The 9th Symposium on Accelerator Science and Technology, Tsukuba, Japan 1993

Study of Ultra-clean Surfaces for Accelerator Structures

K. Saito, S. Noguchi, E. Kako, M.Ono, T.Shishido, M.Yoshioka, P.Kneisel*, H. Miwa**,
T. Suzuki**, K.Kurosawa*** and K.Nishitani****

KEK, National Laboratory for High Energy Physics
Oho, Tsukuba-shi, Ibaraki-ken, 305, Japan
*; CEBAF, Continuous Electron Beam Accelerator Facility
12000 Jefferson Avenue, Newport News, VA 23606, USA
**; Nomura Plating Co., Ltd.
Satsuki-cho, Kanuma-shi, Tochigi-ken, 322, Japan
***; Sony Co., Ltd. Atsugi Technology Center
4-14-1, Asahi-cho, Atsugi-shi, Kanagawa-ken, 234 , Japan
****ATC Co., Ltd.
36-7, Namiki-cho, Hachioji-shi, Tokyo-to, 193, Japan

Abstract

For a TeV energy physics R&D on electron/positron linear colliders has been conducted hard at many laboratories from technologies of both normal conducting and superconducting. The high field gradient issue is a key to realize such a machine. Field emission limits seriously field gradient of rf cavities. Its cure is to eliminate particle contamination on cavity surfaces. It is a common issue in both normal conducting and superconducting cavities. We have started to study ultra-clean surfaces of niobium and copper applying semiconductor technologies. In this paper several results by various rinsing methods are presented and its relation with cavity performance is discussed.

I. INTRODUCTION

Since on superconducting rf cavities a thermal instability problem leading to a quench at surface defects has been suppressed by developing niobium material with a high thermal conductivity at low temperatures, presently field emission is a main field limitation. For normal conducting cavities field emission makes a problem with dark current. A source of field emission is dominantly dust particles remaining on cavity inner surfaces. Several cures against field emission have been applied for superconducting cavities; i.e. use of ultrapure water rinsing in the chemical surface treatment, assembly in a clean room. However, still it can not be overcome and a powerful tool which removes dust particles has to be developed.

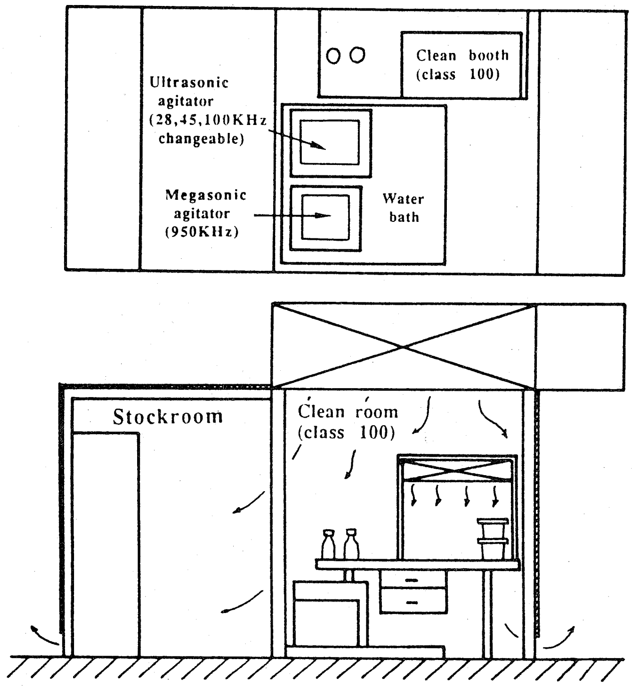

Fig. 1 Setup for rinsing silicon wafers with various methods.

We have started a study to make an ultra-clean metal surfaces more dedicatedly because it is a common important issue for linear colliders. In order to eliminate dust particles, the following points will be important; surface treatment in a clean environment, use of clean chemicals and development of a powerful tool to remove particles. In this paper, as the first output how many dust particles remain on the surface after cavities are rinse by means of various methods or in different working environments is reported. In addition, a result with high pressure water rinsing is discussed from cavity performance. We are investigating amount of particles and size distribution in chemicals, however, there is no space to describe it, so it will be presented somewhere else.

