

Recent Developments in SRF Cavity Cleaning Techniques at KEK

K.SAITO[#]

High Energy Accelerator Research Organization (KEK), Accelerator Lab
1-1 Oho, Tsukuba-shi, Ibaraki-ken, 305-0801 Japan

Abstract

To date, KEK has developed cleaning techniques for superconducting niobium RF cavities. These include: electro-polishing (EP), buffered chemical polishing (BCP), barrel polishing (BP), high pressure rinsing (HPR), megasonic hot water rinsing, degassing water, ozonated water cleaning, waste water treatment, and so on. In this paper, we present findings from electro-polishing of cavities, which was investigated during the previous two years, and from several newly developed water cleaning technologies.

1 INTRODUCTION

KEK has developed many cleaning technologies related to surface preparation of niobium superconducting RF cavities. This development encompasses a variety of approaches from cavity surface cleaning technologies including: electro-polishing [1], chemical polishing [2], barrel polishing [3], high pressure water rinsing [4], megasonic water rinsing and so on, to the water cleaning methods including: high concentration ozonated water [5], degassing of water [6] and waste water treatment, etc.

Among these cleaning technologies, electro-polishing is still being investigated very intensively [7, 8]. During the previous two years, we have achieved a high gradient performance of 40 MV/m with five additional 1300 MHz single cell cavities. Two 3-cell cavities have achieved a gradient > 30 MV/m since the last SRF workshop in Abano. There is no doubt about the impact of electro-polishing on achieving high gradient SC performance. Furthermore from the cavity assembly point of view, the impact of a long-term air exposure on high gradient performance was studied.

An ozonated water rinsing method has successfully developed at KEK for KEK-B superconducting cavities by K.Asano et al. [9]. We tested this cleaning method on our high gradient cavities to see if further improvements could be achieved. To implement this technology, we developed a new method to make highly concentrated ozone gas (100%) and ozonated water (150 ppm). This technology has not yet been applied to cavity preparation but it might have a promising future, if not in our field in other fields such as the semiconductor industry.

We have successfully used degassed water to achieve cost effective maintenance of ultrapure water producing systems. Bacteria contamination problems in the system are easily resolved by this technique. In addition, for the chemical polishing of niobium cavities, we recently developed a new wastewater treatment method, which uses an electrolytic treatment with Coca-Cola or steel cans. This method might also be applicable for water contamination problems in the plating and other industries.

In this paper we present our experiences with electro-polishing and new water cleaning techniques which have been obtained since the last SRF workshop.

2 ELECTRO-POLISHING

The horizontally rotated, continuous, electro-polishing method was developed at KEK as part of the TRISTAN SC project [1]. This technology was developed to solve two problems associated with standard electro-polishing approach [10]: 1) insufficient current density at equator section of a cavity and 2) insufficient venting of hydrogen gas developed by the process. The original idea of electro-polishing niobium axi-symmetric bodies came from SIMENS [11], but the major change made to their approach is that of continuous electro-polishing. The SIMENS method considers current oscillation as an important electro-polishing condition [12]. Continuous electro-polishing regards a current density as much as 30 - 100 mA/cm² as the most important condition. This method makes electro-polishing much simple. It also allows for easier quality control.

2.1 High gradient performance; $E_{acc} \sim 40$ MV/m achieved by electro-polishing

After the last SRF workshop, we continue to investigate achieving high gradients in niobium SC cavities with electro-polished, 1300 MHz, niobium single cell cavities. With five more cavities we reproduced the high gradient of 40 MV/m without any Q-degradation as shown in Figure 1 [7]. To achieving these results, the procedure for cavity preparation included a chemical polishing (CP) and an electro-polishing of the cavity, but did not include any heat treatment. This procedure was applied to several other cavities. The high gradient performance; $E_{acc} > 30$ MV/m was reproduced [13].

