6.2. Temperature Maps

Figure 7(b) shows that the temperature maps from the exposed cavity and its reference state are quite similar at about the Same fields. The distribution and intensity of emission sources did not change significantly on exposure of the cavity to clean air.

7. ROOM TEMPERATURE CYCLING

For completeness, the influence of cycling cavities to room temperature without any other physical disturbance needs to be studied, since like exposure to clean air such cycling is also an inescapable part of the other exposure tests reported above. To this end, we tested two cavities. After an RF test ensuring good reference condition, each was left in the cryostat and allowed to warm to room temperature by natural heat transfer. The cavity was ion pumped at room temperature for about 24 hours and then cooled down again.

7.1. Overall RF Performance

Figures 8(a) and 8(b) show comparisons between the two cavities in their reference states and after room temperature cycling. In both cases the overall RF performance was not significantly changed by cycling to room temperature.

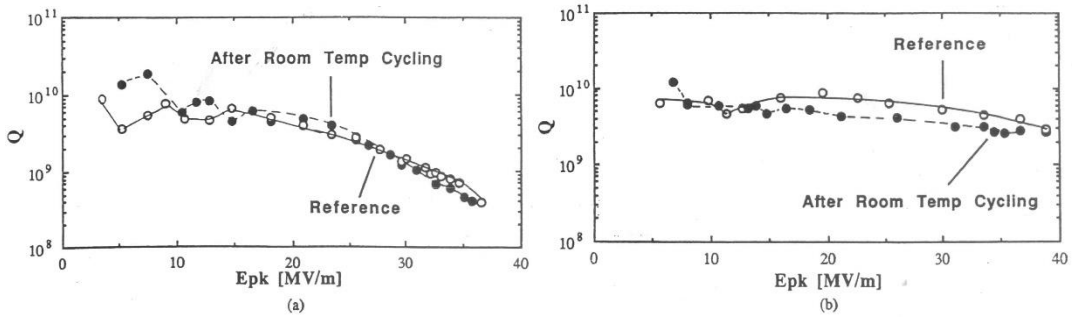

Fig.8. Overall RF performance of two cavities before(reference) and after room temperature cycling.

7.2. Temperature Maps.

Temperature maps corresponding to the cavities in Fig. 8 are shown in Fig. 9. The results apparent there are typical of those from several more room temperature cycling tests: the locations of the major emitters sometimes change and sometimes do not, but (consistent with Fig. 8) the total emission level remains essentially the same.

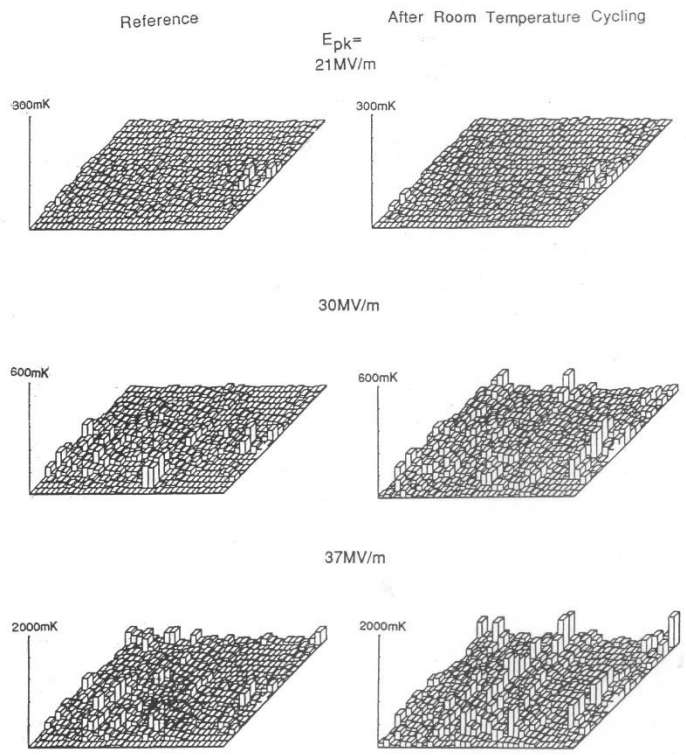

Fig.9(a). Comparison of temperature maps before(reference)and after room temperature cycling,corresponding to the cavity and treatments of Fig.8(a).

