Evaluation of dust particles in the KEK clean room and cleaning methods used for SC cavity assembly

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Abstract

Size and number of particles in the clean room used for SC cavity assembly were evaluated using a particle counter and wafer inspection system. The assembly area was class 10-20 when no one is working and it increased momentarily by about 2 orders of magnitude when 6 people were assembling a cavity. Dusts coming off from clothing, aluminum foil for wrapping, and wiping cloth were counted. Effects of the several cleaning methods used during assembly, e.g. blowing with filtered air and wiping with a cloth wetted with ethanol, were checked using a Si wafer. It was found that micron or sub-micron size particles are difficult to be eliminated. Blowing with ionized air had some effect to reduce particles probably because the electrostatic force can be reduced due to the neutralization of the charges on the dust. It was also found that wet surface tends to get more contaminated than dry surface.

Introduction

Today, clean work is essential for SC cavity assembly. At KEK, a 10m x 10m large clean room was constructed for the large-scale production of TRISTAN 5-cell cavities in 1987. Since then, we believe, this clean assembly have helped produce high-quality cavities constantly [1].

There is, however, still much room for improvement on our clean work. For higher field and stable cavities such as TESLA and B-Factory, we need to know if our present clean room or clean work is really clean enough for them. To begin this assessment, we started measuring the cleanliness of our clean room more precisely and counted the particles from various things being used in the clean room. In addition, we applied a wafer inspection system, which is commonly used in semi-conductor industries for the evaluation of the particles, to checking our cleaning methods. Although this paper presents only size and number of the particles we counted, we are planning to check the material of the particles in the future.

Cleanliness of the clean room

Clean room

Figure 1 shows our clean room. There is a class 100 clean room called clean booth inside of a class 1000 clean room called prep. room so that the filters of cleaner room have longer life. The ceiling of the clean booth is covered with HEPA filters and two small filtering units are installed on the ceiling of the prep.

room. In the prep. room, there are 2 vacuum stations to evacuate the assembled cavities. In this room, we do not usually wear special clothes. However, in the clean booth, we put on masks, gloves, hoods, mask covers and clean coats.

Particle counter

We used a particle counter, KC-21 of RION Corp., which focuses He-Ne laser on a small area through which particles pass and reflect light. This reflected light is detected by photo diodes, and its amount and number are correlated with the size and the number of the particles. Normally correction of particle counters is performed with poly-stylene latex spheres. Therefore, the measured size corresponds to the size of this poly-stylene sphere and might not be the same size as the material being measured.

The flow rate of the air is changeable between 0.1CF and 1CF. The smallest measurable size is 0.1μm.

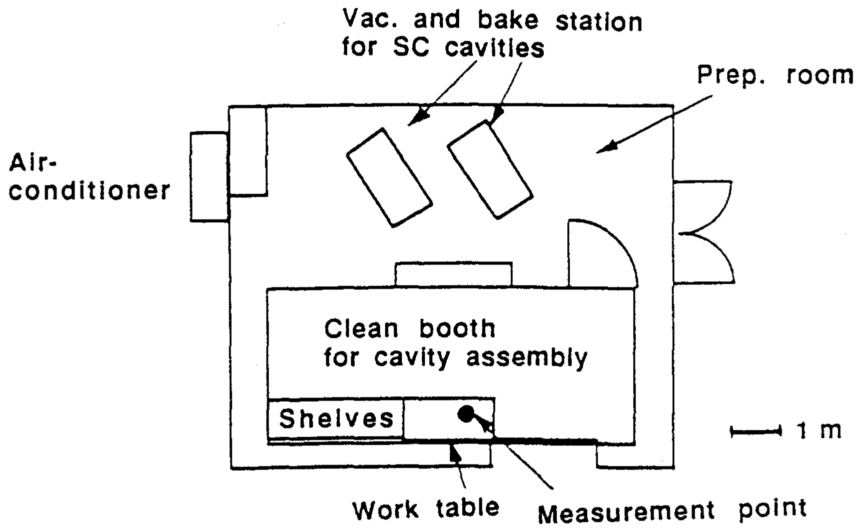

Fig. 1. Schematic representation of KEK clean room for SC cavity assembly

Results

The cleanliness of the clean booth was measured on the work table shown in Fig. 1. The particles stuck to the hoods, masks, gloves, mask covers, aluminum foils and wiping cloth were measured by rubbing them at 5-10 cm above the particle counter.

The results are shown in Table 1. In the Table, number of each column shows the number of the particles that have diameters greater or equal to this number and smaller than the diameter written on the right.

This measurement was carried out using the 0.1CF mode, i.e. the measurement time is one tenth of the time taken for the 1CF measurement, to reduce measurement time. With this mode, sometimes no particle was counted on the work table in the clean booth as shown in Table 1. For more detailed measurement about the cleanliness of the clean booth, we used 1CF mode and measured 22 times consecutively. Table 2 shows the result. Particles of 0.2-0.3 μm are the most in number and the maximum total number was 147 as seen in Table 2.

During the tests on the tools, just for a comparison, we added some measurements on human bare hand, bare head and portable duster as shown in Table 1. It was interesting to find that 50-60% of the particles produced by a bare hand are around 0.1μm, although it may differ much from one person to another. Nevertheless, needless to say, gloves are necessary for clean work. Duster also showed many particles only around 0.1-0.15μm. These particles seem to be the misty particles that come out of the duster and it should not be harmful.

Table 1. Number density of the particles contained in various items.

	Particle density (1/0.1CF)					
Item	0.1μm	0.15μm	0.2μm	0.3μm	0.5μm	total
Clean booth	0	0	0	0	0	0
Glove #1	10	2	3	2	7	24
#2	2	3	3	1	5	14
#3	10	3	10	8	26	57
#4	14	9	8	8	6	45
Wiping cloth[1)]#1	14	4	1	4	71	94
#2	19	10	6	10	159	204
#3	20	3	6	12	191	232
#4	24	3	11	22	359	419
#5	11	4	2	6	132	155
Al foil[2)] #1	4	3	2	2	9	20
#2	4	3	3	2	4	16
#3	11	3	5	10	8	37
#4	5	1	8	8	16	38
#5	12	2	4	4	10	32
Hood #1	58	23	54	54	125	314
#2	112	50	85	104	194	545
#3	123	60	93	118	228	622
#4	121	56	67	112	246	6
#5	43	33	48	82	137	3
Coat #1	62	51	70	93	649	925
#2	71	36	62	128	618	915
#3	73	56	91	114	758	1092
#4	58	37	68	99	536	798
#5	45	23	61	77	382	588
Bare hand 1st	3172	858	701	364	246	5341
2nd	4168	1321	1443	923	438	8293
3rd	3528	1350	1826	1045	414	8163
Mask #1	13	14	7	8	81	123
#2	47	19	16	14	58	154
#3	15	26	43	35	148	267
Mask cover #1	24	13	26	34	91	188
#2	30	21	46	43	83	223
#3	50	34	61	73	110	328
#4	19	9	33	33	67	161
#5	134	51	83	80	138	4
Bare head	696	317	410	293	255	1971
Duster[3)]	4939	135	59	5	2	5140

1)Cellulose wiper named Lint-free PS-2, Asahi Kasei Co.

2)20 μm-thick foil , frequently used for wrapping or covering things.

3)Portable type duster named GUST II. We use either filtered N_2 gas or this duster.

To know the change of the number of particles when some people are working, we measured time evolution of the particle number when 6 people assembled one SC cavity. Figure 2 shows the result. The particle density increased for about 10 min. then decreased to a certain level. The maximum density became as much as 2-3 orders of magnitude greater than the start value as shown in the figure, depending on the particle size.

Figure 3 shows the particle density in the case of both there is no one working and there are 6 people working (max. value) as a function of particle size together with the lines of Japanese Industrial Standard (JIS) Class and US Federal Standard Std-209D Class. As one can see, our clean booth is class 20 or less. Despite this cleanliness, dust particles increase significantly during the assembly.

Table 2. Particle counts on the work table in the clean booth. There was no one in the room. The counts were repeated 22 times.

Particle density (1/CF)					
0.1μm	0.15μm	0.2μm	0.3μm	0.5μm	Total
0	1	1	1	0	3
0	1	4	3	0	8
3	4	8	4	1	20
4	5	14	5	1	29
7	8	19	8	1	43
10	12	22	8	4	56
11	13	22	11	3	60
8	12	20	13	2	55
9	12	19	8	3	51
13	16	27	15	3	74
15	14	34	14	3	80
11	13	26	12	2	64
12	14	24	11	2	63
13	17	35	20	3	88
18	17	35	18	4	92
21	28	42	22	5	118
20	23	40	21	4	108
27	33	57	23	7	147
26	23	40	19	5	113
14	13	31	16	4	78
17	14	29	18	2	80
19	21	39	20	6	105

Particle counts with a wafer inspection system

Wafer inspection system

We used a system, LS-6000 manufactured by Hitachi Electronics Engineering Co., which is normally used for inspection of Si wafers in semi-conductor industries. In this system, Ar laser is focused on a tiny area of 20μm x 200μm oval and its reflected light is analyzed. The Si wafer being inspected rotates and the laser scans from the center to the edge, counting the number of particles and memorizing the amount of the reflected light.

Samples

As listed in Table 3, we prepared 16 samples, of which 2 were unused sample as a back ground, 2 were used for the measurement of cleanliness of clean booth and prep. room, 5 were used for simulating cleaning procedure in dry condition and the rest for wet condition.

