Long Term Air Exposure Effect on the Electropolished Surface of Niobium Superconducting RF Cavity

K. Saito#, E.Kako, T.Shishido, S.Noguchi, M.Ono, and Y.Yamazaki
Accelerator Research Organization (KEK), Accelerator Lab,
1-1, Oho Tsukuba-shi, Ibaraki-ken, 305-0801 Japan

Abstract

From QA issue of superconducting RF cavities, it is important to investigate the air exposure effect of the clean niobium surface. In our class 1000 clean room, we have carefully introduced filtered air into a 1300 MHz single cell niobium cavity after the baseline test and exposed the surface to the air for totally 65 days. On the way, the cavity was tested at the exposure time: 45 minutes, 1 , 3, 7, 17, 33 and 65 days. An increased residual surface resistance about 10nΩ was observed after one week exposure, then it was saturated for further air exposures. The maximum field gradient was also degraded from 34 MV/m to 27 MV/m after the 3 day's exposure and limited by multipacting. This contribution presents the air exposure effect on the high gradient cavity performance.

1 INTRODUCTION

Quality control of the superconducting (sc) RF cavities is a very important issue in this field. The performance of sc cavities is very sensitive to surface contamination. Cavity assembly environment effects much on the performance. The horizontal assembly which is a work to assemble sc cavities into horizontal cryostats contains many procedures to degrade the performance. In the TRISTAN horizontal assembly, some performance degradation was observed [1]. The reason is not yet understood. We started to investigate the performance degradation. In this time, we studied the air exposure effect on the high gradient performance by electropolishing. This effect has been worried for a long time because the cavity performance is so sensitive to the surface condition. In this experiment we observed the performance degradation which the residual surface resistance increases about 10 nΩ and the high gradient is reduced from 34 MV/m to 27 MV/m by the air exposure for one week. The air exposure promotes multipacting seriously. The air exposure period of one week is too long for a real horizontal assembly work. However, one has to remind the effect which the field gradient is limited by multipacting.

2 EXPERIMENTS

One electropolished 1300 MHz single cell niobium bulk cavity : K-11 manufactured with RRR=200 niobium material from Tokyo Denkai was used in this experiment. To investigate problems in the TRISTAN horizontal assembly procedure, we followed the procedure. At first we treated the cavity with electropolishing and high pressure water rinsing (HPR). Then we confirmed the baseline cavity performance : Eacc,max=32 MV/m and no field emission. Then pure nitrogen from a cold evaporator was carefully vented into the cavity without disassembling. The nitrogen/air venting system is shown in Figure 1. The nitrogen/air was vented through a 0.01μm filter at a flow rate of 900 cc/min. This gas venting method does not introduce particle contamination into cavities [2]. The cavity volume is a 4 l and is filled in 10 minutes up to one atom. Then, the inner surface was exposed the nitrogen/air for a period. After that, the cavity is vacuum evacuated quickly with a 85^{o}C baking up to ~ 1 x 10^{-7} torr by an evacuation system consisted of a turbo-molecular pump and rotary pump, then evacuation is switched to ion pump. The final vacuum pressure is ~ 1 x 10^{-9} torr. The cavity was sealed off with a metal valve (V6), and fixed a vertical test stand, then cooled down. This procedure was repeated for every air exposure experiment. Air exposure tests were carried out at the exposing times for 45 minutes, 1,3,7,17,33 days and totally 65 days. The air in the class 1000 clean was vented into the cavity through a 0.01 μm filter without any controlling moisture.