[#] E-mail: ksaito@mail.kek.jp

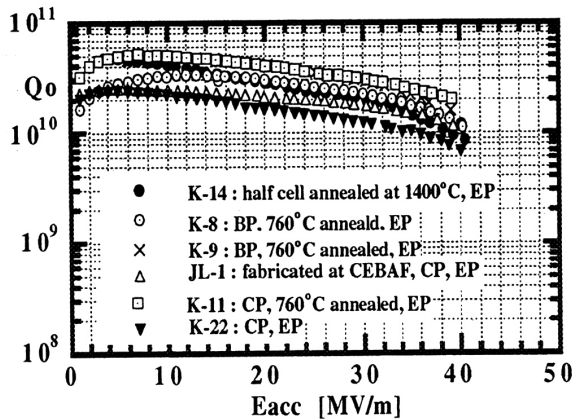


Figure 1: High gradient performance with electro-polished 1300 MHz niobium bulk single cell cavities

2.2 Elimination of Heat Treatment from Cavity Preparation

The above results forced us to reconsider the requirement for heat treatment after electro-polishing, which has been commonly accepted. Using a virgin, 1300 MHz, niobium cavity manufactured from Tokyo Denkai material with RRR=200, we removed material from the cavity surface in steps of 40 μ m using electro-polishing until 240 μ m of material was removed. No heat treatment was done during this experimental series. To test for hydrogen Q-disease after each material removal step, a set of measurements was done: a cold test after a fast cool down, and a further cold measurement after exposing the cavity to 100K for one night. In these measurements, the temperature dependence of surface resistance was carefully measured and the residual surface resistance (Rres) was evaluated. A difference in Rres was noted after each step, for both the fast cooldown and the over night exposure. The high gradient performance (Eacc,max) was also compared in both tests. In Figure 2, the test results are presented. No additional increase in Rres resulting from 100 K exposure is observed after any material removal step. Eacc,max was also not effected by the 100K exposure. Hydrogen Q-disease was not observed after any material removal step.

Two reasons are advanced for why the hydrogen Q-disease does not occur. One is that the cavity has no mechanical grinding before electro-polishing. The other reason is that in the horizontal rotating, continuous electro-polishing method, hydrogen gas easily escapes. The detail discussion is described in reference [14].

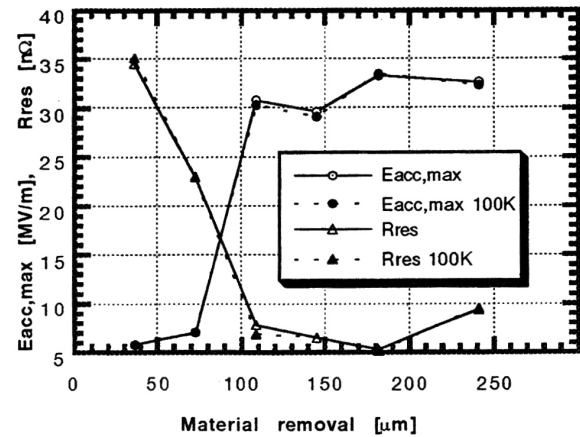


Figure 2: Changes in Eacc,max and Rres after fast cool down and after exposure at 100 K for one night after successive material removal steps of 40 μ m over the range 30- 240 μ m.

2.3 High gradient performance in multi-cell cavities

So far, the high gradient performance achieved with electro-polishing has only been obtained with 1300 MHz, single cell, niobium cavities. The superiority of electro-polishing should be confirmed in multi-cell structures. We applied electro-polishing to three 3-cell cavities and to one 9-cell cavity. The two 3-cell cavities have achieved Eacc > 30 MV/m. One 3-cell cavity was limited to 27 MV/m. The 9-cell cavity was limited to 22 MV/m by multipacting. We have already reported that multipacting was observed in electro-polished, single cell cavities [16]. It is processed out in a rather short time with single cell cavities, but the process time is longer in multi-cell cavities. Recently we noticed that a higher temperature bake-out; e.g. 140°C, is very effective at suppressing multipacting [15].

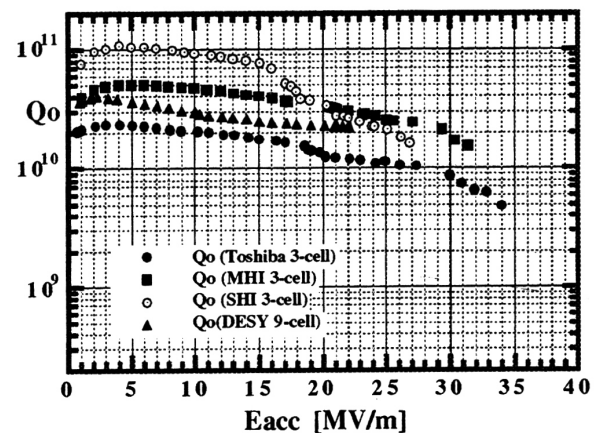


Figure 3: High gradient performance with electro-polished 1300 MHz, multi-cell, niobium bulk cavities (three 3-cell cavities and one 9-cell cavity).