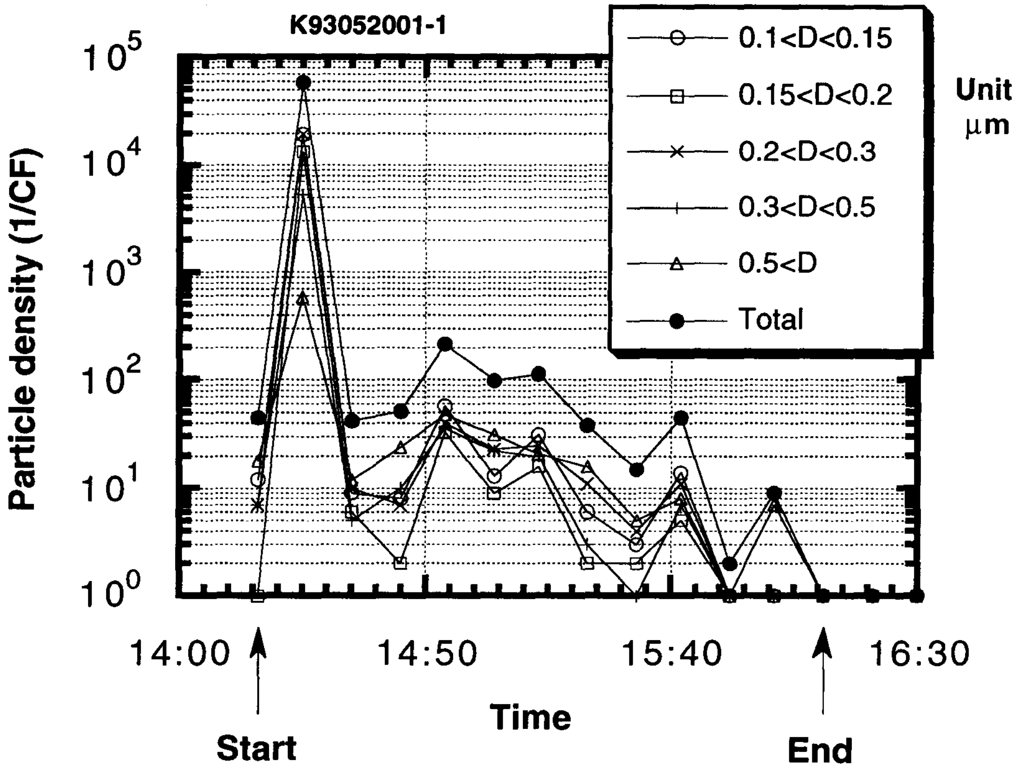

Fig. 2. Time evolution of the number of the particles in the clean booth when 6 people assembled an SC cavity.

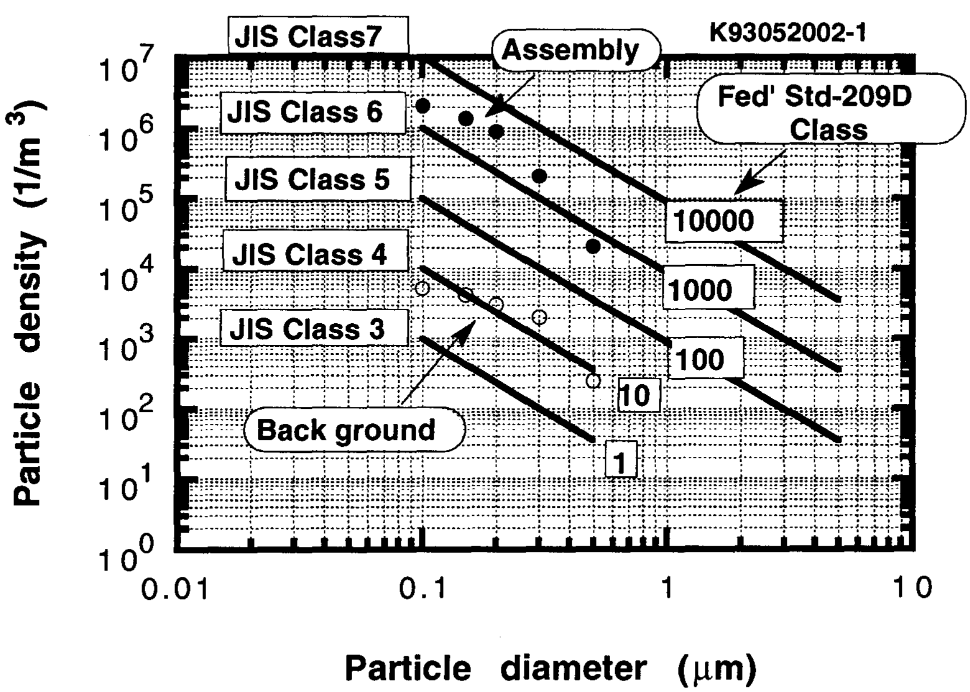

Fig. 3. Particle densities when there is no one and when 6 workers were assembling an SC cavity.

Results

The particle maps and the size distribution of each sample are shown in Figs. 4 through 19 in order of the sample number as shown in Table 3. The actual outputs of these maps and graphs are colored according to the size range of the particles.

Figure 4 shows one of the unused wafer. Fig. 4(a) shows the particles equal to or bigger than 0.2μm and Fig. 4(b) shows the particles equal to or bigger than 0.1μm. As one can see, unused wafer was found to be covered with many particles smaller than 0.2μm. Thus, we decided to count only the particles equal to or bigger than 0.2μm to make the difference from other samples clearer. Therefore, all the rest figures show only particles equal to or bigger than 0.2μm .

Table 3. Samples for the tests using a wafer inspection system.

Sample No.	Conditions	Fig.
1-1	Not used, clean packed in the manufacturer	4
1-2	Not used, clean packed in the manufacturer	5
1-3	Put on a work table in the clean booth for 5 hours	6
1-4	Put on a low table in the prep. room for 5 hours	7
2-1	Put on a 92cm-high table outside the clean room for 10 min.	8
2-2	After 2-1, wiped with the wiping cloth wetted by ethanol and blown with filtered N_2 gas at $5kg/cm^2$ in the clean booth.	9
2-3	After 2-1, wiped with the wiping cloth wetted by ethanol in the clean booth.	10
2-4	After 2-1, blown by a duster named GUST II in the clean booth.	11
2-5	After 2-1, blown by filtered N_2 gas at $5kg/cm^2$.	12
3-1	After wetting with ethanol, put on a 92 cm-high table outside the clean room for 10 min.	13
3-2	After wetting with the ultrapure water kept in the clean room for months, put on a 92 cm-high table outside the clean room for 10 min.	14
3-3	After 3-1, blown by $5kg/cm^2$ filtered N_2 gas in the clean booth.	15
3-4	After 3-1, blown with a portable duster, GUST II.	16
3-5	After 3-1, blown with a neutralized gun named TOP GUN.	17
3-6	The same as 3-1	18
3-7	After wetting with the ultrapure water kept in the clean room for months, put on a 92 cm-high table outside the clean room for 10 min.	19

The samples starting with 2- were put on a table outside the clean room to study how the dusts collected outside can be taken off with various cleaning procedures. The samples starting with 3- were wetted with either ethanol or water and put outside the clean room to study the effect of various cleaning procedures on the dusts stuck on a wet surface.

Figure 20 shows a summary of all the samples. The numbers were normalized to the full surface area since the measured area sometimes stopped in reaching the count limit due to too many particles.

The results can be summarized as follows,

1) The wafer put on a table in the clean booth for 5 hours collected about 500 $0.2\mu m \leq$ particles. This is reasonable when we think of the down flow at 10-20cm/s.

2) The sample put on a table in the prep. room for 5 hours collected about 1200 0.2μm≤ particles.
3) The sample put outside the clean room for 10 min. collected about 6200 0.2μm≤ particles.
4) Wiping the dry sample with the wiping cloth wetted with ethanol raised the number of particles. Total number became twice as many. The increase of the 1μm≤ particles were drastic.
5) When blowing the dry sample with 5kg/cm^2 N_2 gas was added after wiping with the wetted wiper, the number of the particles became even more. The total number got 3 times as many as that before cleaning.
6) Blowing the dry sample with either filtered N_2 gas or portable duster reduces about 20-30 % of the particles. Only the particles smaller than 1μm decreased in number.
7) Wet wafer was found to have 2.5-3 times as many particles as the dry one. Especially, 1.5μm≤ particles increased in number drastically. The reason for this is not known now because there is an ambiguity that there might have been many particles in the liquid before collecting the dusts.
8) 5kg/cm^2 N_2 gas could reduce only about 5% of 1.5μm≤ particles on the ethanol-wetted sample , but 1.5μm> particles increased in number.
9) Portable duster could reduce about 50% of 1μm≤ particles on the ethanol-wetted sample , but 1μm> particles increased in number.
10) The ionized air-gun, Top Gun, could reduce 75% of 1μm≤ particles on the ethanol-wetted sample, but 1μm> particles increased in number more than portable duster and N_2 gas did.
11) Comparing the sample 3-1 and 3-6, it is found that the number of 1μm≤ particles does not change from one sample to another, but smaller particles changes much.
12) In contrast to the ethanol-wetted sample, the particles on the water-wetted sample decreased a lot with 5kg/cm^2 N_2 gas (See sample 3-7). 73% of 1μm≤ particles were dusted off, also the number of smaller particles did not change much.

