

# HIGH RELIABLE SURFACE TREATMENT RECIPE OF HIGH GRADIENT SINGLE CELL SRF CAVITIES AT KEK

F. Furuta, K. Saito, T. Saeki, H. Inoue, Y. Morozumi, Y. Higashi, and T. Higo  
 KEK, High Energy Accelerator Research Organization, 1-1 Oho, Tsukuba 305-0801, Japan

### Abstract

We have continued the series tests of single cell superconducting cavities at KEK. These tests are aimed at establishing a high yield production of surface treatment that would reliably allow cavities to reach gradients in excess of 45 MV/m with high Q in vertical tests. The cavity shape is all of the KEK Low Loss design. Early results from this series test demonstrated that reaching gradients as high as 50 MV/m was feasible. However, the initial yield was of order 50 %. In order to increase the yield we have modified our surface preparation followed an established KEK procedure (KEK-WG5 recipe). We have succeeded to reach the >95 % yield with gradient of > 45 MV/m.

### INTRODUCTION

For the ILC mass productions, one of the big concerns is how to push up the production yield on the cavity performance. So after our successful principal proof of the high gradient 50 MV/m with high gradient shapes: Low Loss (LL) shape by J. Sekutowicz [1], Reentrant (RE) shape by V. Shemelin [2] and Ichiro shape (IS) based on Low Loss, we have revisited surface treatment issue to produce reliably the ACD (Alternative configuration design) goal,  $E_{acc} > 40$  MV/m,  $Q_0$  value  $> 0.8E+10$  with yield  $> 90$  %.

### CAVITY PREPARATION

After the 1<sup>st</sup> ILC workshop on Nov. 2004 we have started the R&D of high gradient cavities [3]. We have tested single cell cavities (LL, and RE shapes) more than 50 times by Nov. 2005, Figure 1 shows the histogram for these results. There were two Gaussian distributions, blue one related to field emission (FE) and red one related to hard quench. At that time, ave.  $E_{acc}$  was  $31 \pm 12$  MV/m, and the scatter was 40 %. These results include several potential problems: skill problem of newcomers, final

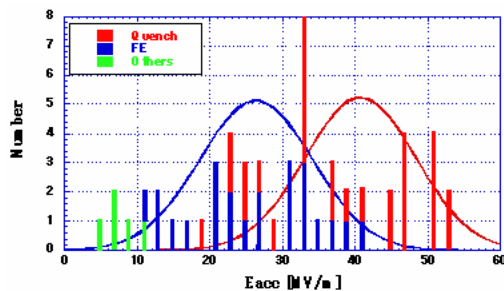


Figure 1: Histogram of cold test results by Nov. 2005.

#fumio.furuta@kek.jp

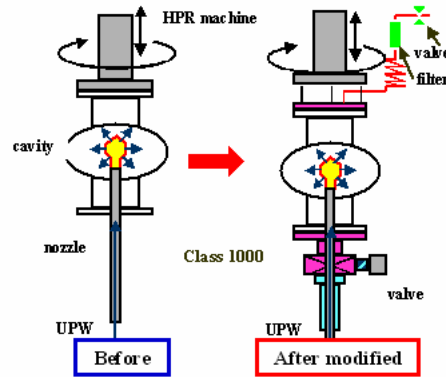


Figure 2: Cavity isolation during HPR

high pressure rinsing (HPR) in cavity preparation, and RF processing method on multipacting (MP) region in vertical. We have improved these issues as follows. Newcomers have skilled up through their experiences. The cleanliness of our HPR room is class 1000. So, our HPR method was modified to isolate cavity inside and outside during the process by water curtain and filtered air flow (Figure 2) [4]. As a water qualification, we started monitoring TOC (total organic carbon) and bacteria in ultra-pure water for HPR. Ave. TOC is less than 5 ppb. Bacteria are less than 1 piece/cc. Those numbers were very stable. We use a variable input coupler in our vertical test for cavity. So when we make RF processing MP, we can change the coupler position and set  $Q_{in}$  value lower  $1E+9$  in order to decrease a damage risk when quench happened.

These modifications effect could be found in Figure 3. It shows the histogram for all IS cavities cold test results since December 2005 (N=112). We have still two Gaussian distributions related to FE (blue) and hard quench (red). The ave.  $E_{acc}$  increased to  $37.0 \pm 10.9$  MV/m, the gradient scatter improved to 29 % but was still too large. But one can see clearly the FE limitation was pushed up and the ave.  $E_{acc}$  was improved about 6 MV/m.