II. SETUP OF EXPERIMENT

We use a laser reflection method to count residual dust particles on samples rinsed with various procedure. Such an equipment is already sold for semiconductor industries in order to control dust particles on silicon wafers. In this kind of equipment a focused scanning laser beam is irradiated on a smooth silicon wafer and the scattered light signal from an eventual dust particle is amplified by means of a photo multiplier. It gives us information with the number of particles and its size distribution. In principle it could be used for other metal surface as well as silicon, however, it needs a lot of time to establish a qualitative analysis. In this experiment we used silicon wafer from easiness and ignored the difference between silicon and niobium in particle adhesion. It will be permissible in the first approximation. Clean silicon wafers(100^{ϕ}, N–type) from the Shinetsu kagaku Co. were rinsed with various methods described bellow at the Nomura plating Co. where surface treatments of

TRISTAN superconducting niobium cavities have been carried out, then brought to a semiconductor laboratory(Sony Co. Atsugi Technology Center) in order to analyze dust particles.

Rinsing methods applied in this experiment are as follows; 1) just rinsed in a clean room with ultrapure water, 2) rinsed with an ultrasonic agitator changeable a frequency (28,45,100KHz), 3) rinsed with a megasonic agitator (950KHz), 4) rinsed with high pressurized ultrapure water(HPR), 5) used reagent grade chemicals, 6) used EL-grade chemicals controlled strictly with particles for semiconductor.

A setup for rinsing is shown in Fig.1. Our large clean room is doubt in the cleanliness because that is used as a stockroom for clean parts of surface treatment and less controlled. We had a small clean booth(class 100) in the clean room in order to make a guaranteed clean space. In this clean booth virgin wafers are taken out and put in a carrier box after rinsing. Rinsing work area is around the water bath and its cleanliness will be 1000. Wafers are handled carefully with a vacuum pincette not to contaminate the surface to be analyzed. Sampling of each rinsing method is done for two wafers. The average value of the numbers of dust particles on the two samples is used in the next section. The number is counted for particles larger than 0.3 µm in size.

III. RESULTS AND DISCUSSION

A. Residual dust particles in rinsing procedure for TRISTAN sc cavities

Recently the rinsing procedure in the surface treatment for TRISTAN sc cavities[1] looks poor to eliminate field emission for single cell cavities. A simulated test on the procedure was conducted and residual dust particles on the surface were estimated. As the first rinsing, wafers were immersed in a reagent electropolishing acid (H_2SO_4:HF=10:1 in volume) for 5 minutes at the outside of the clean room, then rinsed for 5 minutes in the clean room. Silicon wafers are little etched by hydrofluoric acid in the acid. The result is shown in Fig.2. The number of dust particles was 13000 and close to that of the wafers which were put outside of the clean room. That was >14000 and the particle counter overflowed due to too much contamination. That value was reduced a little bit after the second shower rinsing but still 11850. After hydrogen peroxide (H_2O_2) rinsing, that increased to 13250. In the final rinsing which ultrapure water was overflowed only inside of the cavity in a hot bath (50^oC), the number of dust particle left on the wafer is 10000. Its particle distribution is shown in Fig.3. When HPR [85kg/cm^2, 10min] was used, dust particles was eliminated to 1640. Its residual particle distribution is shown in Fig.4. If surfaces are dried once, it is difficult to remove dust particles. In such a case, that is reduced to only 8500 even by means of HPR and 2650 with a megasonic agitator [950KHz] for 10 minutes. Megasonic rinsing is more effective than HPR.

B. Effect of a megasonic agitator

The dust particles which adhered at the outside of the clean room are eliminated to 7930 by just ultrapure water rinsing in the clean room. On the other hand if one uses a megasonic rinsing, they are reduced to 640 close to the background level (700) of the rinsing environment in the clean room. It should be emphasized that the megasonic rinsing is most effective in any case of tests in the series(see Fig.2) .

C. Dust particles from environment or chemicals

If an EL-grade acid is used in the clean booth, dust particles are shut out to 70. This value is extremely small comparing to that of others. Its particle distribution on a wafer is shown in Fig.5. On the other if a reagent acid is used in the same environment, that increases to 400 but they are removed to 120 easily by means of the megasonic rinsing. Many dust particles come from the environment and the reagent acid.

D. Rinsing with a reagent alcohol

Alcohol rinsing is often used for niobium sc cavities due to its easy drying out. Particle contamination in the rinsing