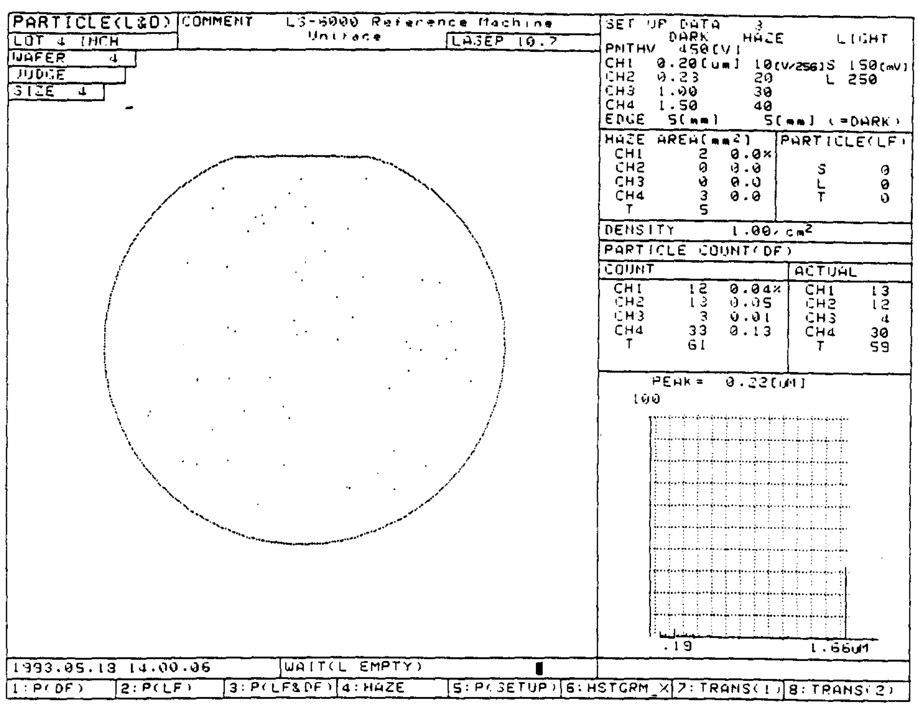

Fig. 4(a). Particle map and size distribution of sample 1-1, unused Si wafer. Particles bigger than or equal to 0.2μm are shown on the map.

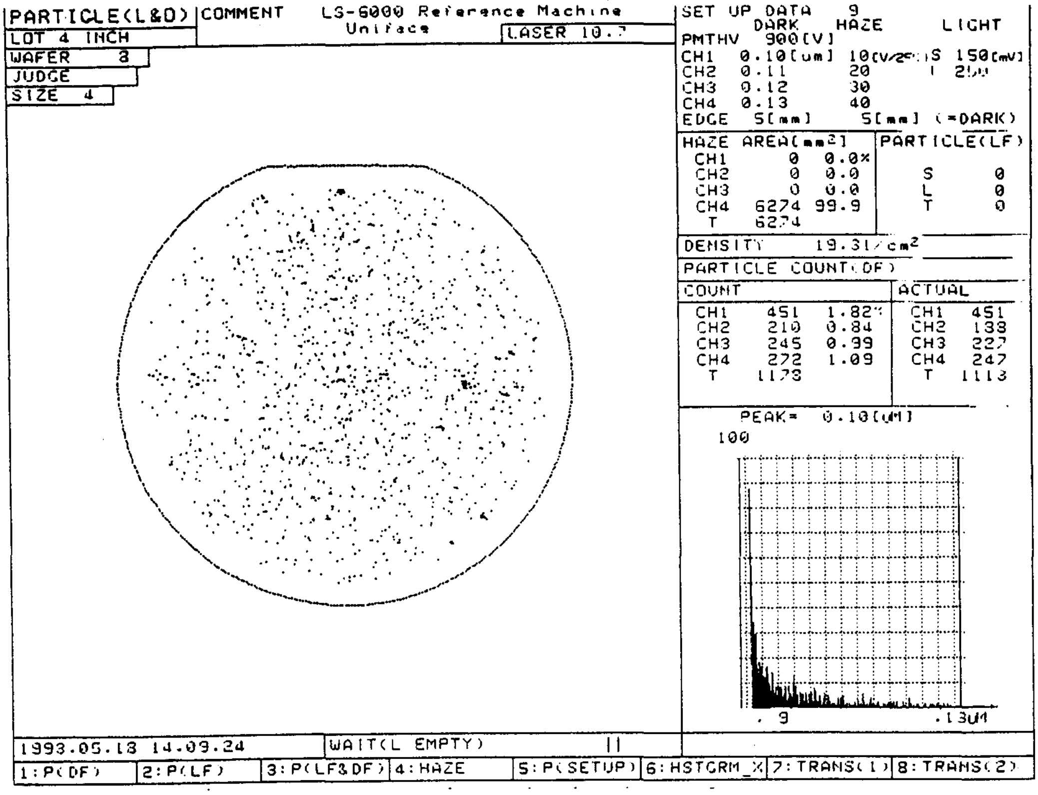

Fig. 4(b). Particle map and size distribution of sample 1-1. Particles bigger than or equal to <u>0.1μm</u> are shown on the map.

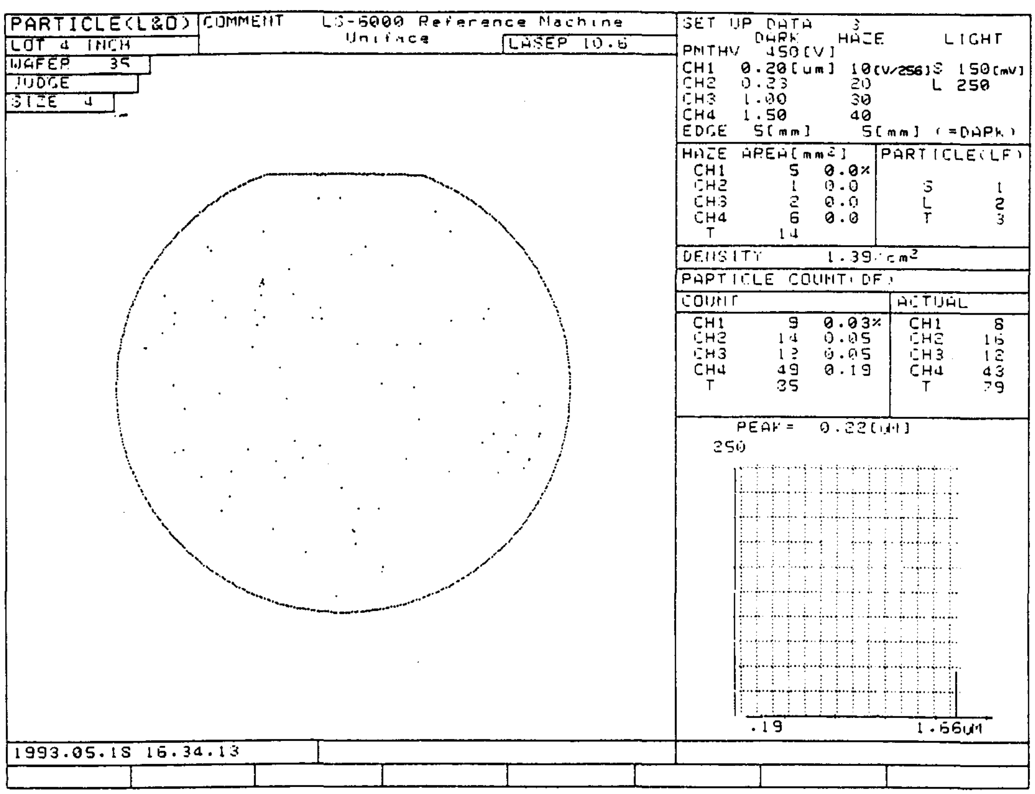

Fig. 5. Sample 1-2. Not used, clean packed in the manufacturer.

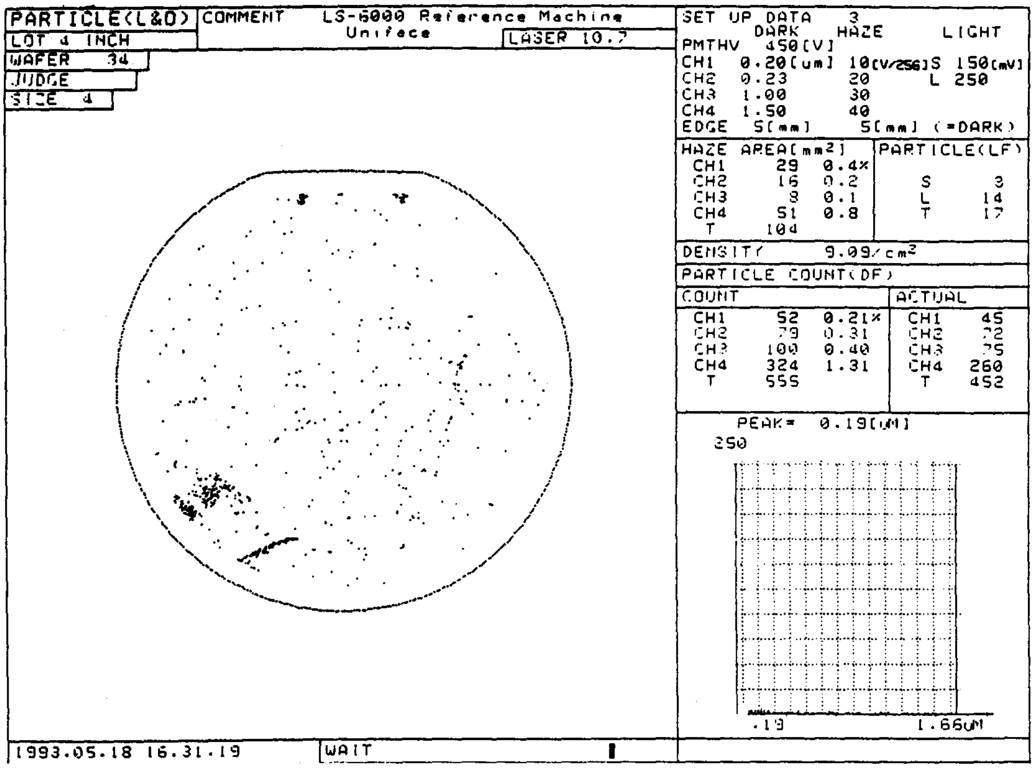

Fig. 6. Sample 1-3. Put on a work table in the clean booth for 5 hours.

