Experience with High Pressure Ultrapure Water Rinsing of Niobium Cavities

P. Kneisel, B. Lewis and L. Turlington
Continuous Electron Beam Accelerator Facility
12000 Jefferson Avenue
Newport News, VA 23606 USA

Summary

In a series of experiments on single and 5-cell L-band niobium cavities high pressure ultrapure water rinsing was used as a cleaning step after buffered chemical polishing. The cavities generally exhibited little field emission loading. In the test series on a single cell cavity peak surface fields of $E_{peak} = 50$ MV/m were reproducibly obtained after each new chemical surface treatment without field emission loading.

Introduction

Since the availability of high purity niobium with RRR—values 2 250, superconducting niobium cavities used in particle accelerators are no longer limited in their performance by thermo-magnetic breakdown at defects at design accelerating gradients $E_{acc} \leq 8$ MV/m.

The principle limitation encountered at field levels above ≈ 8 MV/m is field emission loading with exponentially increasing losses as the field levels are increased. Therefore, except in a few exceptional cases, progress towards higher gradients for future applications of RF-superconductivity can only be achieved if the onset of field emission loading can be shifted towards higher fields.

It is generally believed that the field emission behavior of a niobium cavity reflects the level of cleanliness of the superconducting surfaces subject to the RF-fields.' Artificial emitters introduced into the cavities during surface treatments and assembly steps are the major causes for the emission of electrons. Emitters intrinsic to the material such as impurity segregation have only been identified after high temperature heat treatments.[1]

Two approaches have been taken in the past to achieve higher performance levels of accelerating cavities:

a. ultrahigh vacuum annealing in the presence of a solid state gettering material such as titanium [2,3] and

b. high peak power processing of cavities.[4]

In both techniques emitters clinging to the surfaces are destroyed. Both approaches have been applied successfully and accelerating gradients in the neighborhood of $E_{acc} \approx 20$ MV/m have been achieved in multi-cell cavities, corresponding to peak surface electric fields of $E_p \approx 40$–50 MV/m. Such activities are being pursued in other laboratories.

At CEBAF production cavities are surface treated with buffered chemical polishing solutions followed by thorough rinsing with ultrapure water. In some exceptional cases very good cavity performances have been measured.[5]

Based on CEBAF's experience in wet chemical processing we have looked at an improved rinsing procedure after the buffered chemical etching by directing a jet of high pressure ultrapure water towards the surfaces. Successful application of such a cleaning method has been reported earlier.[6,7]

In the following, results of cavity performance after application of this procedure are reported.

The High Pressure Rinsing System

The system is schematically shown in Figure 1. It consists of a high pressure pump, a filter, a spray nozzle and a mechanical system, which allows the scanning of the interior cavity surface with the high pressure water jet. Because of budget constraints we chose the ;following - by far not optimized - components for the system: the commercial, inexpensive high pressure pump (Kärcher Model "Special 1855–878") supplies ≈ 81 of water per minute at 80 bar; the filter is a 0.1 µm cellulose filter (Domnick–Hunter "Asypor") rated for 80 bar and located in an unpolished stainless steel housing. Connecting Lines are made of teflon with stainless steel braids; the spray nozzle and the rigid feedline from the filter to the nozzle are made of type 304 stainless steel. The "scanning system", moves the cavity up and down while also rotating it. Both up–and–down–speed and rotational speed are adjustable by means of motors with variable speed controls.