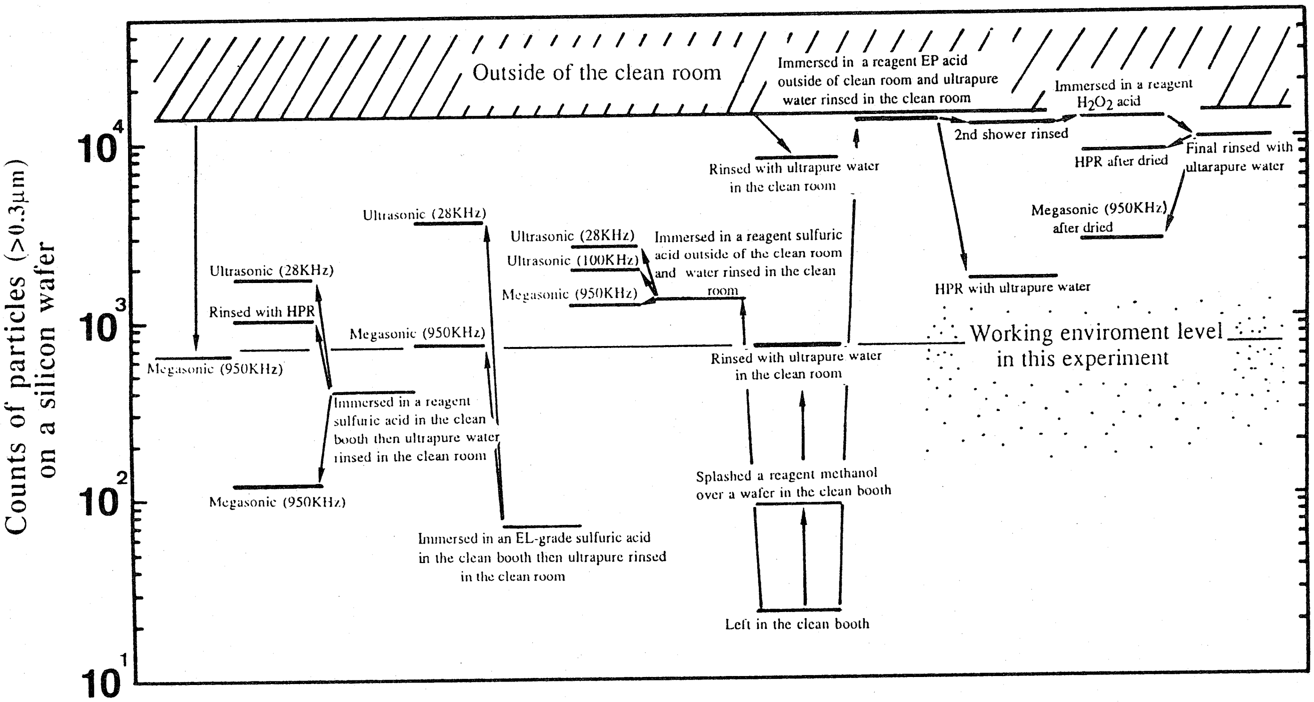

Fig. 2 Results with the number of dust particle left on silicon wafers with various rinsing methods.

was investigated. A reagent methanol was splayed over wafers in the clean booth. The number of residual particles was only 90. Even the reagent alcohol, it is unexpectedly clean. It was testified also by a particle measurement in the solvent.

E. Residual particles with HPR and cavity performance

Figure 5 shows a result on HPR with a CEBAF's 1.5GHz single cell cavity. The cavity was electropolished by 120 µm at KEK, then sent back CEBAF. The first measurement is on just as received but alcohol rinsing was conducted at CEABF used a reagent methanol. The field gradient was limited to 20 MV/m due to field emission. In the second measurement HPR was done for the cavity at a pressure of 84kg/cm^2 for 20 minutes at CEBAF[2]. The accelerating field gradient (Eacc) reached 27.6MV/m with a Qo value of 10^{10} without field emission. After the measurement the cavity was chemically polished by the CEBAF's standard procedure, then HPR was carried out again in the same condition as before. Qo values dropped a little bit but the Eacc reached 33MV/m and the no field emission below 28MV/m was reproduced. As the intensity of the X-rays was very low at the higher field over 28MV/m, it is not clear that field emission still limited the accelerating field gradient. However, if field emission still limites the field, one could upgrade the field limitation using a more powerful rinsing tool as the megasonic agitator.

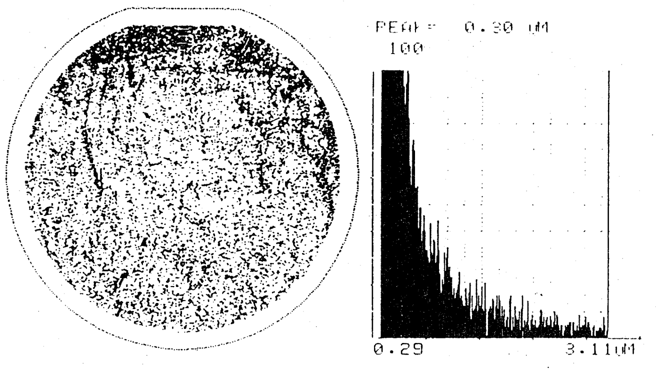

Fig. 3 Distribution of dust particles left on a wafer by the same rinse as one for TRISTAN sc rf cavities.