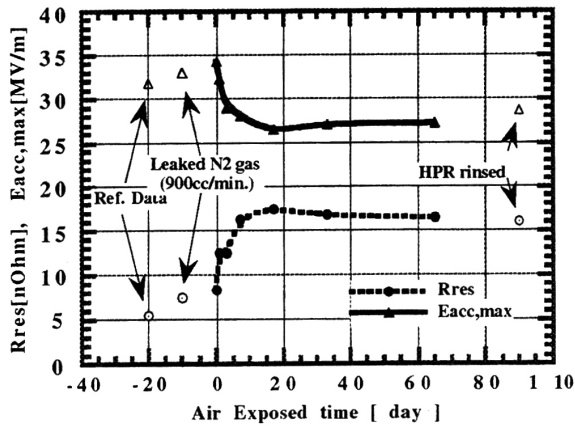


Figure 4: Air exposure effect on the high gradient performance of electro-polished cavities.

2.4 Air exposure effect on the electro-polished surface

From a quality control point of view we investigated the air exposure effect on the electro-polished surface [17]. A cavity was electro-polished, removing 30 μm of material, and rinsed with HPR. After confirming a baseline performance, it was exposed to the air vented through a 0.01 μm filter. The air exposing was done for 45 minutes, and for 1, 3, 7, 13, 33 and 65 days. The results are presented in Figure 4. The Rres increased about 10 n Ω after being exposed for one week, but did not change after further air exposure. The superconducting parameter band gap was slightly degraded by 0.05% after a day. The attainable gradient was reduced from 34 MV/m to 27 MV/m by multipacting after one-week exposure. The gradient degradation also did not change after one-week exposure.

3 OZONATED WATER RINSING

3.1 Ozonated water rinsing effect on high gradient

Multipacting is often observed in our cavities. A potential source of multipacting is hydrocarbon contamination on the surface. Ozonated water rinsing is effective at removing such a contaminant. Ozonated ultrapure water rinsing (OUWR) has been successfully applied at KEK with 508 MHz, KEK-B, superconducting niobium cavities [9]. We tried applying this cleaning method to a 1300 MHz, single cell cavity. The results are shown in Figure 5. The baseline performance (O) achieved with electro-polishing (EP:30 μm) and high pressure rinsing (HPR) was confirmed before OUWR. After this measurement, a further 30 μm was removed from the cavity with EP, after which it was rinsed by OUWR. No HPR rinsing was done. The ozone concentration in the water was 3 - 4 ppm, which was the same as for the KEK-B cavities. As seen in Figure 5 (●) the cavity

performance was degraded. Even with OUWR, multipacting appeared at around 15 MV/m. Field emissions started at 18 MV/m. Since no HPR was performed, particle contamination was suspected as the cause of this field emission. The cavity was again electro-polished (30 μm), and then rinsed in turn with OUWR and HPR. The performance was worse (θ) A Q-degradation appeared. No x-rays were observed in connection with the Q-degradation. Furthermore, we changed the rinsing order after EP but the results were even worse (π). In this case also, x-rays were not observed in association with the Q-degradation. Finally we treated this cavity just with HPR, and achieved even poorer performance (x). Thus OUWR did not contribute to the suppression of multipacting. It has to be emphasized that a combination of OUWR and HPR results in a serious Q-degradation with high gradient cavities.

3.2 A method of high density ozonated water

We have developed a method to make a highly concentrated ozonated water [5]. Significant ozone gas can be dissolved in water. The solubility limit is about 400 ppm. To make highly concentrated ozonated water, a key is in how to make the highly concentrated ozone gas. The boiling point of ozone is 161 K, which is much different from that of oxygen (90.2K) or nitrogen (77.3K).