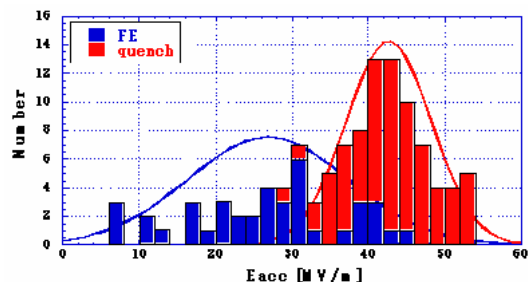


Figure 3: Histogram of all cold test results for IS cavities since Dec. 2005.

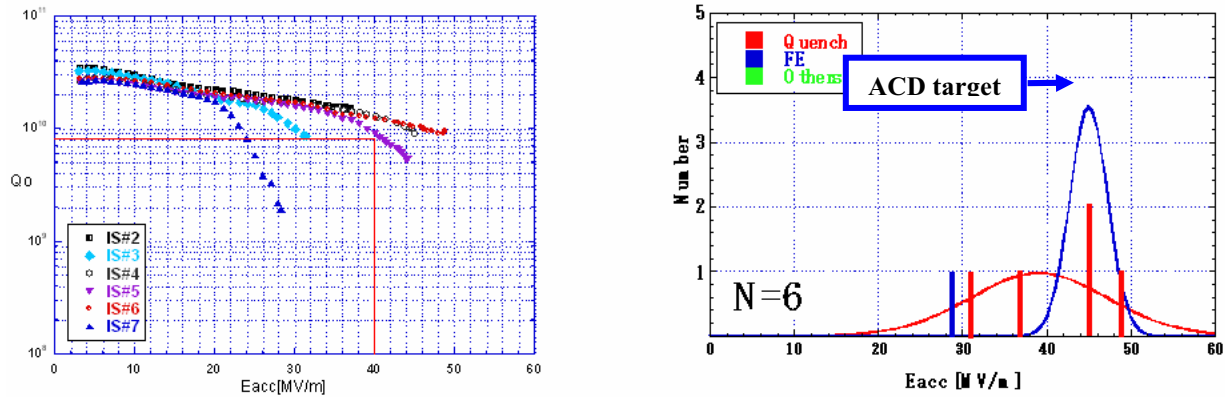


Figure 4: Results for recipe of CBP + CP + AN + EP (80  $\mu\text{m}$ ) + HPR + Baking, blue line is ACD target.

To investigate the gradient scatter we have made a single cell R&D plan. We newly fabricated 6 Ichiro single cell (IS) cavities and performed series of tests on preparation recipes. We applied them the KEK-WG5 standard recipe, which consists of Centrifugal barrel polishing (CBP 100  $\mu\text{m}$ ), Buffered chemical polishing (BCP 10  $\mu\text{m}$ ), Annealing (750  $^{\circ}\text{C}$  for 3 hours), Electropolishing (EP, 80  $\mu\text{m}$ ), High pressure rinsing (HPR) with ultra-pure water at 7 MPa for 1hour, Assembling in class 10 clean room, and Evacuation with Baking at 120  $^{\circ}\text{C}$  for 48 hours. After the surface preparation, these cavities were tested in a vertical cryostat at 2 K.

### PILOT STUDY FOR HIGH YIELD

After preparation by the KEK-WG5 recipe, the first vertical test was done for each cavity. The Q vs. Eacc plots and the distribution of reached gradients are presented in Figure 4. The IS#4, #5, and #6 achieved the gradient of 45 MV/m level, but the rest 3 cavities were limited at 28~37 MV/m by FE or hard quench. The ave. Eacc was  $39.1 \pm 8.2$  MV/m, the scatter 20 %, and the yield rate for ILC ACD acceptance 50 % in this first trial. We have found the KEK-WG5 recipe consisted of a heavy final EP (80  $\mu\text{m}$ ) has a large gradient scatter. This recipe could not satisfy the ILC ACD accept goal.

The limitations could be classified into three categories. The first one is still failures by mistakes in HPR or assembly in clean room (category-1) resulting in particle contamination. The field emission (FE) would be due to the category-1. The category-1 could be recovered by re-HPR. The category-2 is the limitation related to chemical contamination like sulphur or oxidation, which might cause Q-slope or FE. It might be recovered by additional light-EP or light-BCP. The category-3 is the limitation related to surface defects, which might be recovered by mechanical grinding CBP.