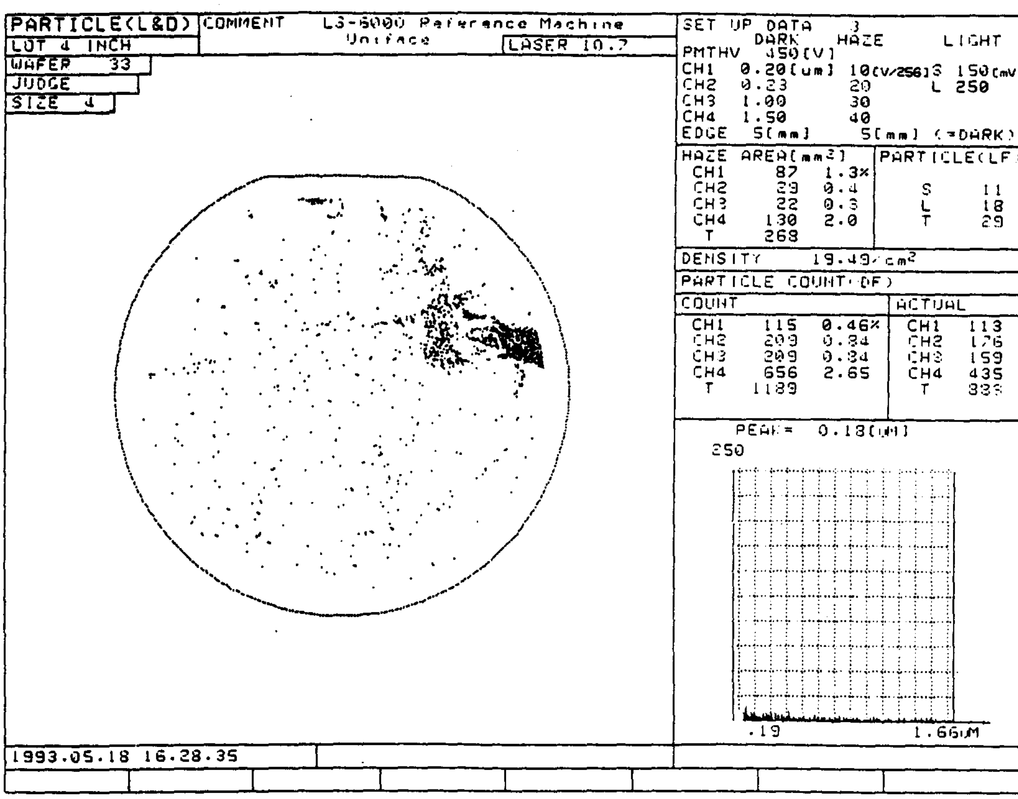

Fig. 7. Sample 1-4. Put on a low table in the prep. room for 5 hours.

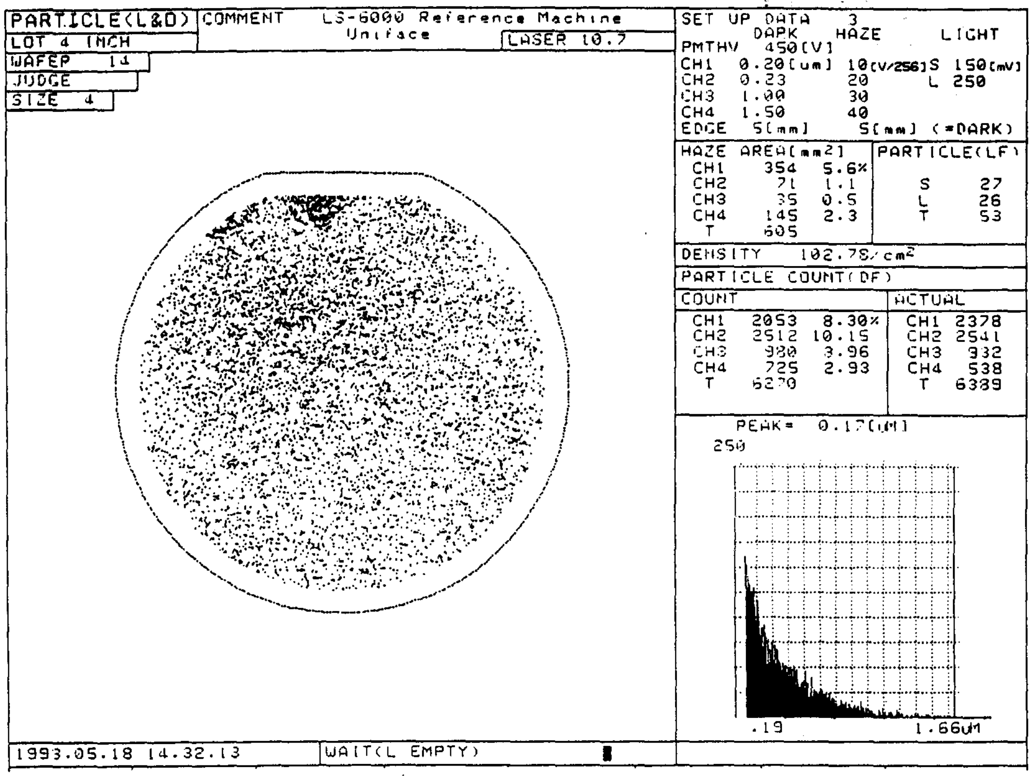

Fig. 8. Sample 2-1. Put on a 92cm-high table outside the clean room for 10 min.

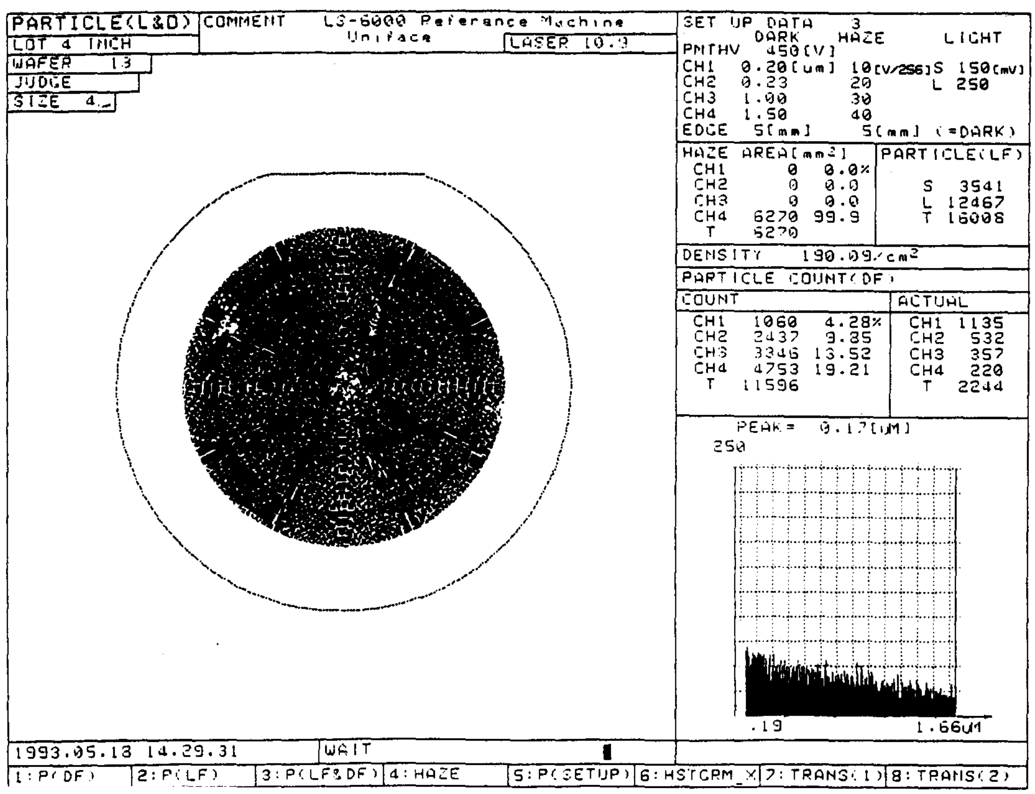

Fig. 9. Sample 2-2. After 2-1, wiped with the wiping cloth wetted by ethanol and blown with filtered N_2 gas at 5kg/cm^2 in the clean booth.