In the vertical test, at first a temperature dependence of the surface resistance(Rs) was carefully measured from 4.2K to 1.5 K and it was fitted by the following formula to evaluated parameters : A, $\alpha=\Delta/k_B$, and the residual surface resistance (Rres) ;

$$Rs(T) = \frac{A}{T}\cdot \exp(-\frac{\Delta}{k_B T}) + Rres \qquad (1).$$

After this measurement, Qo-Eacc curve was measured at

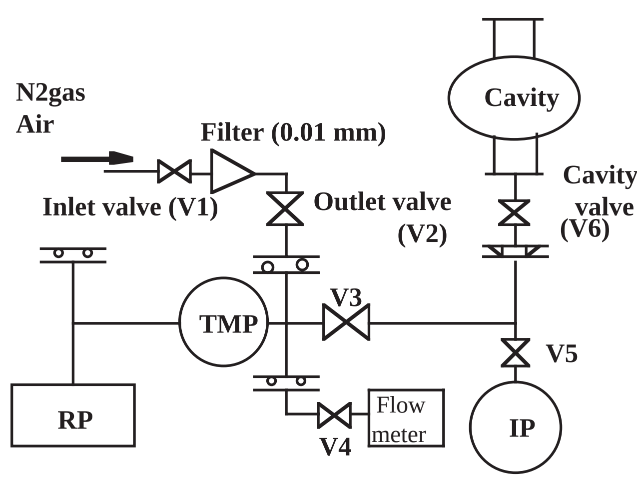

Figure 1 : Nitrogen/Air venting system

E-mail : ksaito@kek.mail.jp

the lowest temperature. After this series test of the airexposures, the cavity was disassembled and treated with HPR and cold tested again to see the high pressure water rinsing effect on the air exposed.

3 RESULTS

3.1 Effect on RF properties

The fitting results of Rs(T) by the formula (1) are summarized in Figure 2 to Figure 4. Figure 2 shows the air exposure effect on the parameter A. This parameter is not influenced by the air exposure within a measurement accuracy. Recently it was found out this parameter is changed by a 140^{o}C baking due to oxygen diffusion [3]. In our vacuum evacuation procedure, 85^{o}C baking takes place but the effect is not observed yet because the temperature is still low. On the other hand, as shown in Figure 3, a small degradation is observed in the band gap ($\alpha=\Delta/k_B$) by the air exposure. The evaluated degradation rate is;