For the experiments reported here we chose a rotational speed of ≈ 4 rpm and a vertical speed of ≈ 70 cm/min. Four jets of high purity water are emerging from the spray nozzle under 45° (up and down) and 180° apart in azimuth as indicated in Figure 1. During the high; pressure cleaning procw single cell cavities were rinsed under these conditions for 20 min with ultra pure water with a resistivity ≈ 18 MR cm; 5 cell cavities were rinsed for ≈ 60 min. Prior to the application of the high pressure water jets the system was operated for ≈ 30 min in order to clean it from possible contamination during non-use. As mentioned above, the system is far from being optimized: At the very least the high pressure pump should be made from stainless steel (the one used is probably made from cast iron) and the filter housing and connecting stainless steel parts should be electropolished. In addition, the system should be incorporated into the ultrapure water polishing loop, thus avoiding areas of stagnant water and eliminating the danger of bacterial growth. Also, a sampling port for paniculate sampling should be added at the downstream side of the filter. Nevertheless, the use of the present system resulted in surprisingly improved cavity performance as discussed below.

Test Procedure and Results

For the evaluation of the benefits of high pressure rinsing after chemical surface treatment, we used a single cell 1500 MHz CEBAF cavity fabricated from high purity niobium. During its lifetime this cavity had been post purified at 1400° C for 4 hrs in the presence of titanium. The cavity was electropolished by K. Saito at KEK, with a removal of 120 µm followed by a high pressure rinsing at KEK and evacuation. The cavity was sent to CEBAF under vacuum, where it was disassembled in the clean room and rinsed with reagent grade methanol prior to Test #1a (see Figure 2). Test #1b was done after a high pressure rinsing of the cavity with the system described here and subsequent methanol rinsing.

Prior to any subsequent experiments the cavity was always chemically treated with buffered chemical solution of equal parts of hydrofluoric (48%), nitric (69%) and phosphoric (86%) acids followed by high pressure rinsing with ultrapure water and rinsing with reagent grade methanol, assembly in the cleanroom and attachment to the cryogenic test stand.

Once a vacuum of $\leq 6 \times 10^{-7}$ torr was reached in the cavity, it was cooled down within less than 30 min to 4.2 K in an ambient magnetic field ≤ 5 mGauss.

The results of this series of tests are summarized in Figure 2 and in Table 1: In all experiments no radiation was detected outside the cryostat for peak surface fields $E_p \leq 50$ MV/m. Except for Tests #2 and #5 no radiation was observed even at surface fields > 50 MV/m, when the Q-value started to decrease. This decrease might be due to emission directed away from the radiation detector towards the bottom of the cryostat or due to a loss mechanism different from field emission such as e.g. heating in the input coupler. In test #5—after reaching a peak field of $E_p \approx 50$ MV/m—a Q-switch occurred and subsequently X-radiation was observed below the original values.

Several experiments with different single cell cavities were carried out after these exploratory tests and showed similar results: a very minute level of field emission loading or complete absence of field emission at surface fields below 50 MV/m .

Experiments with fully equipped 5-cell production cavity pairs were not as successful. Nevertheless, in about 5 tests out of 10 the Q_0 remained constant with increasing accelerating gradient and significant field emission loading was not observed. One of the more spectacular results on a production cavity pair is shown in Figure 3: In the original test both cavities of this pair exhibited strong field emission loading at modest field levels; after this test the cavity pair was disassembled and merely high pressure rinsed prior to re-assembly and testing. The performance of the cavities had changed dramatically: No field emission loading was encountered up to the quench fields of the cavities.

Additional experiments were carried out with a production cavity after post-purification at 1400° C for 4 hrs in the presence of titanium. In several tests, after chemical surface treatment and high pressure rinsing had been applied, accelerating gradients of up to $E_{acc} = 17$ MV/m corresponding to peak surface fields of ≈ 43 MV/m without field emission loading were measured.[9]

Conclusions

From these experimental results we can draw a few conclusions;

a. High pressure rinsing as a final cleaning step after chemical surface treatment resulted in consistent performance of single cell cavities with a very small spread in the data: field emission loading seemed to be absent for peak surface fields $E_p \leq 50$ MV/m.

b. Rinsing with reagent grade methanol as a last step in surface preparation prior to assembly does not conmbute to field emission loading for $E_p < 50$ MV/m and dust or dirt from the test fixture also apparently does not contribute to loading below these field levels.