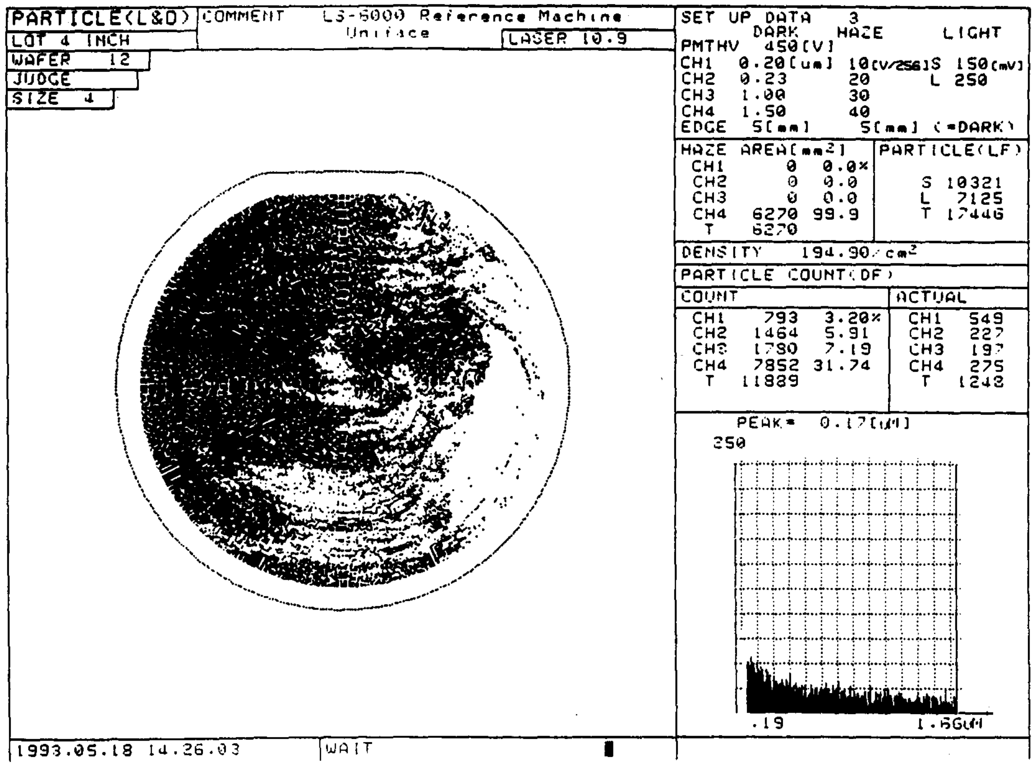

Fig. 10. Sample 2-3. After 2-1, wiped with the wiping cloth wetted by ethanol in the clean booth.

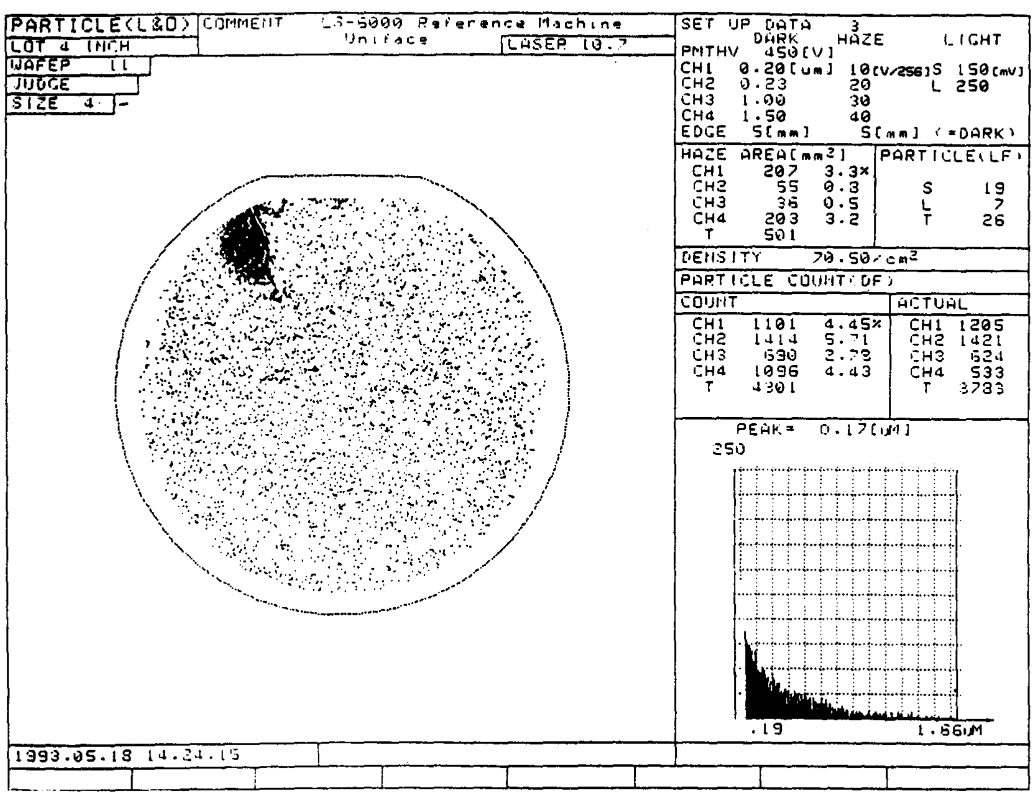

Fig. 11. Sample 2-4. After 2-1, blown by a duster named GUST II in the clean booth.

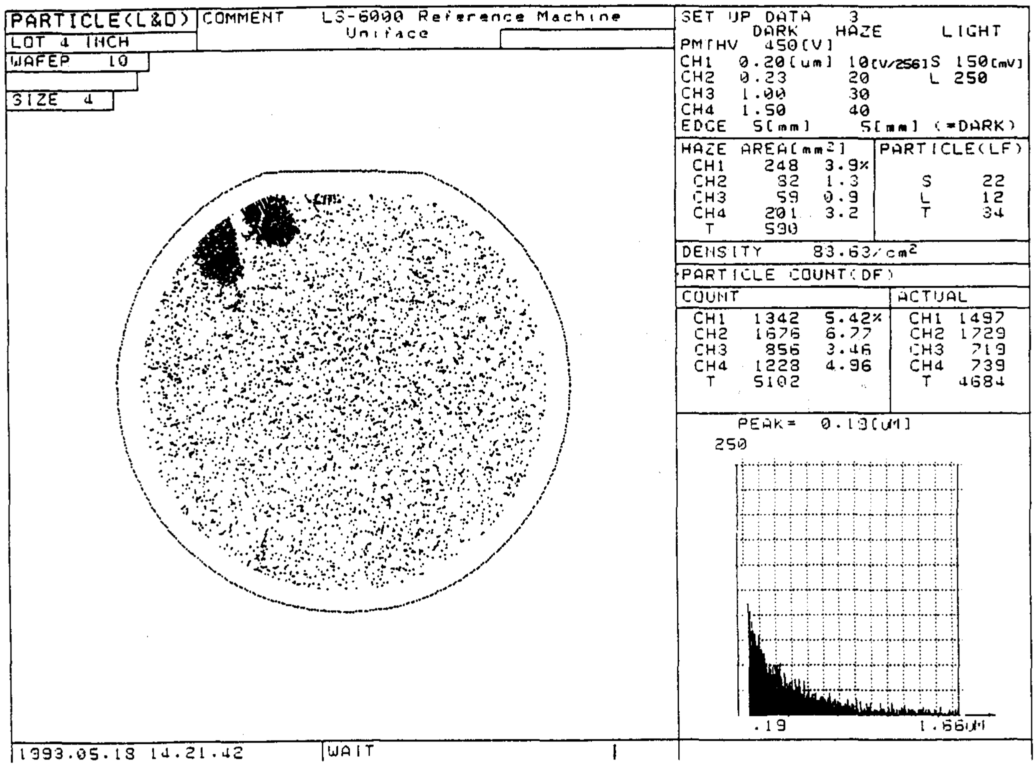

Fig. 12. Sample 2-5. After 2-1, blown by filtered N_2 gas at $5kg/cm^2$.