In order to investigate limitation mechanism on these 6 cavities, we applied additional several surface-treatments as a pilot study. The results are summarized in Figure 5. We firstly applied re-HPR, and then tried HF-rinsing for 20 minutes. These methods do not remove the SRF-niobium surface. Even the HF-rinsing removes just the

natural oxide layer ( $\sim 100$   $\text{\AA}$ ) on the SRF niobium surface. The Q-factor of IS#7 was improved remarkably, but no significant improvement was observed in gradients by the re-HPR. The rest 5 cavities were almost no change in both Q-factor and maximum gradient. HF rinsing also have no remarkable improvement on the cavity performance. Here, we can conclude that the limitation of these cavities is not the category-1: particle contamination.

Secondly, we applied light CP (10  $\mu\text{m}$ ) for only one cavity and flash EP (3  $\mu\text{m}$ ) with fresh EP acid for the rest cavities. The gradients were improved up to 40 MV/m. Q-slope limited the gradient in the BCP'd cavity. This additional light material removal is not so effective to improve the gradient but effective to reduce the gradient scatter. Here we can conclude that the limitation in the KEK-WG5 recipe is no related to category-1 neither category-3. It is close to the category-2 but the chemical contamination could still stay underneath of the SRF-surface.

Finally, we tried final EP (20-30  $\mu\text{m}$ ) + EP (3  $\mu\text{m}$ , fresh acid) and optionally HF-rinsing. All cavities successfully reached the gradient of 45 MV/m level. These results suggests us that the source of the scatter could be generated during a heavy EP process and immigrates into the niobium bulk if the EP process duration is long enough. The immigration might continue during vacuum

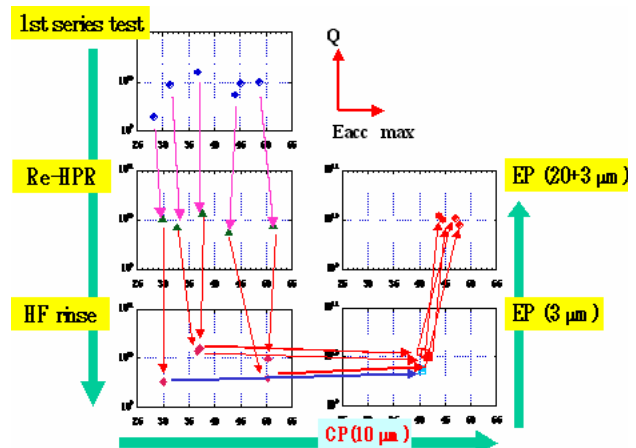


Figure 5: Pilot study for surface preparation

evacuation after the EP process. So if the cavity is once contaminated and stays long duration at the room temperature, the contamination would diffuse deeper than 3  $\mu\text{m}$ , thus even the flash EP (3  $\mu\text{m}$ ) could not remove the contamination. The additional EP (20  $\mu\text{m}$ ) could remove this diffused contamination. However, EP (20  $\mu\text{m}$ ) has still a risk of re-contamination during this process. The flash EP after the EP (20  $\mu\text{m}$ ) might eliminate the re-contamination risk. In these series of tests, we also checked hydrogen Q-disease after exposing the cavity around 100 K for longer than 12 hours. We tested totally 8 times with 4 cavities. No Q-disease was observed.

### CAVITY SERIES TESTS

After the 1<sup>st</sup> series of tests, we found that the scatter of cavity performance comes from some contaminations that cannot be removed by only HPR or HF rinsing. Sulphur is well known as one of contamination in EP process. In the second series of tests, firstly we applied an additional 3  $\mu\text{m}$  EP with fresh acid (flash EP) on the KEK-WG5 recipe. In addition we tried to remove the sulphur contamination by additional rinsing methods after EP process. We chose H<sub>2</sub>O<sub>2</sub> rinsing and degreasing because the both can dissolve sulphur. These series of tests are connected to the international single cell R&D plan that recommended in the TESLA Technical Collaboration meeting at Frascati on Sept. 2006 [5].