c. Mere high pressure rinsing can improve surface cleanliness resulting in improvements of Q-value and field emission loading behavior .

d. Comparing *test* 1a and 1b and excluding methanol rinsing as a source for field emission sires. one can conclude chat particulate matter or chemical residue is not totally removed by *just* purely rinsing (here rinsing with ultrapure methanol), but that a certain impact of the rinsing agent (here ultrapure water) is needed to overcome the forces between the surface and the particulates.[8)

e. In all experiments the reported gradients were measured with no or only very short (= min) RF-processing necessary. Usually the fields in the cavities could be established immediately just by raising the incident power levels.

f. As far as the tests results on the 5-cell cavities is concerned, one has to keep in mind that these assemblies are of a much more complex nature than single cell cavities and the possibility of introducing particulates through peripheral parts such as HOM-loads, ceramic RF-windows or gate-valve assemblies is always present. Nevertheless. several successful experiments demonstrated *the* usefulness of the high pressure rinsing and future experiments with temperature mapping capability are needed to identify the nature of field emission loading after high pressure rinsing,

Acknowledgement

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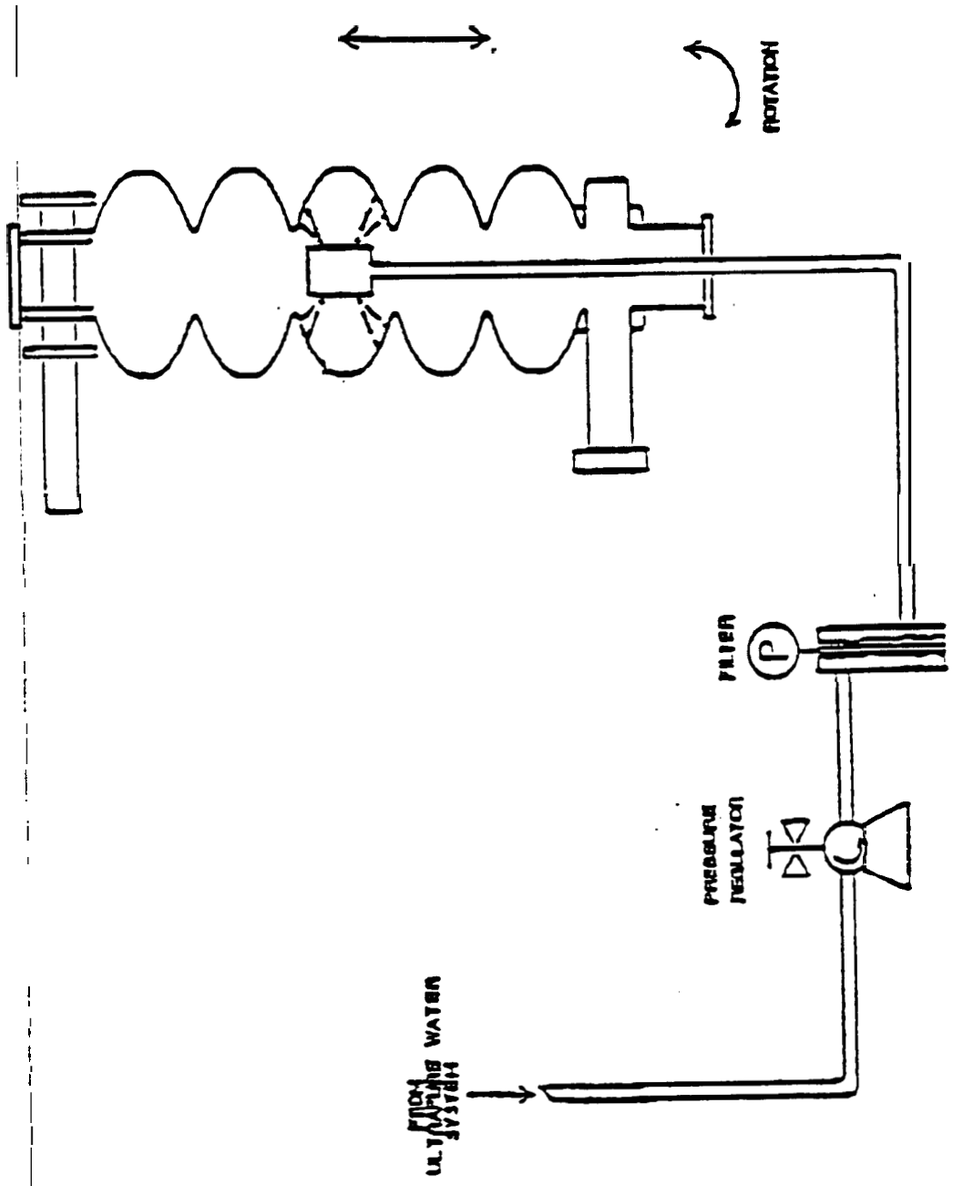