$$\frac{d\alpha}{\alpha} = -0.046\% / day. \qquad (2).$$

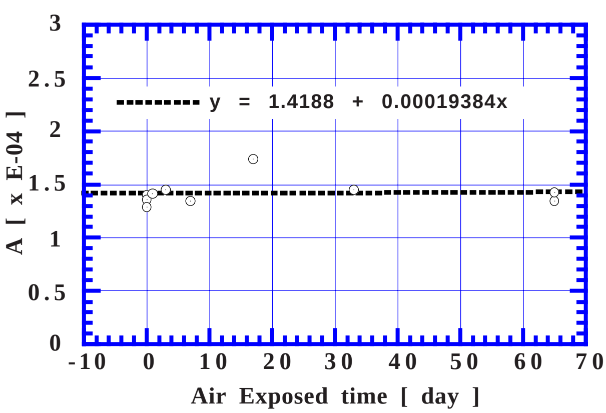

Figure 2 : Air exposure effect on the parameter A

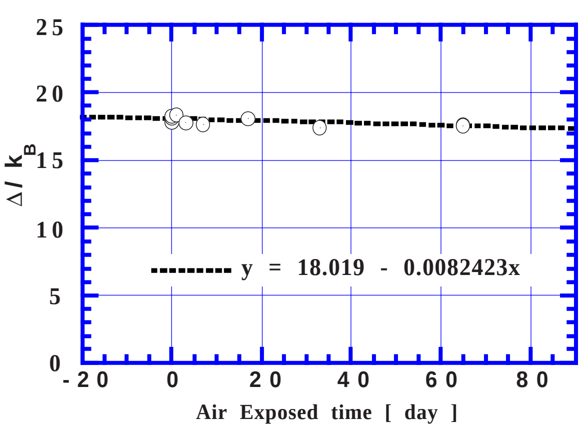

Figure 3: Air exposure effect on the band gap

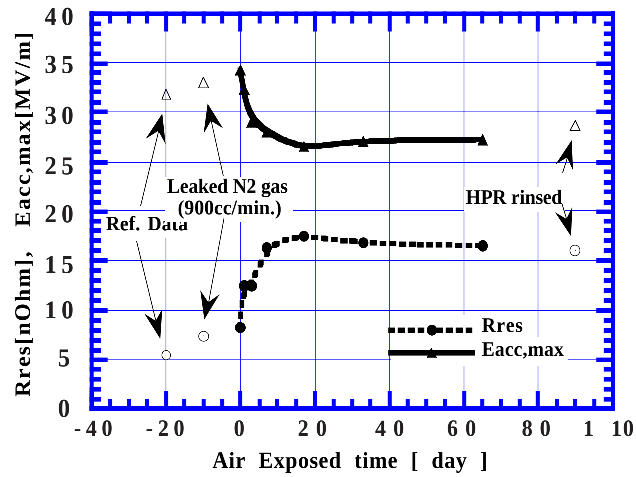

Figure 4: Air exposure effect on residual surface resistance and field gradient

The reference data , N2 gas vented data and the high pressure water rinsed data are intentionally shifted with the time to get an easier view.

Air exposure increases a residual surface resistance as presented in Figure 4 (●). An increase of about 10 nΩ was observed by the first one week air exposure and it saturated with further air exposures.

3.2 Effect on high gradient

The air exposure effect on the high gradient is summarized in Figure 4 (▲). All the Qo-Eacc curves in these measurements are presented in Figure 5. The corresponding 1/Qo vs. Eacc^2 curves are presented in Figure 6. Figure 7 and Figure 8 present the x-ray occurrence during RF processing and after the RF processing respectively.

Similarly to Rres, the gradient was degraded from 34 MV/m to 27 MV/m in the first one week air exposure. This degradation saturated for the further air exposures. As seen in Figure 6 and Figure 7, multipacting (MP) levels are observed at 15 - 18 MV/m and at 23-30 MV/m. The lower MP level disappears by RF processing, but the higher MP level can not be eliminated by RF processing. The multipacting in the higher level becomes more serious with increased air exposing time, and limits the gradient at 27 MV/m by the air exposure for one week. After that, the field degradation saturates with further long air exposures. After the 65 day's air exposure, we took HPR this cavity but the cavity performance was not improved. HPR can not remove the degradation.

4 DISCUSSION

4.1 Increased residual surface resistance

The additional Rres about 10 nΩ was observed by one week air exposure, and saturated with the further air exposures. This degradation should be related to surface