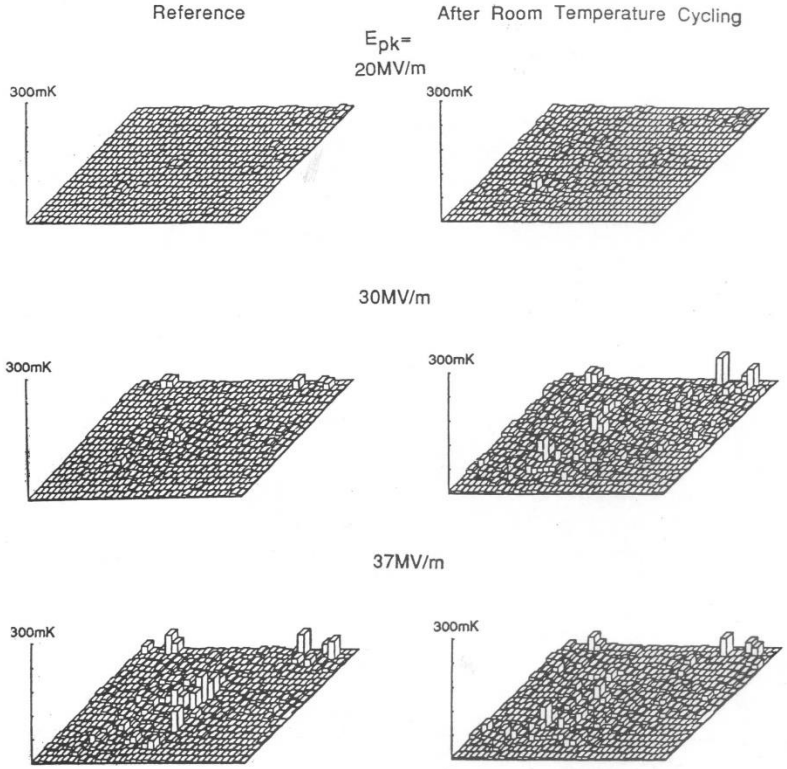

Fig.9(b). Comparison of temperature maps before(reference)and after room temperature cycling,corresponding to the cavity and treatments of Fig.8(b).

8. CHANGES IN EMISSION SITE β AND S

Heating sources on the inner surfaces of superconducting cavities can be broadly categorized into two types. One is caused by RF power dissipation at a localized defect on the surface. This heating is easily identified as proportional to the square of the local field, and does not concern us in this paper. The other kind of heating is due to power deposition by a field emitted current, and is of primary concern here. We assume that, whatever the detailed mechanism of the emission process, the emitter current I varies with the electric field E according to a Fowler-Nordheim law [7]:

$$I = \frac{AS\beta^2 E^2}{\phi} \exp\left[-B\frac{\phi^{3/2}}{\beta E}\right] \qquad (1)$$

where I is in A, E is in V/cm, ϕ is in eV (4eV for Nb), and A and B are constants. For a geometrical protrusion, S is the emissive area (in cm^2) and β is the geometrical field enhancement factor. By plotting $\ln(I/E^2)$ versus l/E, one can obtain β and S from the slope and intercept of the fitted straight line. While it is doubtful that literal geometrical protrusions are responsible for the emission in our cavities, we may nevertheless characterize a given emission site by the β and S obtained by the above procedure. Since our measurements give △T rather than I, a rather more complicated analysis is necessary to extract these parameters; it is described in references [2] and [8].

We are interested here in whether, and if so how, the β and S of emitters are changed by the exposures discussed above. It is difficult to answer this about an individual emitter, as it is rare for a specific emitter to survive one of these exposure tests. Statistically, however, we can extract some information from the more than a hundred emitters we have analyzed from the tests. The plot of S' vs β in Fig. 10(a) shows an example. (The intercept S', obtained most directly from our analysis, is proportional to S.) Six emitters identified from a particular cavity in its reference state are plotted as open circles. While the β's and S's vary greatly among these emitters, they fall in a band as indicated by the solid line. Other emitters identified on the same cavity after a chemical exposure form their own band, significantly shifted toward the "north-east". Helium processing (as previously reported in [4]) moves the band "south-west", in the direction of less emission and toward the reference band.