Figure 1 : Schematic of High Pressure Rinsing System

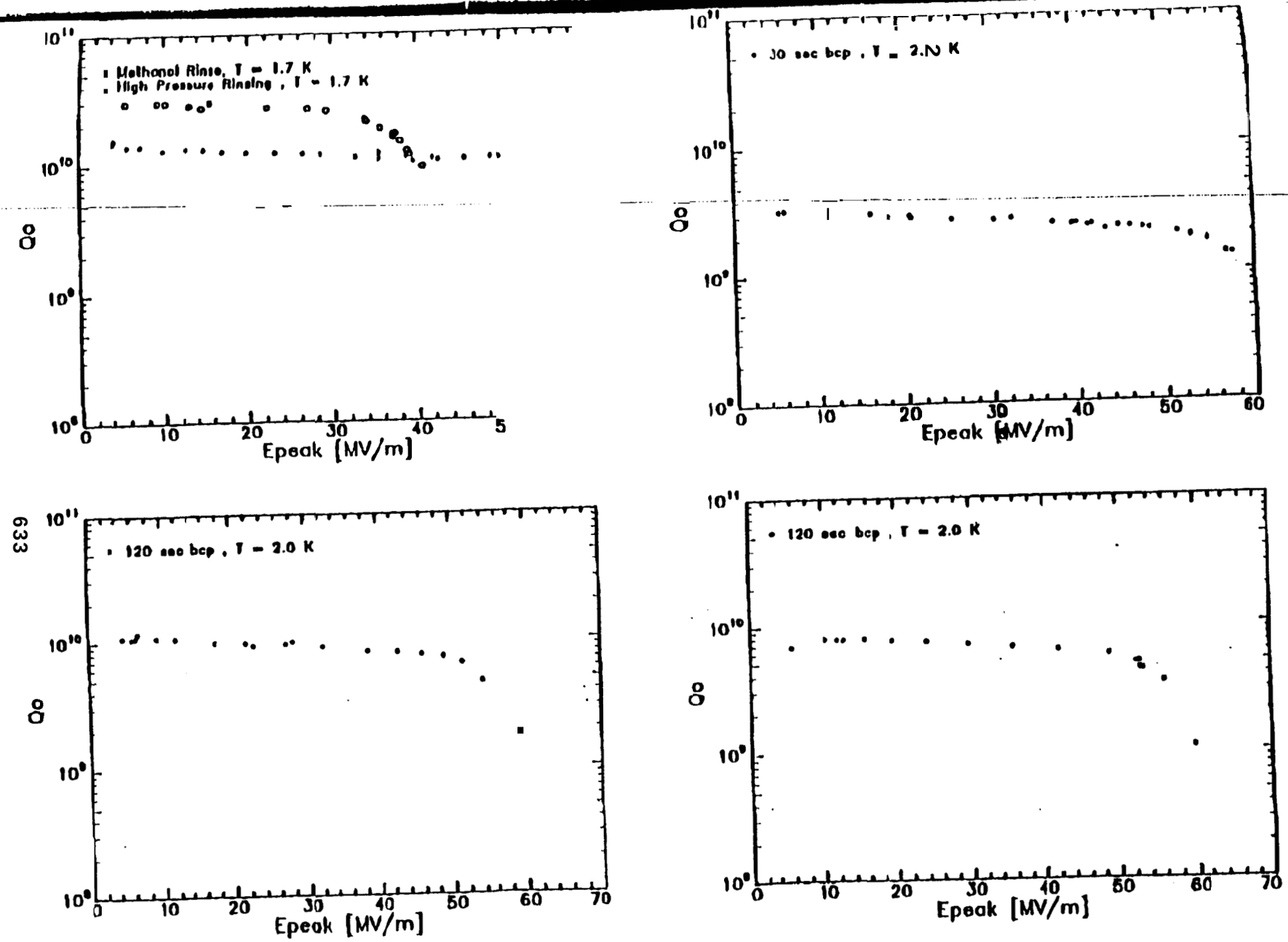