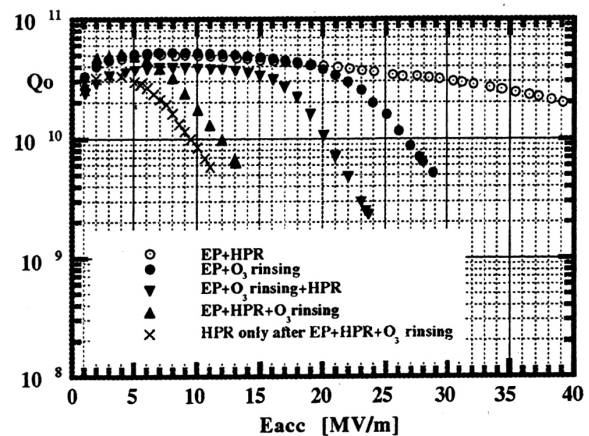


Figure 5: Ozonated water rinsing effect on high gradient cavities

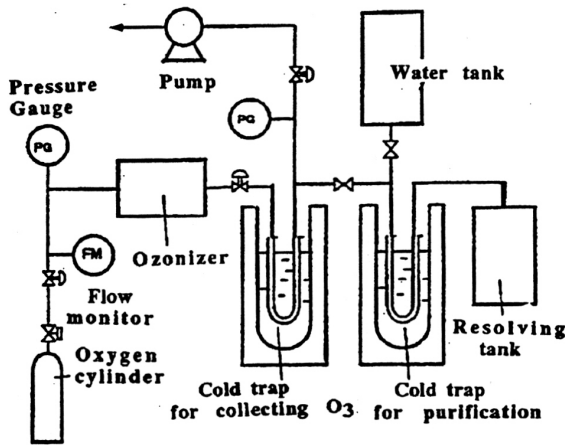


Figure 6: Ozone ice making system

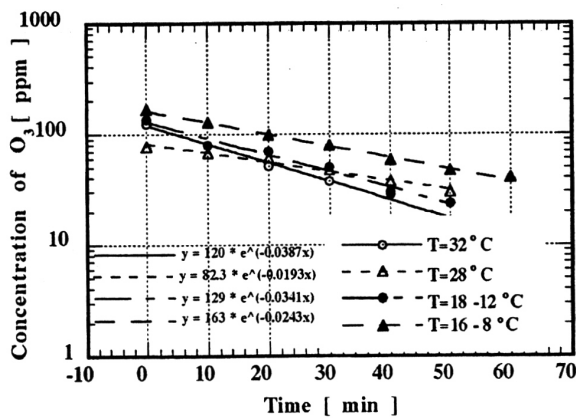


Figure 7: Decay of the highly concentrated ozonated water

Conventional ozonizer makes ozone gas by Corona discharge in oxygen gas but the efficiency is only a few percents. Taking advantage of the different boiling temperature characteristics, if one cools down the mixed gas of oxygen and ozone from the ozonizer to a temperature between 161 K and 90.2 K, liquid ozone or ozone ice will be generated. After separating the liquid or ice from the remaining gas, one warms it up to a temperature over 161 K, producing a very pure ozone gas. Dissolving this gas in water results in highly concentrated ozonated water. Applying this concept, we made an experimental system to produce ozone ice. The system is presented in Figure 6. Using this experimental set up, we produced highly concentrated, ozonated water (150 ppm). The highly concentrated ozonated water decays in a rather short time; e.g. in 40 minutes depending on the water temperature. The measurement of the decay time is shown in Figure 7.

The ozone concentration in the water produced by today's commercially available systems is 30 ppm maximum. The concentrations produced by KEK are

higher by 5 times. This method can also produce very pure ozone gas. We are investigating the decay time of the ozone ice. A much longer time is expected than for the highly concentrated ozone water because the temperature is so low. If ozone ice is stable, it can be delivered to users. This method could be used for in-situ ozone gas cleaning of the vacuum systems for accelerators. Furthermore, it will be very useful to the semiconductor technology or for sterilizing techniques. Even the ozone hole might be repaired by this method.

4 WATER CLEANING TECHNOLOGIES

4.1 Degassing of water

The propagation of bacteria is a problem for TOC (total organic carbon) in ultrapure water. To lower TOC levels, one has to install an ultraviolet sterilizer in the ultrapure water system. However, even if this is done, the filters must be changed frequently due to stacking of killed bacteria in the filters. This makes the maintenance cost very expensive. We have invented an easier maintenance method to prevent bacteria propagation [6]. If one removes the dissolved oxygen in the water, bacteria will be killed and there is no way to live in the system. The principle of the degassing of water is presented in Figure 8. Today such a system is commercially available. We installed a degassing system in our ultrapure water making system. We investigated the relationship between the operation time and the number of bacteria alive in the ultrapure water or TOC level. Figure 9 shows the result of this sterilization effect. The number of bacteria was reduced from 200 pieces/ml to a few pieces/ml in a 30-minute operation. TOC level was also lowered gradually. The final level reached, 80 ppb, and was very stable [6]. This method has not yet been used for the cavity preparation but it is expected to be very useful.