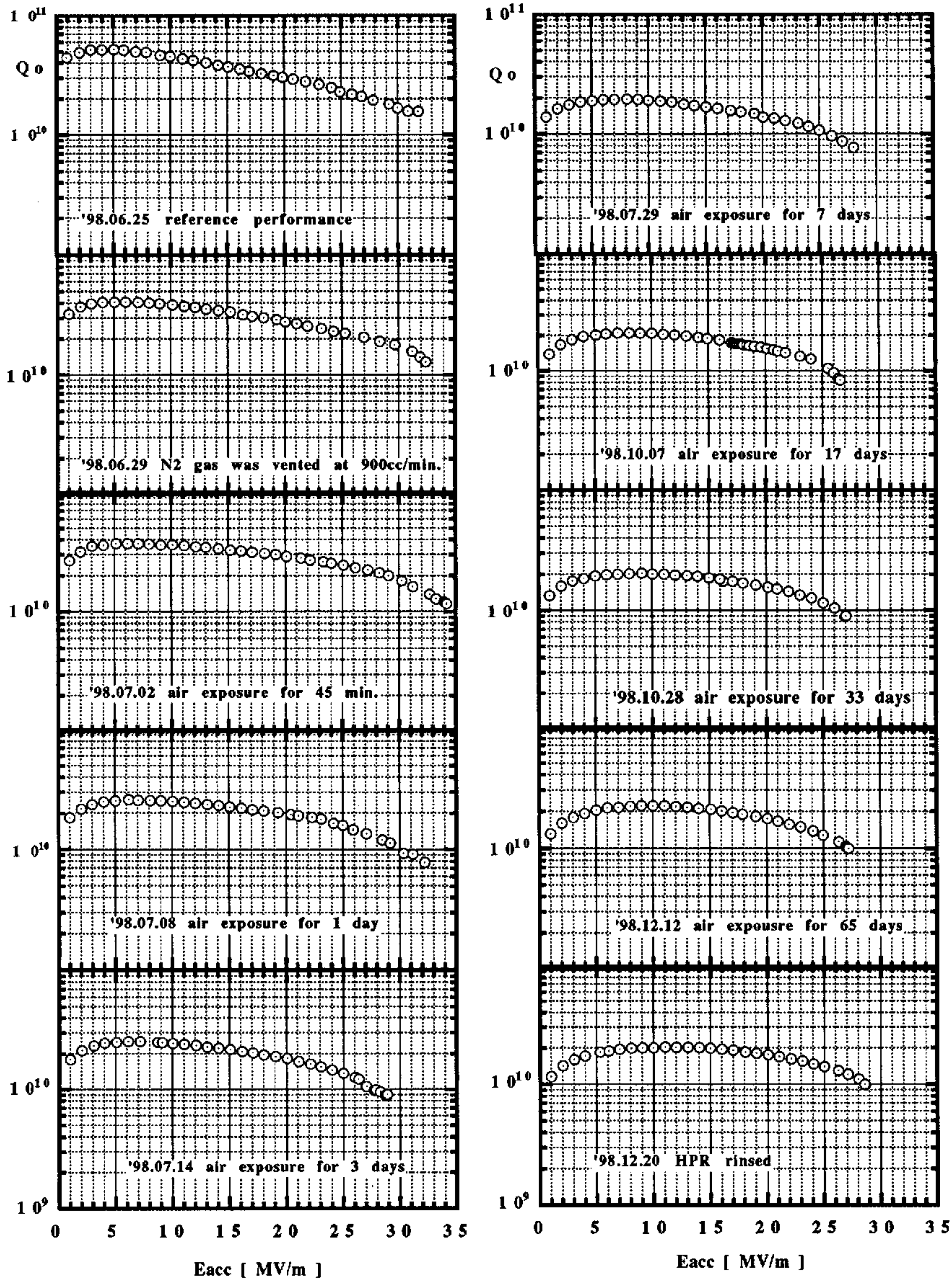

Figure 5 : Qo-Eacc curves

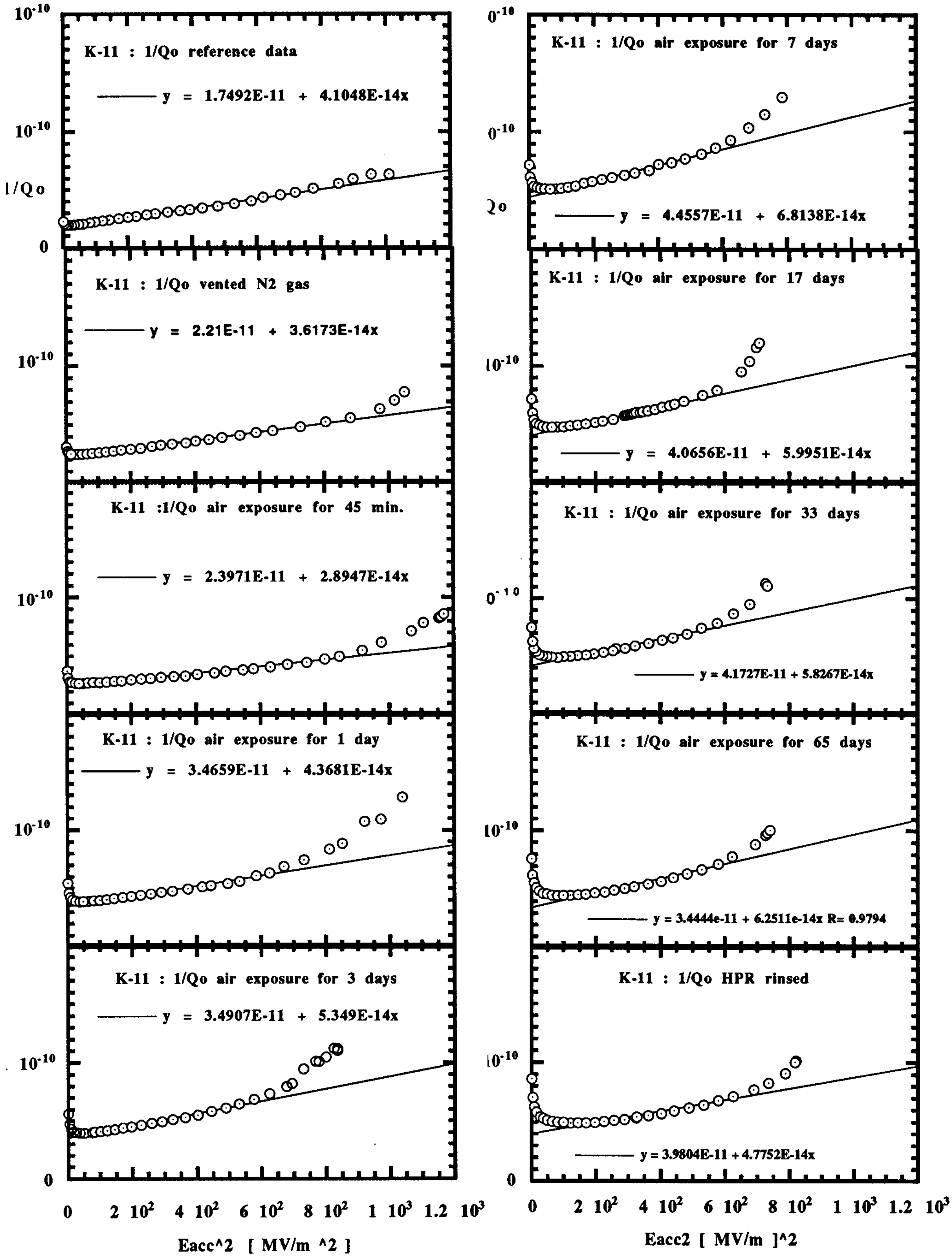