Figure 2 : Results of Single Cell Experiments

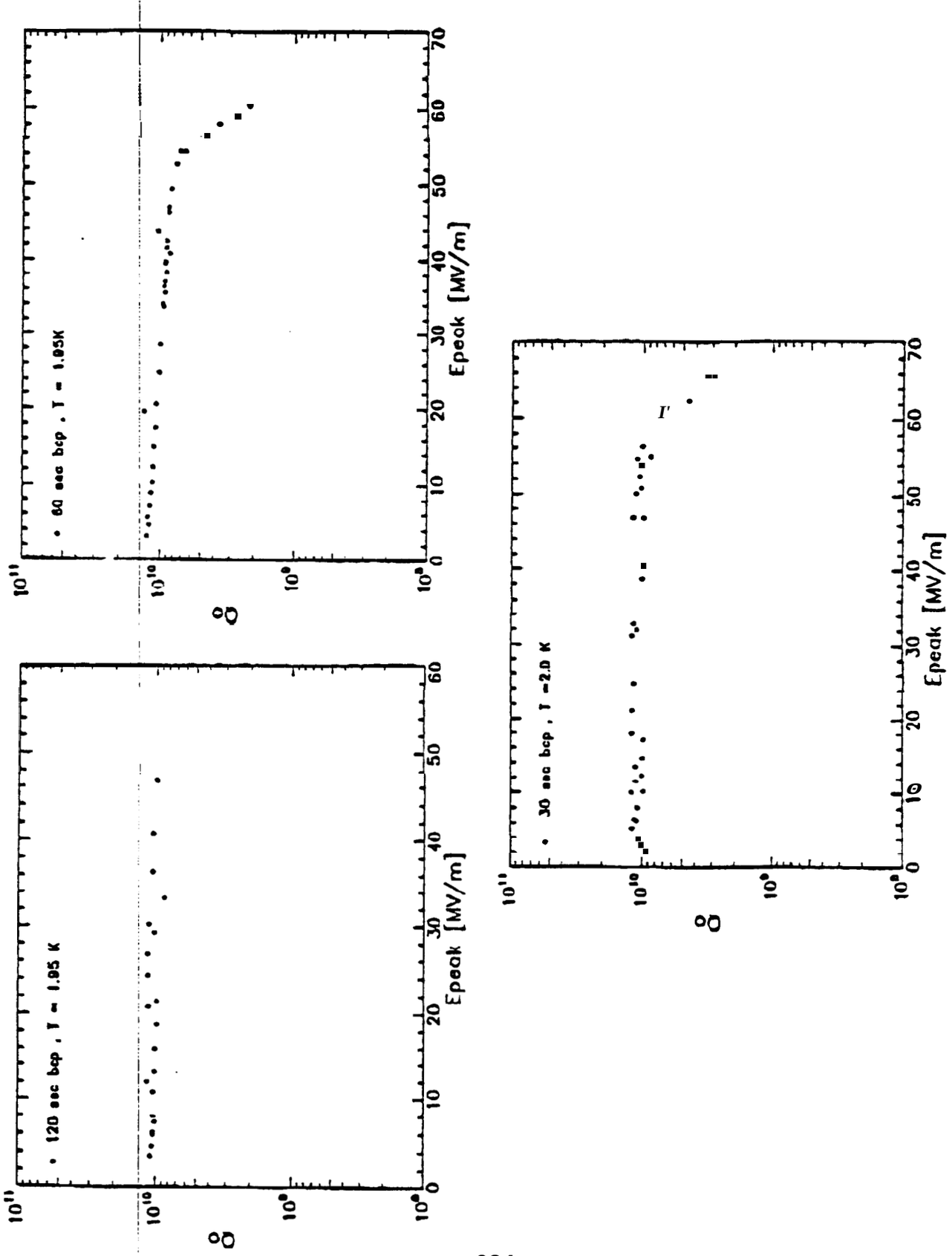