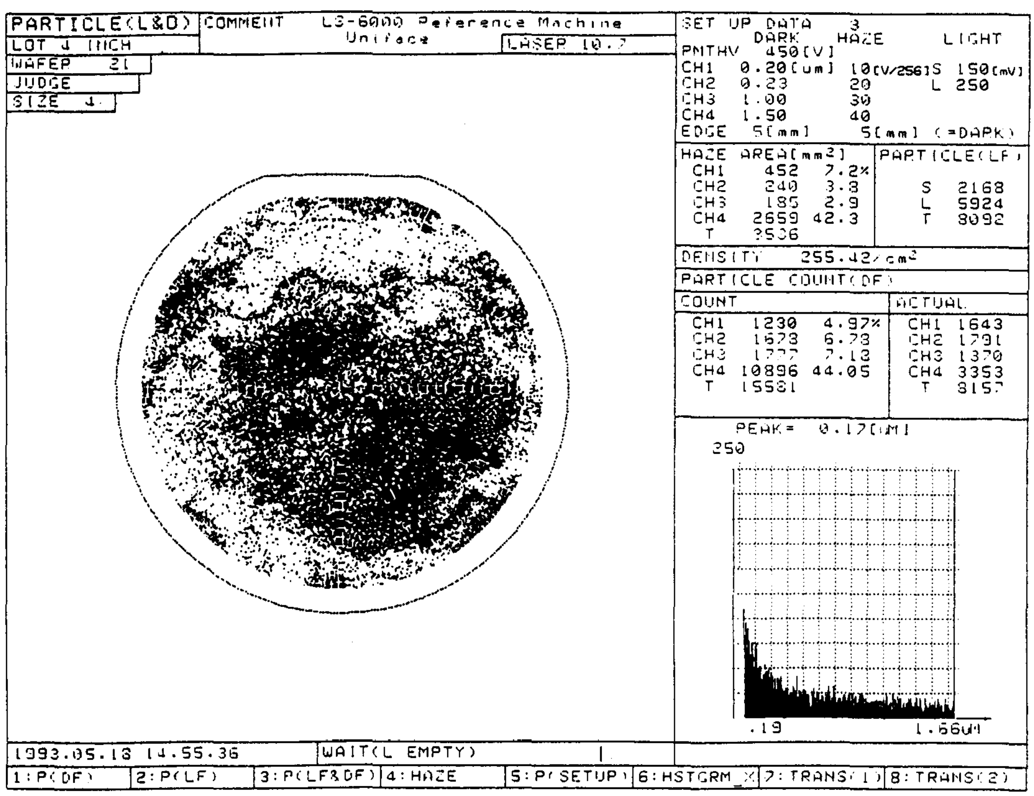

Fig. 13. Sample 3-1. After wetted with ethanol, put on a 92 cm-high table outside the clean room for 10 min.

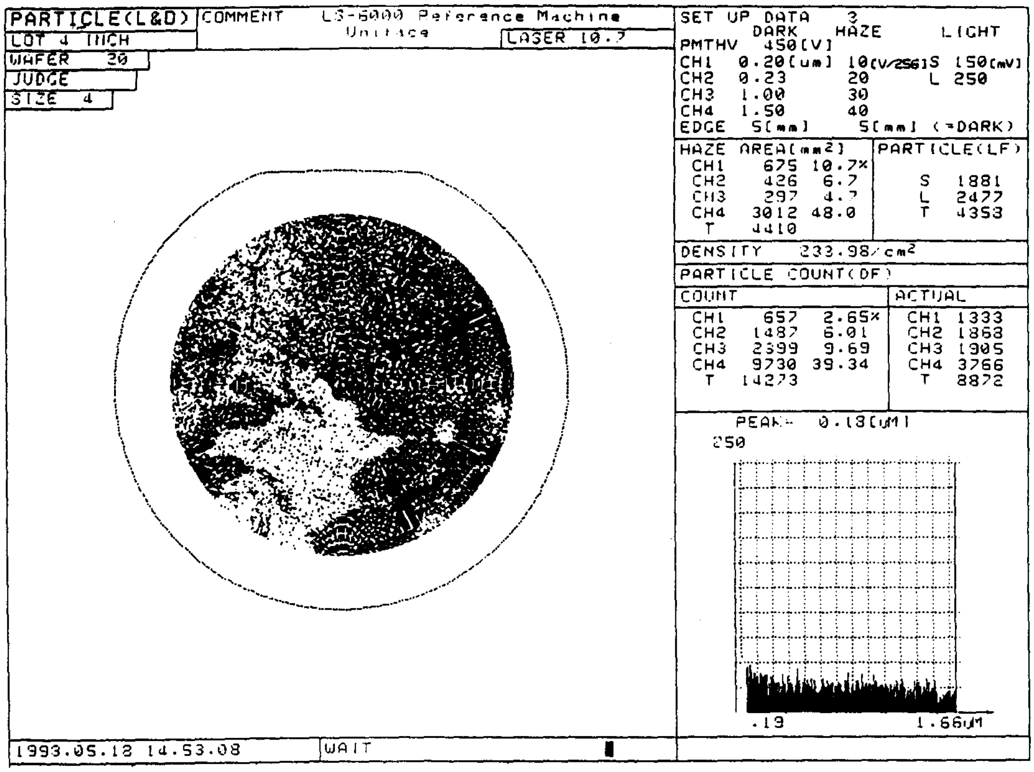

Fig. 14. Sample 3-2. After wetted with ultrapure water kept in the clean room for months, put on a 92 cm-high table outside the clean room for 10 min.

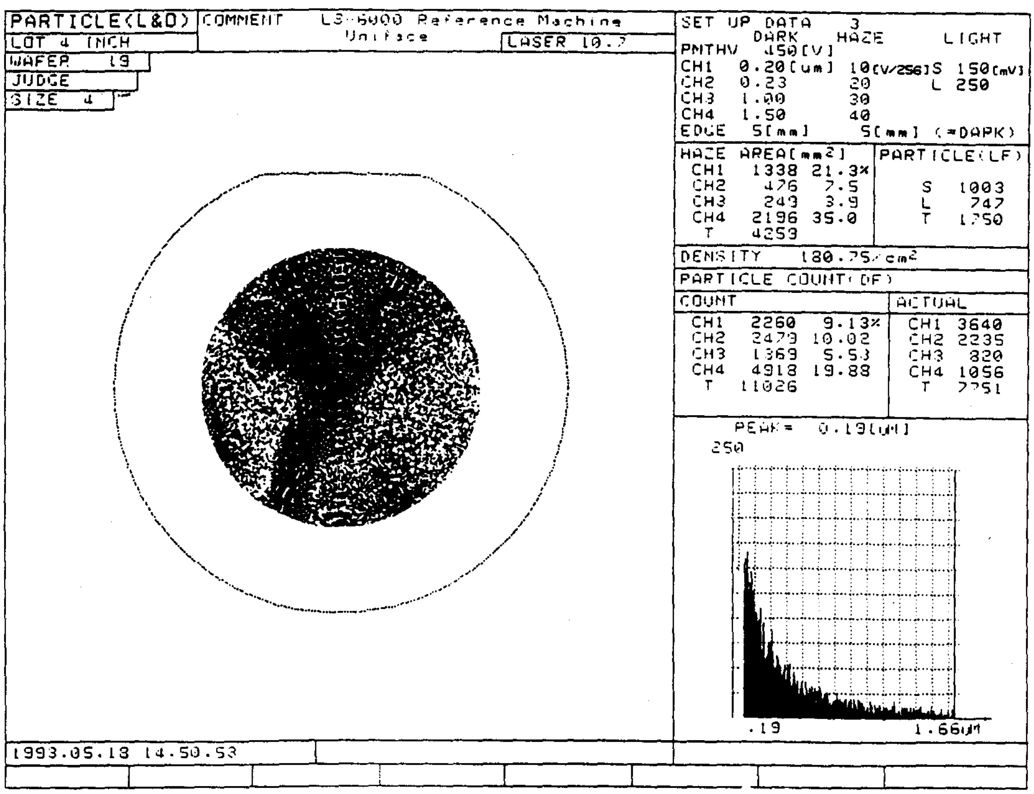

Fig. 15. Sample 3-3. After 3-1, blown by 5kg/cm^2 filtered N_2 gas in the clean booth.

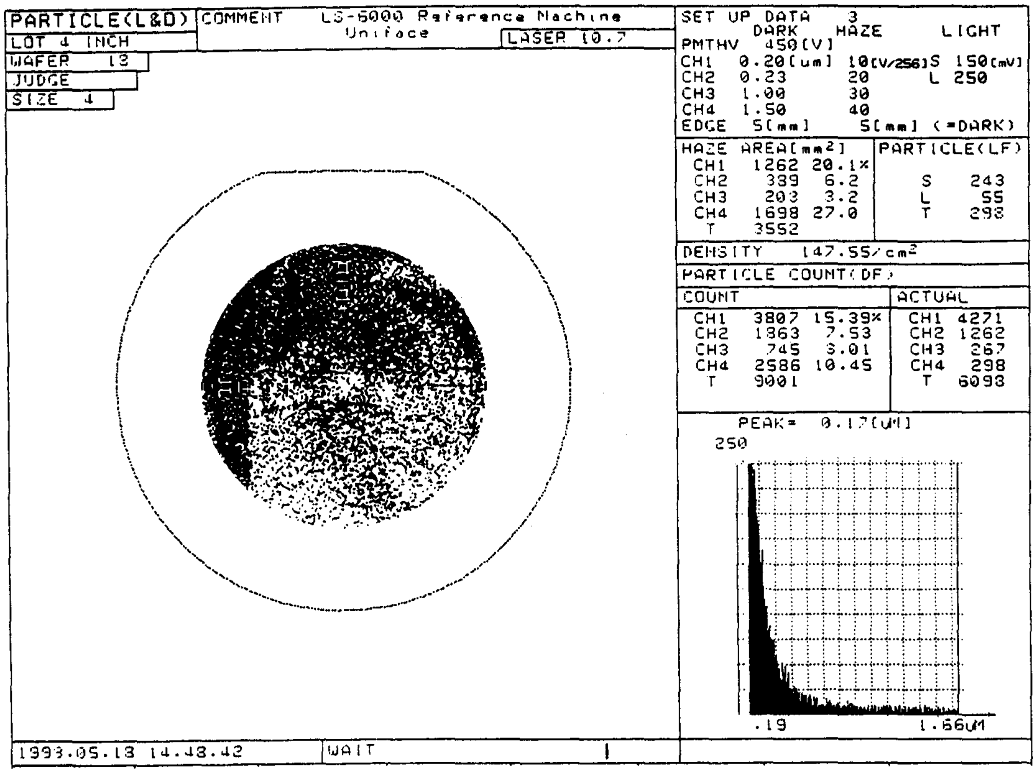

Fig. 16. Sample 3-4. After 3-1, blown with a portable duster, GUST II.