Figure 6 : 1/Qo vs. Eacc^2 curves

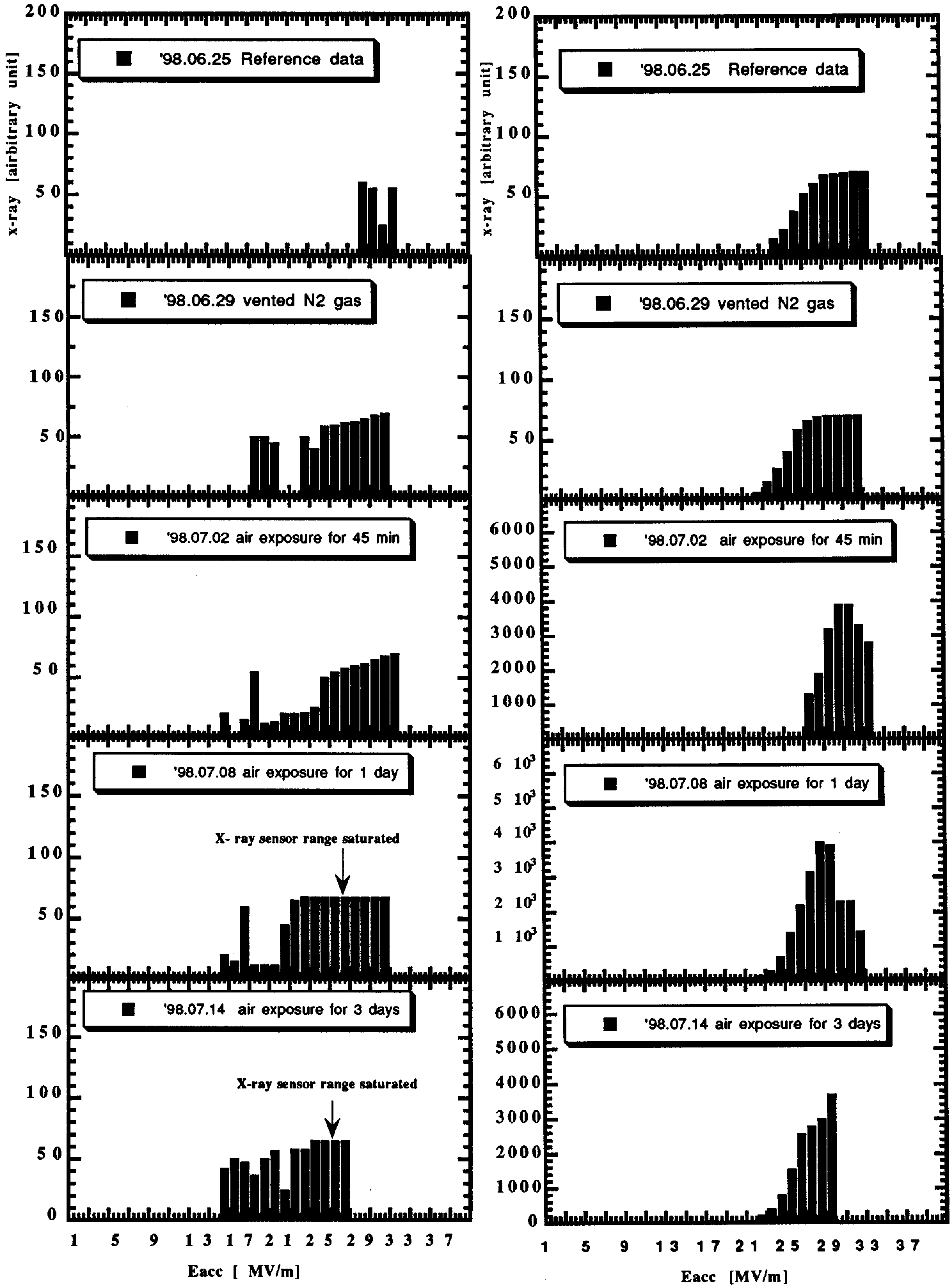

Figure 7 : Occurrence of x-ray : from the baseline measurement to the 3 day's air exposure
(a) During RF processing (b) After RF processing