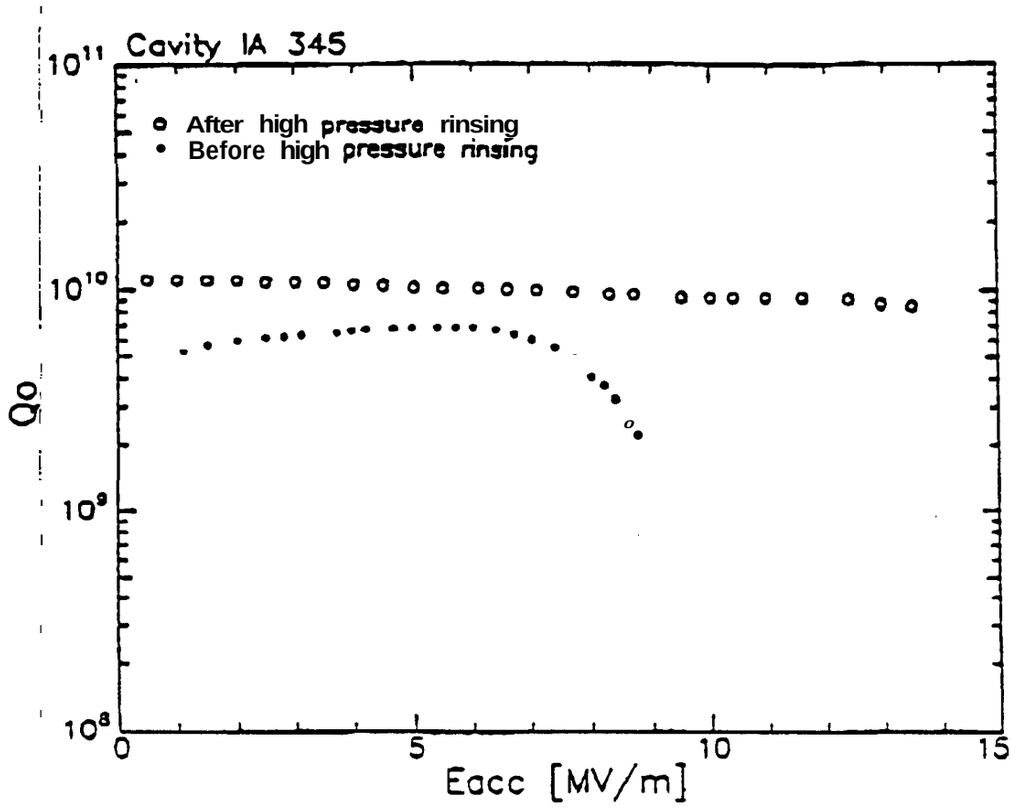


Figure 2 : Results of Single Cell Experiments (cont'd)