4.2 Waste water treatment using electrolytic treatment

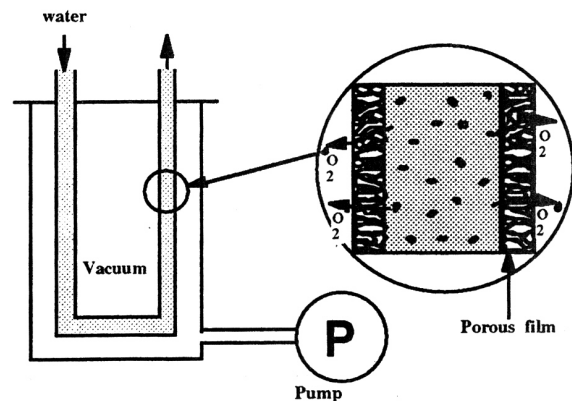


Figure 8: Principle of degassing water

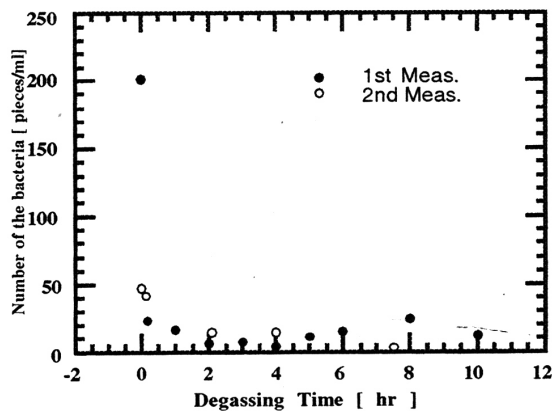


Figure 9: Sterilizing effect of degassing water

We have a problem with the treatment of the wastewater resulting from BCP acid treatment of niobium cavities. The concentration of dissolved chemicals in the wastewater, which include fluorine (F), phosphorus (P) and nitrogen (N), will be severely controlled by the upgraded environment policy in Japan. Presently, the allowed concentrations are 16 ppm for P, 15 ppm for F, and 60 ppm for N. In the near future the number for F will be lowered by a factor of two. With N and P, the total amount permitted has yet to be announced. We are evaluating a new water treatment technology in order to meet the required levels [18]. An electrolytic treatment is considered a good cleaning technology for the wastewater. We tested this method using Coca-Cola cans (aluminum) and steel cans. They were cut and put in an electrolytic tank with the wastewater. For this method, the fundamental principle is electrolysis of water. When applying a voltage to the bath with the immersed cans, aluminum or steel chips that are weakly connected to the anode are dissolved into the water. Many contaminants are absorbed by the aluminum or iron ions. After the reaction, the water is adjusted in pH to about 8, and the contaminants are precipitated with hydroxides.

This electrolytic treatment method significantly reduces the concentration of F, P, and N. The results are presented in Table 1. In this method, the initial concentration of 220 ppm (P), 180 ppm (F) and 120 ppm (N) was lowered to 7.3 (P), 24 (F) and 47(N), respectively. These numbers are still too high when compared to the future environment standard values. However, when an RO (reverse osmosis) film filters the treated water, the concentrations are further reduced as shown in Table 1. The resultant water includes fewer chemicals and less particle contamination. This water can be reused for cavity rinsing. The concentration of N is still high compared to a city water level, but it is possible to lower this concentration by repeating this procedure. This method is potentially applicable for contaminated water

produced by commercial concerns such as plating companies. Since we reuse the Coca-Cola or steel cans, the operating cost is reasonably low if a proper RO film is selected.

Table 1: Impurities in the treated water

	P [ppm]	F [ppm]	N [ppm]	Electrical conductivity [μ s]
waste water	220	180	120	3000
waste water by ET	7.3	24	47	640
reduction rate	96.7%	86.7%	60.8%	
filtered water by ROM after the ET	0.2	3	19	140
reduction rate from original wastewater	99.9%	98.3%	84.2%	
city water (Kumagai city)	0.1 >	1 >	4.6 >	300

ET: Electrolytic treatment, ROM: Reverse osmosis membrane

5 SUMMARY

Several findings with electro-polishing are described. The positive impact of electro-polishing on high gradient performance of SC cavities has been established. The heat treatment for removal of hydrogen from the cavity, which was considered to be inevitable, is no longer needed after electro-polishing.