#### CBP+CP+AN+EP (80 $\mu\text{m}$ )+EP (3 $\mu\text{m}$ )+HPR+Baking

In the pilot study, we found that additional 3 $\mu\text{m}$  EP with fresh acid was effective to reduce the scattering. So we modified the KEK-WG5 recipe to add the flash EP after the EP (80  $\mu\text{m}$ ). We retreated 6 IS cavities with new recipe: CBP + CP (10  $\mu\text{m}$ ) + Anneal + EP (80  $\mu\text{m}$ ) + Flash EP + HPR + Baking, then cold tested. The results are shown in Figure 6. Ave. Eacc was 41.7 $\pm$ 4.4 MV/m. The scatter was 11 %. The yield rate for ACD accept was 67 %. The scatter became half compared with the 1<sup>st</sup> series of tests, so we again confirmed that the flash EP is effective to reduce the scatter. However, the amount of

the flash EP (3  $\mu\text{m}$ ) might be too small to remove the contamination underneath of the SRF-niobium surface.

#### +EP (20 $\mu\text{m}$ )+HPR+Baking

After the 2nd series of tests, we applied the recipe: +EP (20  $\mu\text{m}$ )+HPR+Baking, which is ILC baseline preparation recipe so far. The results are shown in Figure 7. 6 cavities were tested with this recipe. Only one cavity did not reached Eacc > 40 MV/m. The ave. Eacc was 46.5 $\pm$ 8.0 MV/m. the scatter was 17 %. The yield rate for ACD acceptance was 83 %. The scatter is still too large for the ILC ACD acceptance. Additional EP (20  $\mu\text{m}$ ) could remove the contamination underneath of the SRF-niobium surface. However, this recipe shows a re-contamination risk with small probability.

#### +EP (20 $\mu\text{m}$ )+H<sub>2</sub>O<sub>2</sub> rinsing+HPR+Baking

4 cavities were tested on H<sub>2</sub>O<sub>2</sub> rinsing. The results are shown in Figure 8. Ave. Eacc was 42.6 $\pm$ 7.6 MV/m, The scatter 18 %, and the yield rate for ACD acceptance 50 %. We will collect two more data soon for this recipe. In this recipe, the scatter is not improved but MP and FE were reduced remarkably as discussed later.

#### +EP (20 $\mu\text{m}$ )+Degreasing+HPR+Baking

5 cavities were tested so far with degreasing. Concentration of detergent was 0.2 %, which is same as JLAB use [6]. The results are shown in Figure 9. The ave. Eacc was 44.2 $\pm$ 6.4 MV/m, the scatter 14 %, and the yield rate for ACD acceptance 60 %. This recipe also did not improve the scatter but reduced MP and FE at higher gradient.

#### +EP (20 $\mu\text{m}$ )+Flash EP+HPR+Baking

6 cavities were tested with flash EP. The results are shown in Figure 10. Ave. Eacc was 46.7 $\pm$ 1.9 MV/m, The scatter only 4 %, and the yield rate for ACD acceptance 100 %.

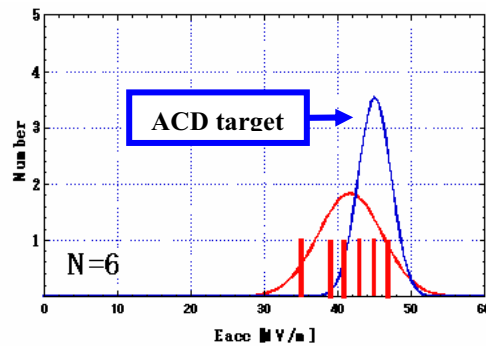
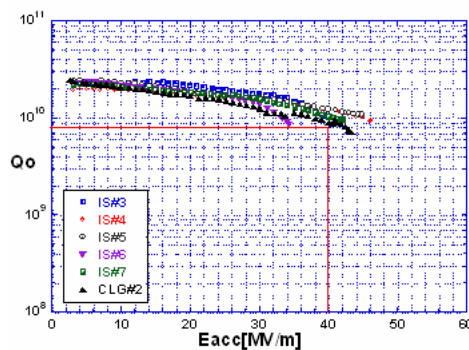


Figure 6: Results for recipe: CBP + CP + AN + EP (80  $\mu\text{m}$ ) + Flash EP (3 $\mu\text{m}$ )+ HPR + Baking