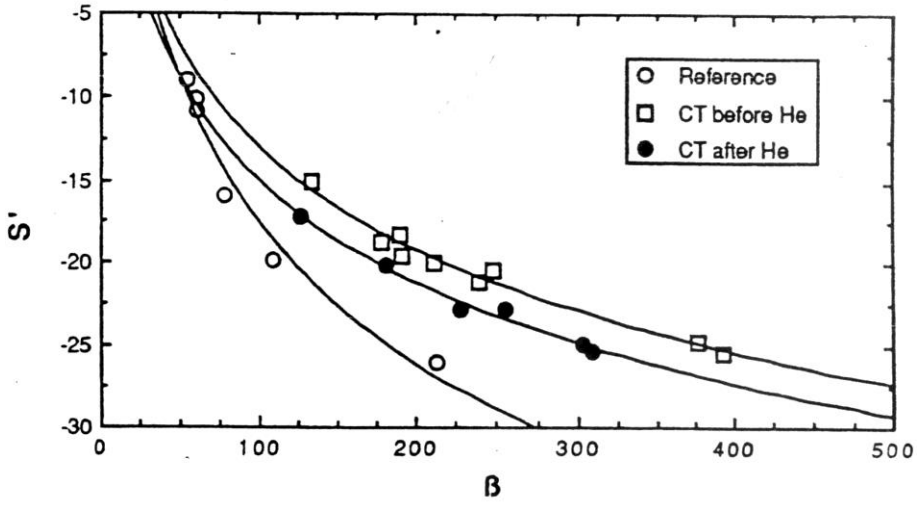

Fig.10(a). Fowler-Nordheim intercept S'(proportional to the emitter area S)vs field enhancement factor β for the emission sites of the cavity of Figures 1(a)and 2(a). The solid lines are drawn through points from similar cavity treatments,before(reference)and after exposure to chemical etching agents(CT).

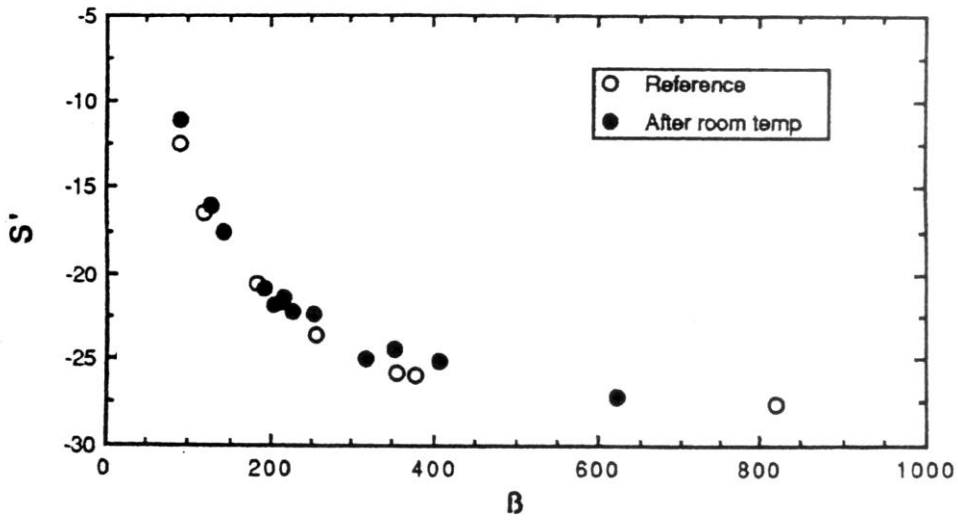

Fig.10(b). S'vs β for the emission sites of the cavity of Figures 8(a) and 9(a),before (reference)and after room temperature cycling.

For contrast, Fig. 10(b) shows the S' vs β distributions for a particular cavity before and after room temperature cycling. It is clear that the two distributions completely overlap-room temperature cycling has no significant effect. A complete discussion of the influence of different treatments on the β and S of emitters will be presented in a future paper.

9. DISCUSSION

The tests described above suggest that the new emission sites introduced by exposure to different media are of two basic kinds. One, produced by chemical etching agents, is not He processable and causes a consistent degradation of cavity performance. The other, like those from DI water, is He processable and the cavity performance is recoverable. Clearly the stage of CT is the weak one in the conventional (non-HT)preparation of SRF cavity surfaces.

It is striking that a chemical treatment degrades a cavity surface even after a previous HT. The cavity appears to have no "memory" of its previous condition after a CT. We see two ways in which this may come about. (1) It may be that HT removes emission sources only from the outer skin of the Nb. A subsequent CT removes this layer, exposing a new "active" layer of Nb. (2) Alternatively, a CT may leave a residue of emission sites (arising from the reaction products or the chemicals themselves) in spite of the rinsing that follows it. A subsequent HT dissolves or evaporates this residue, leaving a relatively emission-free surface. Which of these two possibilities is in fact the case is not yet clear, we plan further tests to resolve the matter.

10. CONCLUSION

The results presented above can be summarized in the following conclusions:

1. Chemical treatment seriously degrades a well processed HT cavity surface by increasing FE; such a degraded surface can not be restored by He processing.
2. Exposure to clean DI water causes a similar degradation, but the emission sources can usually be He processed to recover the previous performance.
3. The high purity methanol rinse is eliminated as an abundant source of emitters. Rinsing with methanol does not degrade the performance of a cavity.
4. Exposure to class-100 dust-free air does not introduce an abundance of emitters.
5. Cycling a cavity to room temperature without any physical disturbance may suppress active emitters and turn on other dormant emitters. However, neither their average strength nor the overall RF performance of the cavity is affected.
6. Statistically, the distribution of β and S of emitters in the cavity after chemical exposure moved in the

direction of higher emission. The trend is toward stronger, as well as more, emission sites.

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