The electro-polished surfaces are degraded by air exposure over a period of one week. Residual surface resistance increases about 10 n Ω . The attainable field gradient is limited by multipacting. Air exposure has to be eliminated during cavity assembly.

Ozonated water rinsing does not seem to suppress multipacting. Combining ozonated water rinsing and high pressure rinsing results in a serious Q-degradation for high gradient cavities.

We introduced several new water cleaning technologies for application in our field. Degassing of water is very effective in reducing bacteria propagation in ultrapure water systems. The new technologies: the ozone ice maker and the electrolytic treatment method are also very useful for treating water contaminated with bacteria and chemicals. These techniques could open a new water cleaning culture.

6 ACKNOWLEDGMENTS

The author would like to thank to Mr. K. Sasaki from Nikkyo Technos Co. Ltd. for his help with the degassing system. He would like to express thanks to Mr. H. Haejima and M. Nagakura from Cosmo engineering Co. Ltd. for support in making the ozone

ice maker and other experiments. A special thank is due to Prof. M. Kihara, director of the accelerator lab at KEK for his continuous encouragement and support for our activities.

method combined electrode reaction and filtering with reverse osmosis membrane, " Proc. of the 2nd Superconducting Linear Accelerator Meeting in Japan, May 27 - 28, 1999, KEK, Tsukuba, Japan, in Japanese, to be published.

7 REFERENCES

- [1] K. Saito et al., " R&D of Superconducting Cavities at KEK, " Proc. of the 4th Workshop on Superconductivity, August 14 - 18, 1989, KEK, Tsukuba, Japan, p. 635.
- [2] T.Higuchi et al., in this workshop
- [3] T. Higuchi et al., " Investigation on Barrel Polishing for Superconducting Niobium Cavities, " Proc. of the 7th Workshop on Superconductivity, October 17 - 20, 1995, Saclay, Gif sur Yvette, France, p. 723.
- [4] K.Saito et al., " Study of Ultra-clean Surface for Niobium SC Cavities, " Proc. of the 6th Workshop on Superconductivity, October 4 - 8, 1993, CEBAF, Newport News, USA, p. 1151 - 1159.
- [5] H. Haejima and K.Saito, " A Production Method of Highly Concentrated (150ppm) Ozonated Water," Proc. of the 1st Superconducting Linear Accelerator Meeting in Japan, May 28 -29, 1998, KEK, Tsukuba, Japan, 104 - 107, in Japanese.
- [6] K.Saito et al., " Degassing Effect in Water - Sterilizing Effect -, " Proc. of the 8th Workshop on Superconductivity, October 6 - 10, 1997, INFN-LNL, Abano Terme, Italy, p.655- 661.
- [7] K.Saito et al., " High Gradient Performance by Electro-polishing with 1300 MHz Single and Multi-cell Niobium Superconducting Cavities, " in this workshop.
- [8] E.Kako et al., in this workshop
- [9] K. Asano et al., " Stable Performance of 508 MHz Superconducting RF Cavities for KEK B-Factory, " Proc. of the 7th Workshop on RF Superconductivity CEA-Saclay, Gif sur Yvette, France , October 17 - 20, 1995, p.419 - 423.
- [10] Y.Kojima et al., Jpn. J. Appl. Phys. 21(1982). p.86.
- [11] A. Roth et al., " Method for the electrolytic polishing of the inside surface hollow niobium bodies, " in US patent 4,014,765, Mar. 29. 1977.
- [12] H. Diepers et al., Phys. Lett., 37A(1971), p.27.
- [14] K.Saito et al., " Hydrogen Q-disease Free Preparation without Annealing for Superconducting Niobium RF Cavities, " Proc. of the 2nd Superconducting Linear Accelerator Meeting in Japan, May 27 - 28, 1999, KEK, Tsukuba, Japan, in Japanese, to be published.
- [15] K.Saito and P. Kneisel, "Temperature dependence of the Surface Resistance of Niobium at 1300 MHz - Comparison to BCS Theory -, " in this workshop.
- [16] K.Saito et al., " Water rinsing of the contaminated superconducting RF cavities," Proc. of the 7th Workshop on Superconductivity, October 17 - 20, 1995, Gif sur Yvette, France, p/379.
- [17] K.Saito et al., " Long Term Air Exposure Effect on the Electropolished Surface of Niobium Superconducting RF Cavity, " in this workshop.
- [18] H. Haejima and K. Saito " New waste water treatment