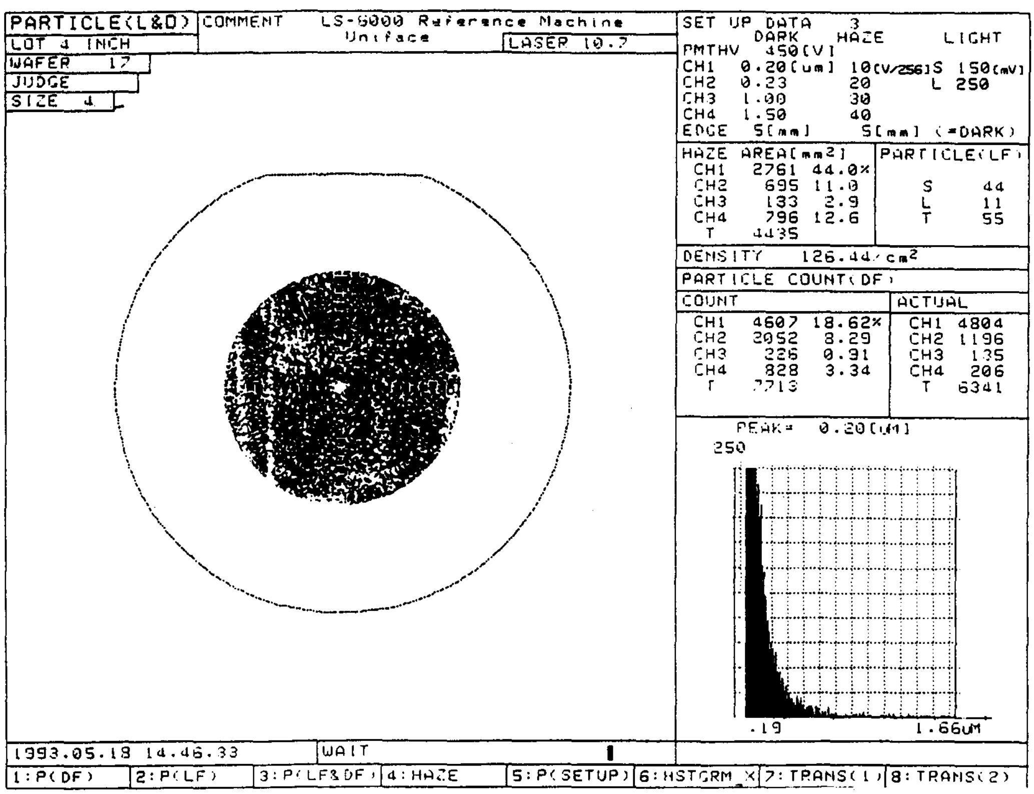

Fig. 17. Sample 3-5. After 3-1, blown with a neutralized gun named TOP GUN.

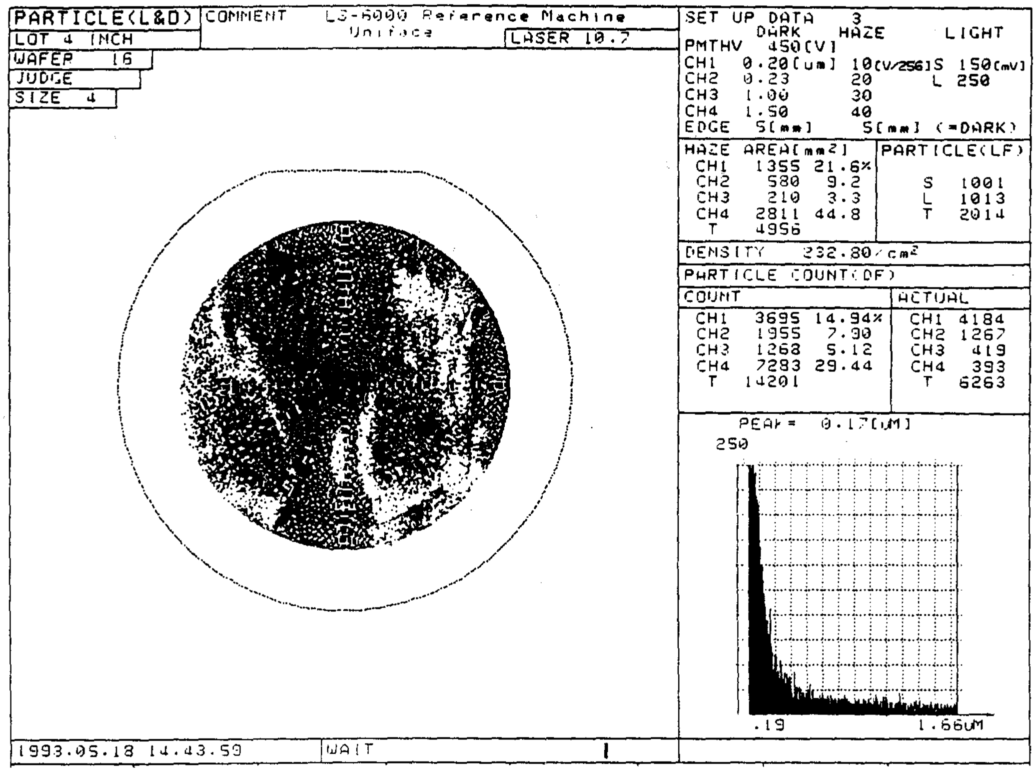

Fig. 18. Sample 3-6. Same as 3-1.

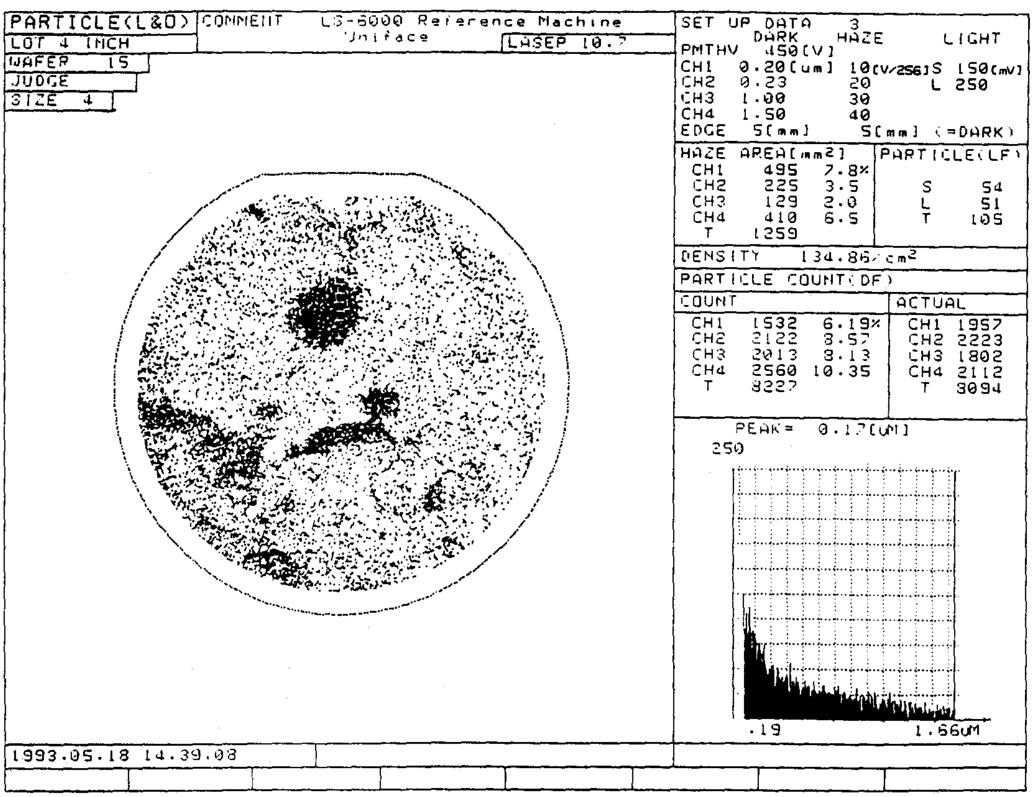

Fig. 19. Sample 3-7. After wetted with the ultrapure water kept in the clean room for months, put on a 92 cm-high table outside the clean room for 10 min.