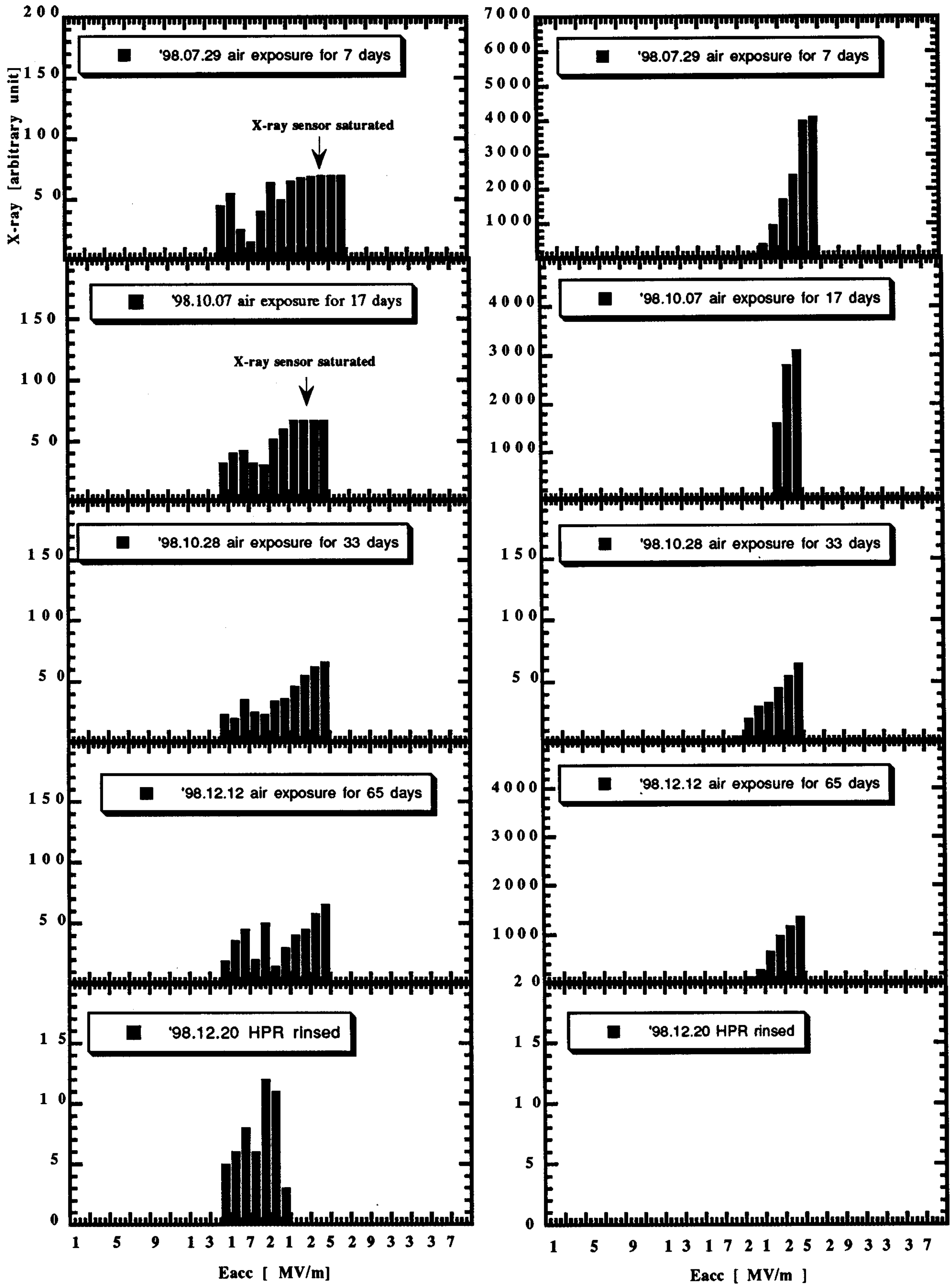

Figure 8 : Occurrence of x-ray : from the 7 day's air exposure to the HPR rinsing after 65 day's air exposure
(a) During RF processing (b) After RF processing

oxidation. The room temperature oxidation effect on niobium property was investigated in detail by Dr. J. Halbritter [4]. In his paper, the degradation of the niobium property is explained as following:

1) Oxygen solution is promoted by local stress generated by nucleation of Nb_2O_5 on the surface, and decreases Tc of the niobium.
2) NbO_x exists in the interface between the natural oxide layer and the niobium matrix. It reduces the electron density at the Fermi surface of the niobium, and weakens the pair interaction.
3) At the Fermi surface of the niobium, electrons locally bound to Nb_2O_{5-y} and conducting electrons make interferences. That weakens the pairing interaction, and brings to a degradation in the material property.

The oxygen solution of one atomic percent in niobium brings a reduction of the critical temperature by 0.93 K [4]. Namely the following relationship is observed between the band gap degradation and the increased oxygen concentration.

$$\frac{dNbOx}{NbOx} = -10\frac{d\alpha}{\alpha} \quad (3).$$

Here, NbOx means the amount of oxygen solute in niobium. Evaluating the concentration of the solute oxygen from (3) for our result: the degradation rate of the band gap = - 0.046%/day, the oxygen concentration increases 0.0046 atomic percent every day.