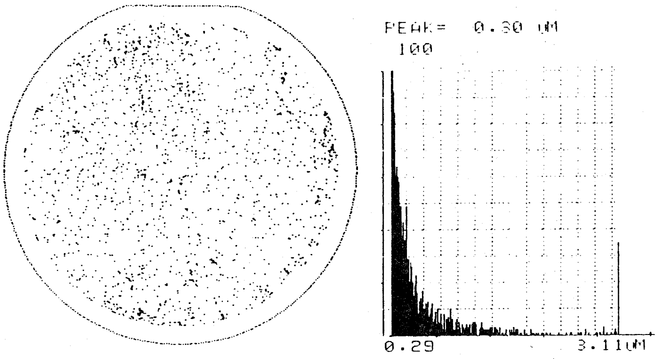

Fig. 4 Distribution of dust particles left on a wafer by means of HPR. The wafer was immersed in a reagent EP acid outside of the clean room before HPR.

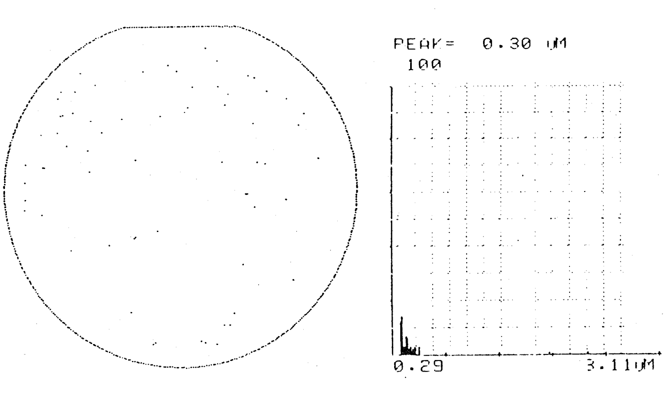

Fig. 5 Distribution of dust particles left on a wafer immersed in an EL-grade sulfuric acid in the clean booth, then rinsed with ultrapure water in the clean room.

IV. SUMMARIES

From an accelerator's point of view, a study on clean metal surfaces has been started. In this paper initial outputs were presented. For the clean surfaces, 1) Working environment, 2) Clean chemicals and 3) Tools to remove actively dust particles have to be considered. If our working environment is improved to class 100 grade and EL-grade chemicals are used, dust particles could be reduced 100 times less from the present level. Even the reagent grade chemicals is used in a class 100 environment, the same reduction could be done combining the megasonic rinsing. It was demonstrated that the megasonic agitator or HPR is a very effective tool to remove dust particles. It was testified that field emission is remarkably eliminated by meas of HPR.

V. ACKNOWLEGMENTS

The authors would like to thank Profs. Kimura, Takata and Kurokawa for their continuing supports and encouragements for our study. Our special thanks go to Mr. Yamagen of the Kaijo Co. Ltd. for borrowing a megasonic agitator.

VI. REFERENCES

[1] K.Saito et.al.,"R&D of Superconducting Cavities at KEK', Proc. of the 4th Workshop on RF Superconductivity, 1989 at KEK, P.635.

[2] P.Kneisel et.al., "A High Pressure Ultra Pure Water Rinsing System for Niobium Cavities", CEBAF Tech. Note TN #93-029.

8

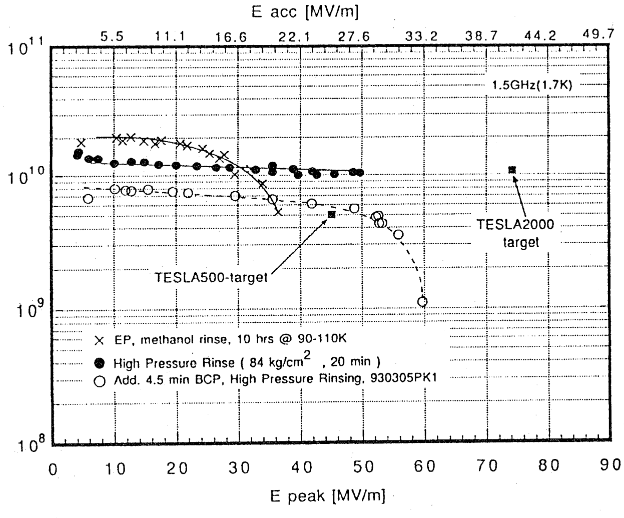

Fig. 6 Cavity performance rinsed with HPR.