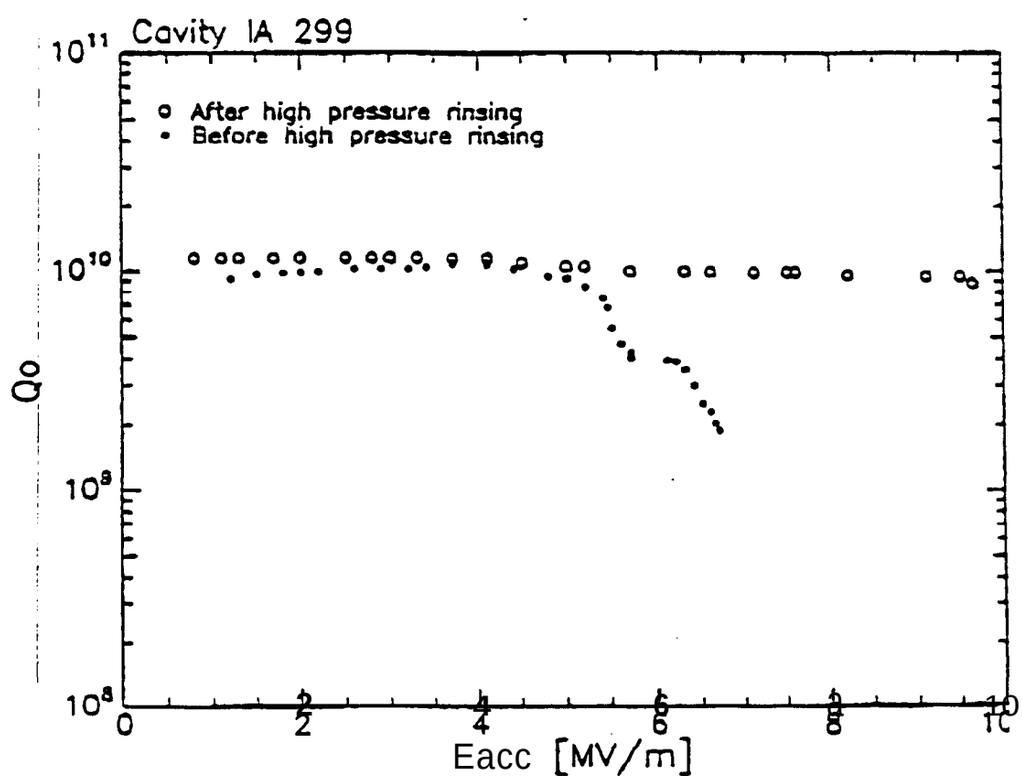


Figure 3 :Results of Production Cavity Pair IA345/IA299 before and after High Pressure Rinsing (measurements have been taken by P.Kushnik and T.Powers)

Table 1 : Results from Single Cell Tests after High Pressure Rinsing (HPR)

Test #	Preparation	Removal rate [kHz]	R_{res} [nOhm]	E_{peak} [MV/m]	$E_{FEonset}$ [MV/m]	Comment
1a	methanol rinse, HPR	0	12.9	36.5	≥ 24	
1b	HPR	0	15.6	49.8	> 49.8	no FE;test limited by He level
2	30 sec bcp; HPR	52	11.4	57.5	≥ 50.5	radiation for E_p ≥ 50.5 MV/m
3	2 min bcp; HPR	133	23.0	57.2	>49.5	no radiation observed at E_p
4	2 min bcp; HPR	147	24.2	59.7	≥ 51	no radiation observed at E_p
5	2 min bcp; HPR	227	25.0	≈ 50	≈ 50	at E_p Q-switch; FE at lower E_p
6	1 min bcp; HPR	101	6.64	60.5	≥ 54	no radiation observed at E_p
7	1 min bcp; HPR	142	11.7	64.8	≥ 55	no radiation up to E_p = 62 MV/m