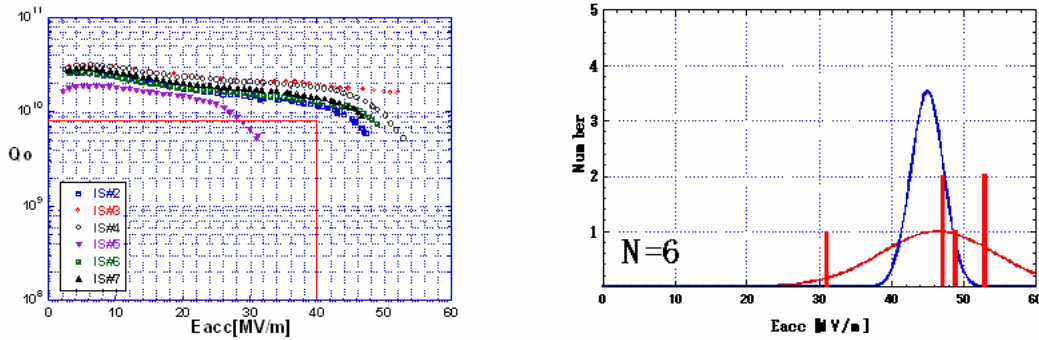


Figure 7: Results of the ILC baseline recipe

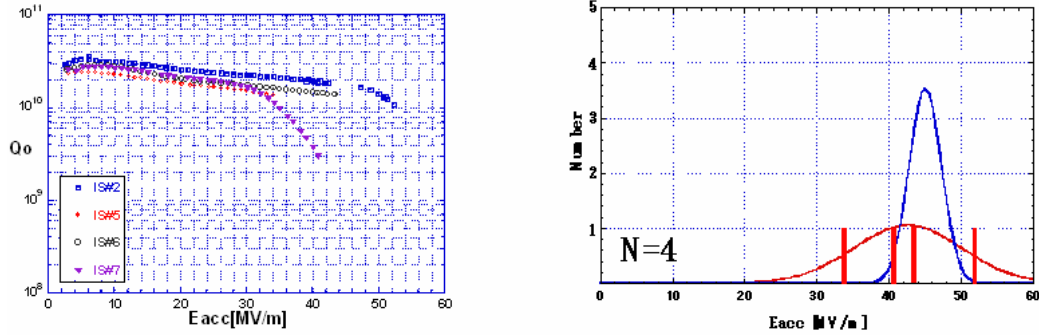


Figure 8: Results of  $H_2O_2$  rinsing

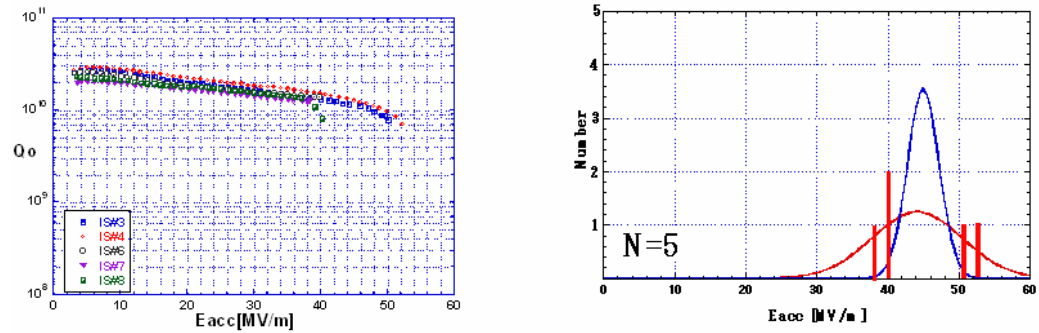


Figure 9: Results of Degreasing

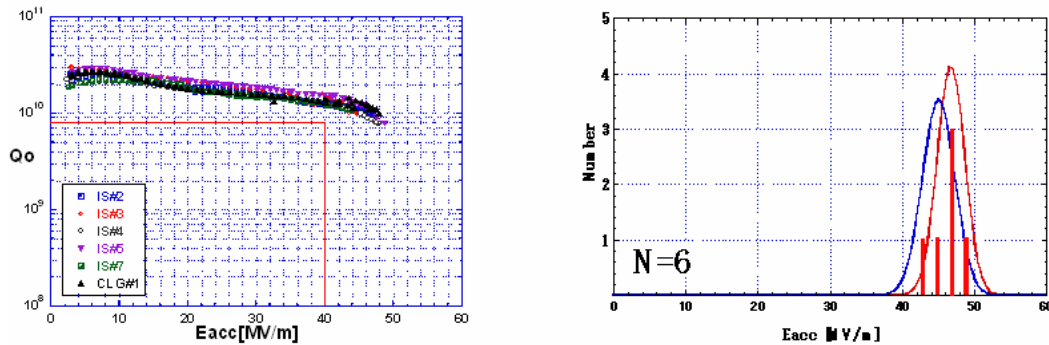


Figure 10: Results of Flash EP (3 $\mu$ m)