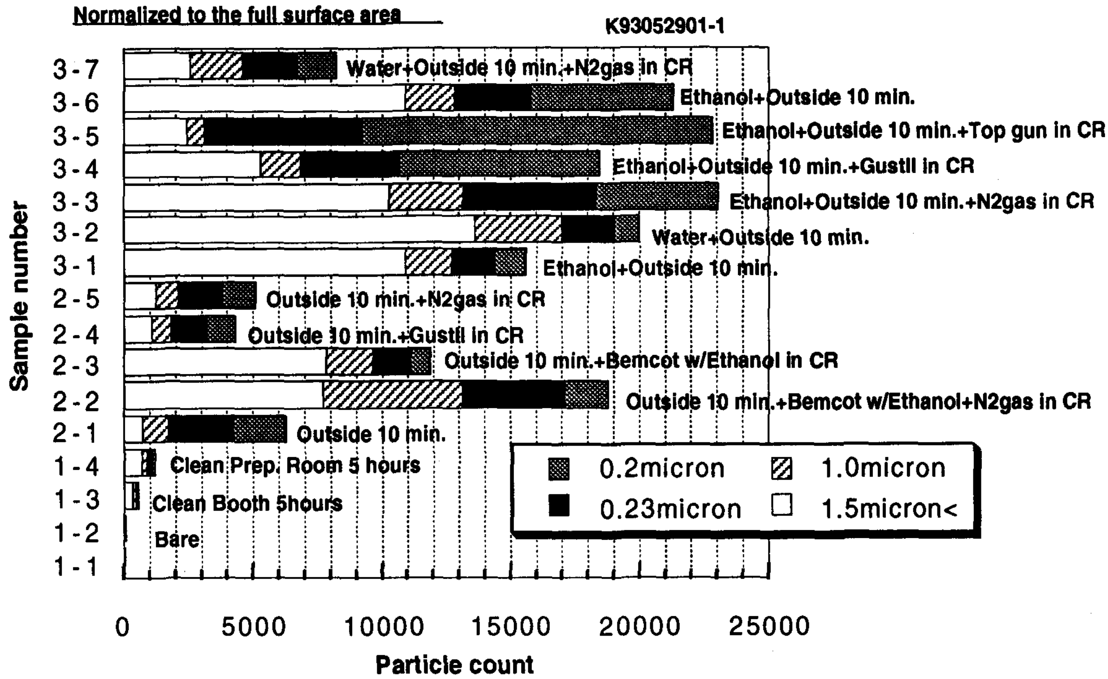

Fig. 20. Summary of the particle numbers after various conditions. Numbers are normalized to the full surface area because the counts sometimes stopped on the way as seen in the maps.

Before judging only from these data, we checked the particles in the cleaning fluid and ultrapure water similar to the one we used for the above-mentioned tests as well as some other chemicals as shown below.

Particles in ethanol used for cleaning

Usually only ethanol is used for assembling SC cavities in the clean room at KEK except when cleaning indium. In cleaning indium, we use HCl:Water=1:1 solution and ultrapure water. To check the particles in our ethanol and water, we counted the particles in the ethanol and the water we use, comparing with other chemicals.

Liquid particle counter

We used a liquid particle counter, KL-22 of RION Co. The min. measurable size is 0.2 µm. This counter shows the number of particles included in 10 ml liquid. This particle size is also poly-stylene equivalent size.

Measurement

We brought an unused ethanol into a class 100 clean booth, dusted off the bottle surface and opened. This ethanol, called special grade ethanol designated as Ethanol(S) below, is the one we use in our clean room most of the time. In the same way, two 2-Propanol bottles, 1st grade ethanol designated as Ethanol(1), ultrapure water kept in the clean room for 16 days, and a particle-controlled Methanol commonly used in semi-conductor industries designated as EL Methanol were prepared.

Results

Figure 21 shows the number of the different size particles. In every liquid, it was found that there are many particles, 14000-20000, having a diameter between 0.2 and 0.3 μm as shown in Fig. 21. Thus probably it is quite difficult to reduce the particles smaller than 0.3 μm. The particles in ultrapure water are probably bacteria because this water was kept in the clean room for 16 days.

Figure 22 shows the particles larger than 0.3 μm. From this figure, one can see that the ethanol we use in out clean room, Ethanol(S), has 500 0.3<D<0.5 particles, 100 0.5<D<1 particles and 15 1<D<2 particles approximately.

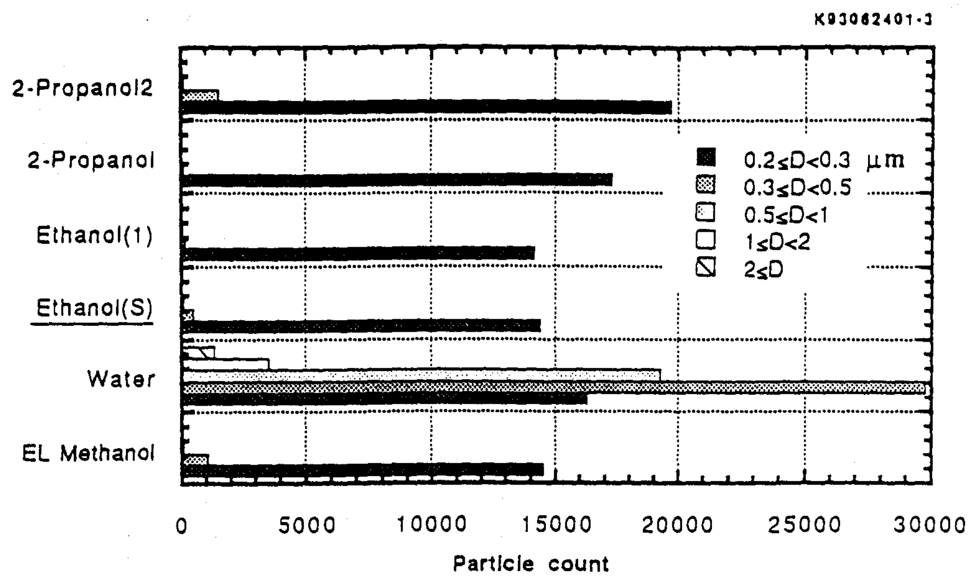

Fig. 21. Number of particles in cleaning ethanol, Ethanol(S), and in other chemicals as well as the ultrapure water kept in the clean room for 16 days.

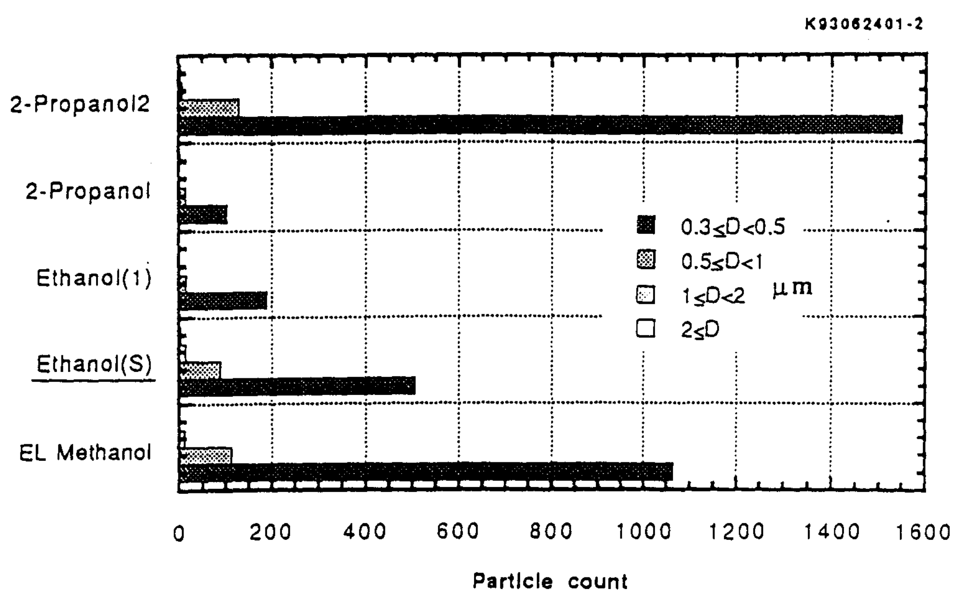

Fig. 22. Same as Fig. 20, but the particles smaller than 0.3 μm and the data on the water were omitted.

Conclusions

Precise particle count measurements showed that the cleanliness in the assembly room or clean booth is class 10-20 without people, but it increases momentarily by 2-3 orders of magnitude when 6 people are working. Among the things used in the clean booth, coats have most dust particles, about 1000/0.1CF.

Using a wafer inspection system, we studied the effect of various cleaning methods we use. The results showed that only 20-30 % can be taken away by 5 kg/cm^2 filtered N_2 or duster for the dry dust particles. When the wafer was wetted by either ethanol or water, the particle increased in number. Though there is still an ambiguity, wet samples seem to have gathered a few times as many particles as the dry samples, especially large particles, $1.5\mu m \leq$, increased in number. The ionized air gun had some effect to eliminate large particles.

The particle count of some chemicals and the ethanol used for the SC assembly showed that, in 10 ml, the number of $0.3\mu m >$ particles is 14000-20000, which seems to be difficult to be eliminated. The ethanol includes 500 $0.3<D<0.5\mu m$ particles, 100 $0.5<D<1\mu m$ particles and 15 $1<D<2\mu m$ particles approximately, which was almost same quantity or fewer than the ethanol whose particle number is controlled for the semi-conductor industries.

We are planning to study the material of dust particles and more effective cleaning methods in the future.

Acknowledgments

The authors would like to thank M.Kojima of Rion Co. for kindly lending us the particle counter. They also would like to thank K.Saito for letting us use the liquid particle counter. Discussions with other members of KEK SC group were useful. Continuous encouragement of E.Ezura, Y.Kimura, Y.Kojima and S.Kurokawa are greatly appreciated.

Reference

[1] Y. Kojima et al. ; Proc. 4th Workshop on RF Superconductivity, KEK, Aug. 14-18, 85 (1989).