4.2 Degradation of high gradient

As seen in Figure 7 and Figure 8, we observed occurrences of x-ray in the high field measurement. Generally x-ray is made by electron bombardment onto a niobium wall. Such a candidate is field emission or multipacting. In the case of field emission, the amount of x-ray should be enhanced exponentially with increased field gradients. As seen in the figures, our x-ray occurrence pattern is different from field emission. The intensity does not exponentially increase. This fact means multipacting is occurring in our cavity.

In Figure 6, abnormal enhancement in 1/Qo is observed in every air exposure, which is similar to field emission. It is also seen in the HPR rinsed surface after the 65 day's air exposure. However, as seen in Figure 8, in the HPR rinsed surface x-ray is not observed after RF processing. Therefore, the abnormal enhancement in 1/Qo with the HPR rinsed surface is by thermal heating. The other abnormal enhancement with air exposed surfaces might be brought by a mixing between the thermal heating and multipacting. Thus, air exposure seems to bring two effects; 1) making thermal heating : " a new Q-degradation" [5], which might be made by oxidation, 2) promoting the multipacting, which might be due to the accumulation of carbon contamination, micro-particles or gas adsorption.

Two multipacting levels are observed at 15-20 MV/m and at 21- 34 MV/m. The lower level is disappeared by RF processing as seen in Figures 6 and Figure 7. The higher level can not be eliminated by RF processing. According to our other experiments, with the lower multipacting level a memory of the RF processing out is observed in the warming up cavities up to 200K. This fact suggests that this multipacting is pronounced by surface gas adsorption. The adsorption energy is evaluated as 0.017 eV from the memory effect. This number is popular in a physisorption. Especially, electropolished surface has a porous natural oxide layer on the surface, so the multipacting might be easily pronounced by the gas adsorption.

5 SUMMARY

We measured the degradation on the electropolished niobium surface with air exposure for 65 days. The following results were made obvious.

1) Residual surface resistance is increased about 10 nΩ in one week, and it saturates for the further air exposures. This degradation is not so serious for lower frequency cavities like TRISTAN 508 MHz cavities, because such a cavity is operated 4.2 K where BCS surface resistance is domain : e.g. 100nΩ. On the other hand, it is much serious in the cavities operated at the superfluid temperature. The increased Rres is comparable or larger than BCS surface resistance.
2) Air exposure promotes multipacting and high gradient is limited by the multipacting.

6 ACKNOWLEDGMENTS

The authors would like to thank to several people of the cryogenic center at KEK : Mrs. K. Mimori, S. Sugawara, M. Iida H. Ohhata for supplying liquid helium for these experiments. They also would like to express great thanks to Miss T. Higuchi and Mr. T. Suzuki from Nonura Plating for electropolishing. They also would like to appreciate Professor M. Kihara, director of accelerator lab at KEK.

7 REFERENCES

[1] K.Saito : " The Future of Surface Treatment Technologies for High Field Niobium Superconducting Cavities ", Proc. of Workshop on AC Superconductivity Sponsored by the Particles and Fields Commission of IUPAP, KEK, Tsukuba, Japan, June 23 - 25, 1992, P.138.

[2] K.Saito et al., " Basic research on horizontal assembly method of sc cavities with high Q and high gradient ", Proc. of the XIX International LINAC Conference, August 23 - 28, 1998 Chicago, Illinois, USA, p.294 - 296.

[3] K.Saito and P.Kneisel, " Temperature Dependence of the Surface Resistance of Niobium at 1300 MHz - Comparison to BCS Theory - ", in this workshop.

[4] J.Halbritter, " On the Oxidation and on the Superconductivity of Niobium ", Appl. Phys. A 43, 1 - 28 (1987).

[5] E.Kako et al., " Cavity Performances in the 1.3 GHz Saclay/KEK Nb Cavities ", Proc. of the 8th Workshop on Superconductivity, Abano Terme, Italy, October 6 - 10, 1997, p. 491 - 502.