The 3  $\mu$ m flash EP just after the EP (20  $\mu$ m) dose not remain the contamination on the SRF-niobium surface, so such a high accept rate could be gained. The 3  $\mu$ m flash EP has no effect on reducing MP, so we can conclude that the source of gradient scatter and MP are different mechanism. Figure 11 shows the material removal dependence of scatter for each recipe. It should be

emphasized that the flash EP always reduces the gradient scatter and the effect is the larger in the smaller material EP removal.

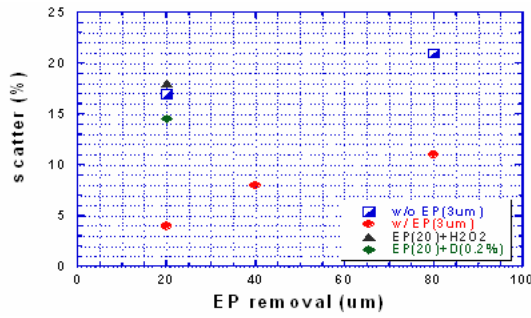


Figure 11: Material removal dependence of the scatter

### CONTAMINATIONS MECHANISM

It is clear that the contaminations produce scatters diffuses into the niobium bulk during EP process. HPR can remove partially contamination. Additional rinsing is also not so effective to remove the contamination. We need a flash EP to take away the source of scatter. Here we propose one model for the contamination mechanism [7]. Sulphur contamination happens during EP process. Some of its might exist as particle and react with niobium, then stays as niobium-sulphide:  $Nb_xS_y$ . This  $Nb_xS_y$  is the source of scatter. Additional rinsing like  $H_2O_2$  rinsing or degreasing cannot remove  $Nb_xS_y$ . HPR also cannot remove it. Only the flash EP can remove the  $Nb_xS_y$ .

### MULTIPACTING

Multipacting (MP) free surface is also needed, because MP sometimes brings another problems: long processing time, triggers serious FE, and so on. In these series of tests, we measured and analysed MP processing for each recipe. We judged MP by X-ray appearance, which suddenly happened at the presumed gradient by MP simulation. Figure 12 shows the MP region in cold test and simulation. Y. Morozumi made this simulation [8]. In his simulation, MP region spreads from 19 to 40 MV/m. In our cold test results, typical MP region was 18 to 26 MV/m and localized. It can be processed out within 10 min. MP process has a cleaning effect on the SRF surface by the electron bombardments, so the secondary emission coefficient lowers and MP disappears at the  $E_{acc} > 26$

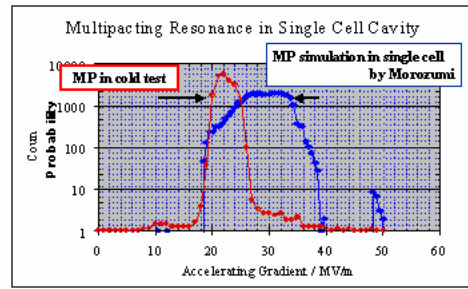


Figure 12: MP resonance in cold test and simulation for IS cavity

MV/m.

Figure 13 shows the MP region in cold test on each recipe. We normalized the MP occurrences by the number of cavities.  $H_2O_2$  rinsing and degreasing reduces MP. The both rinsing methods dissolve and eliminate sulphur. MP seed could be sulphur contamination. Figure 14 shows the probabilities of X-ray appearance after MP processing on each recipe.  $H_2O_2$  rinsing and degreasing shows fewer probabilities compared with another recipes. So, dissolving sulphur is effective to reduce X-ray after processing.

### PERSPECTIVE FOR X-RAY FREE CAVITY

We used a plunger pump for our HPR system. But recent results showed that this pump made contaminations after a long-term operation (6-10 hr). These contaminations brought FE, but these could be recovered by additional short HPR (1 hr). We analysed the contaminations from pump and found it was silicon of the silicon grease at plunger O-ring sealing. We replaced this pump to a diaphragm pump. After that we tested 5 cavities on this new pump. 3 of 5 cavities were treated with the recipe: Degreasing + HPR. Another 2 cavities were brand-new, so the recipe: CBP + CP + AN + EP + flash EP + Degreasing + HPR + Baking, was applied to them.

Figure 15 shows the results of MP and X-ray monitoring. 4 cavities showed MP free, and only one cavity showed some MP, but it was very light. About X-ray, 2 cavities showed X-ray free. So the series tests

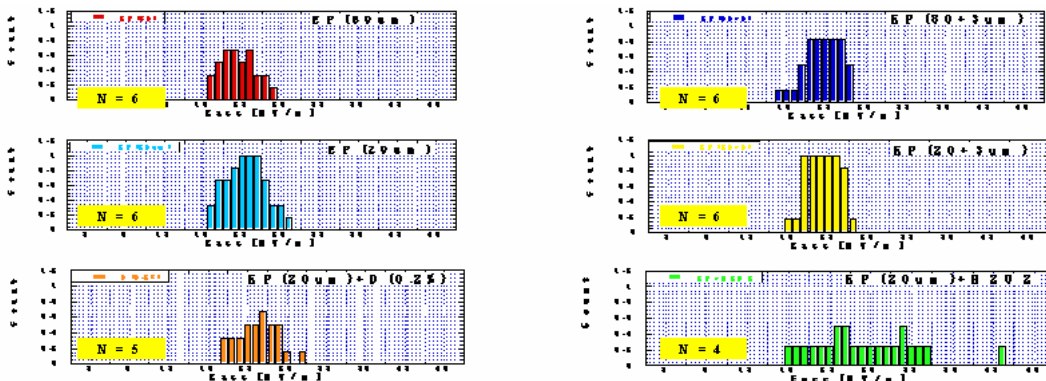


Figure 13: MP region in vertical test for each recipe

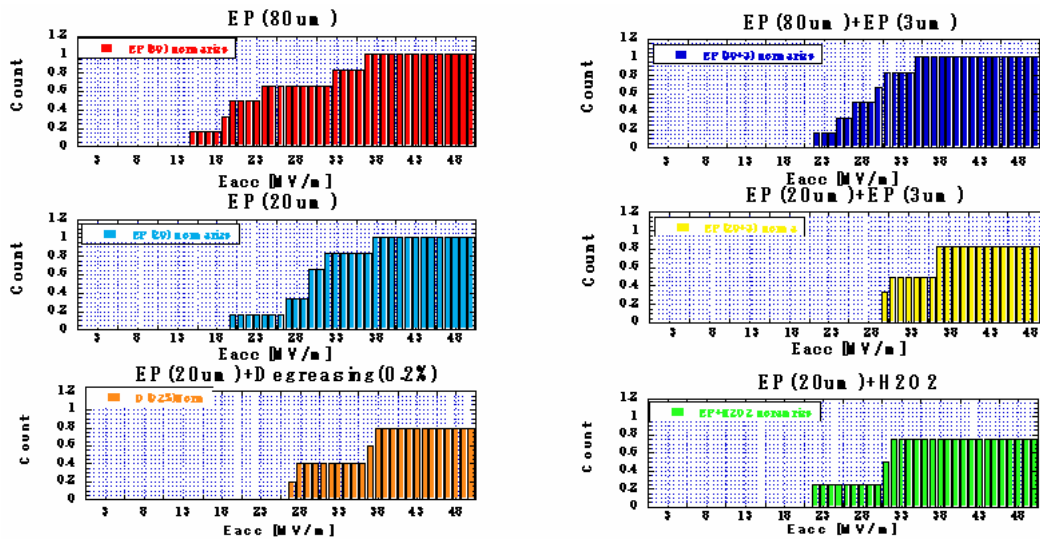


Figure 14: Probability of X-ray appearance for each recipe

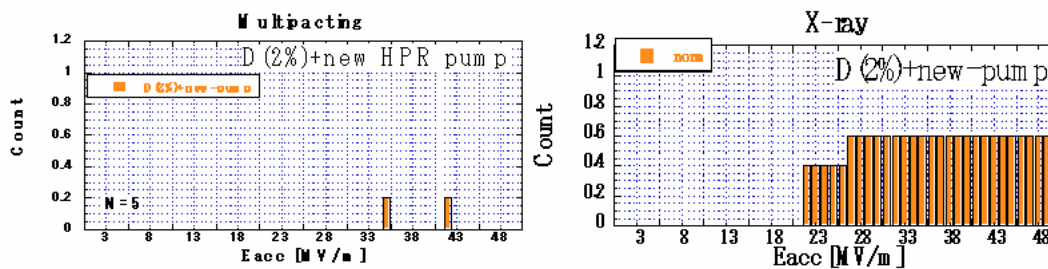


Figure 15: recent MP and X-ray results with new diaphragm HPR pump

mentioned above include MP and X-ray influence by the old pump contaminations. We estimated the real probability X-ray appearance by recipe itself. Figure 16 shows the re-evaluated probability of X-ray appearance. From these results, we can expect that MP/X-ray free cavity could be feasible.

## SUMMARY

After proof of principle 50 MV/m by single cell cavities [9], we have concentrated to solve the scattering problems of cavity performance. We have found that scattering of cavity performance comes from sulphur contamination produced in EP process and cannot be removed by only HPR. We conclude it exists underneath of the SRF niobium surface as niobium sulfide ( $Nb_xS_y$ ). Scattering might relate to  $Nb_xS_y$  contamination. It can be removed only by “flashing” like additional  $3\mu m$  EP. “Rinsing” was not enough to remove it. The contamination also depends on EP material removals. After a heavy EP removal, the flash EP of  $3\mu m$  is not enough to get narrow scatter. Current best recipe at KEK is: CBP + CP + AN + EP (80  $\mu m$ ) + EP (20  $\mu m$ ) + Flash EP+ HPR + Baking. This recipe can satisfy the ACD accept goal. From the MP analysis, the source of MP and scattering were different mechanism. Degreasing and  $H_2O_2$  rinsing can reduce MP/FE. Both dissolve sulphur, so the source of MP is reduced.

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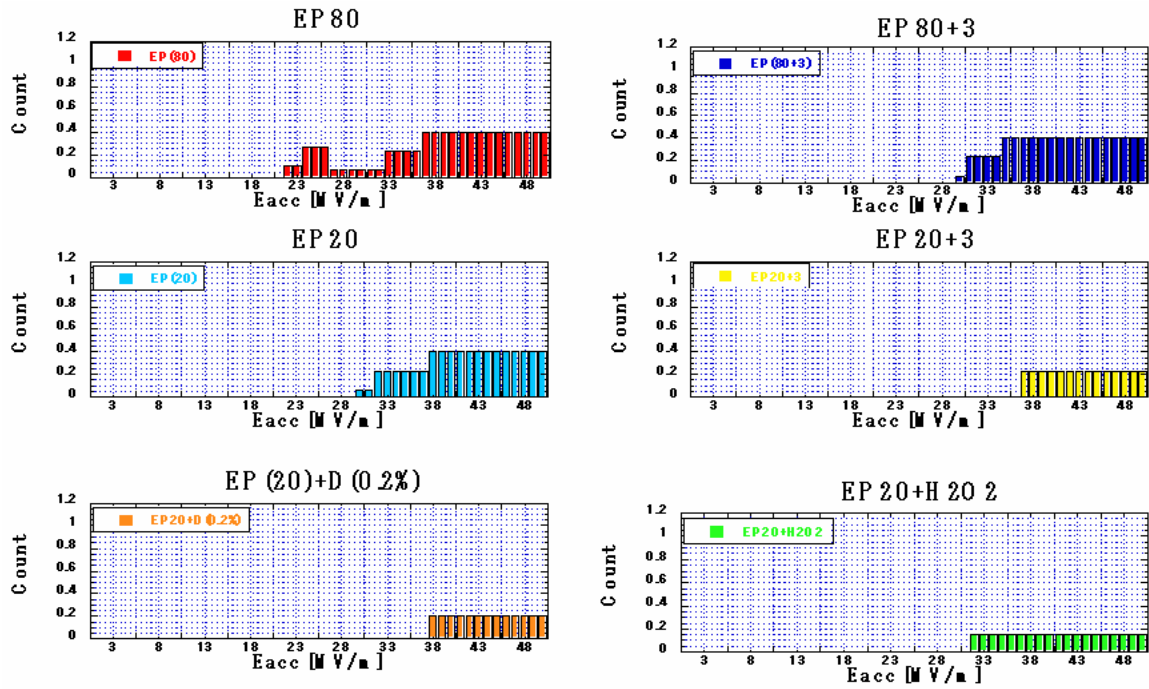


Figure 16: Re-evaluated probability of X-ray appearance