Vacuum particle generation and the nucleation phenomena during pumpdown

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(Received 27 March 1989; accepted 28 October 1989)

This paper explores the source of an unexpectedly large number of particles found while examining the rough pumping cycles of a vacuum system. It is proposed that these particles are generated through nucleation of moisture onto fine particles during pumpdown, and that turbulence enhances this process. A brief discussion about the particle monitor used for data acquisition and a review of previous experimental results on the dependency of particle generation on turbulence is included. The body of the paper contains experimental results supporting the nucleation hypothesis, and a discussion on the effects and prevention of particle nucleation. The experimental results include a comparison of particle generation for two gases of similar original particle content but different humidity levels, reduction of particle count using a molecular sieve to remove the moisture in the chamber, and a systematic change of the relative humidity (by adding or removing moisture) in the chamber.

I. INTRODUCTION

In current microelectronics manufacturing, clean-room technology has been the primary approach for particle control. However, decreasing design rules and increasing circuit density have been posing more demanding cleanliness requirements.[1,2] To meet the stricter requirements, efforts are being made to carry out processes under vacuum and to implement automatic operations.[3–5] The aim is to eliminate human interaction and to minimize particle contamination.

In a previous work of Chen *et al.*,[6] a comparison study of clean room and vacuum demonstrated that vacuum can provide an intrinsically cleaner environment. It is also shown that once the vacuum environment itself is reached, it does not contribute particles. Of course, mechanisms moving in a vacuum and the process itself will inevitably generate some particles, but Bowling and Larrabee[7] found that in a high vacuum, the particles will fall quickly due to the force of gravity. Thus, wafers placed in a face-down configuration in a vacuum can self-shield their front faces from particulates.

However, during a process cycle, a wafer will inevitably experience pumping, vacuum processing, and venting operations. In Hoh's study,[8] it was shown that a wafer, after being subjected to fast pumping and venting processes, will be contaminated by numerous particles. In a recent review[9] on sources of particulate and atomic-scale impurities in vacuum deposition and etching systems, O'Hanlon also indicated pumping and venting as a particle generating source.

Recently, a particle flux monitor designed by high yield technology (HYT)[11] has made *in situ*, real-time monitoring possible in vacuum (e.g., in load-lock systems and ion implanters).[6,10,11] In Ref. 6, an unexpectedly large number of particles were found at the beginning of rough pumping. These were related to the existence of turbulence characterized by the Reynolds number. Their source or generation mechanism still needs to be examined. This paper will discuss the generation of these particles and an interesting phenomenon of moisture condensation, and relate the two to each other.

We will first discuss the physical measuring mechanism of the particle monitor we are using and the proper interpretation of its data. Then, we will review some of our previous experimental results on the dependency of particle generation on turbulence through time dependent Reynolds number (RE). The main discussion follows the examination of these results and the unexpectedly large number of particles detected during rough pumping. A working hypothesis is proposed: Moisture in the air tends to condense onto fine particles during pumpdown and turbulence enhances this process. Various supporting experimental results are then described, taken under conditions of systematically varied ambient humidity and back-filling methods. Finally, the effects of these nucleated particles and the prevention of nucleation are discussed.

II. THE VACUUM PARTICLE COUNTER AND ITS MODELING

The particle counter used in this study is the PM-100 flux monitor (HYT),[11] developed for use in vacuum. The system includes a sensor head, preamplifier, and controller. The sensor is a compact, self-contained probe that is designed to fit directly into process chambers and pumping lines. The optical system uses a laser beam that is reflected back and forth between two parallel mirrors, creating a light net. Particles passing through this net will scatter light, which is collected by two photodiodes mounted above the two mirrors, giving an electrical impulse signal. Due to considerations for digitization and prevention of repeated counting, a discrete sampling method has been used, with 0.01 s sampling intervals. In each interval, if there is at least one impulse larger than the preset threshold, the controller accumulates one particle count, otherwise there is no particle count. In this way, the particle counter can count a maximum of 100 particles per second. When two or more particles pass through the light net together, the sensor will give only one count. If the actual particle flux is too high, the counting efficiency will drop quickly, resulting in measurement saturation.

Assuming that the movement of individual particles is independent, the number of particles passed in time Δt_1 is also independent of that in Δt_2, if Δt_1 and Δt_2 do not overlap. Then, the number of particles passed through the light net in Δt has a Poisson distribution[12]:

$$\text{Prob}(k \text{ particles}) = \frac{[\Delta t f(t)]^k}{k!} e^{-\Delta t f(t)}, \tag{1}$$

where $f(t)$ is the instantaneous particle flux which is assumed relatively constant during Δt. With this model, given the flux $f(t)$ and the sampling interval Δt, the counting efficiency of the monitor (the ratio of the recorded particle count to the number of particles passed through the light net) can be obtained as

$$E[f(t)] = \frac{1 - e^{-\Delta t f(t)}}{\Delta t f(t)}. \tag{2}$$

When $f(t)$ is very small, the efficiency is approximately $1 - 0.5\,\Delta t f(t)$, i.e., decreasing from 1 as a linear function of $f(t)$. However, when $f(t)$ is very large, a closer approximation is $1/[\Delta t\, f(t)]$, i.e., inversely proportional to $f(t)$ and *approaching zero* very rapidly.

On the other hand, once a measurement has been taken using the HYT monitor, we can calculate the maximum likelihood estimation of the actual particle flux by the following formula:

$$\hat{f}(t) = 100 \ln\left[\frac{100}{100 - f_m(t)}\right], \tag{3}$$

where $f_m(t)$ is the indicated particle flux by the particle monitor. Figure 1 shows the relationship between the estimated and the measured particle flux, together with the relative error in the reading. Due to the low efficiency of the monitor at high flux, it is not surprising to note that when the indicated flux is high and approaching the saturation level, the corresponding actual particle flux is extremely high. The nonlinearity makes a remarkable difference in interpreting the experimental data.

III. VACUUM PARTICLE DYNAMICS

In preparation for the main discussion, the following is a review of previous experimental results on the effects of turbulence on dynamic particle counting. As presented in Sec. IV, this effect is one of the two major factors responsible for particle generation. Details can be found in Ref. 6.

A. Dynamic particle counting during pumping

In the previous work, dynamic particle counting was investigated using a small load-lock vacuum chamber located in a class 100 clean room. To concentrate on the pumping effect, the sensor head was placed directly above the rough pumping chamber inlet, at the point of greatest air flow and where largest particle counts were expected to occur. Well over 1000 particles were observed during the initial rough pumping stage when the air flow was large and turbulent.

To examine the quantitative relationship between turbulence and particle count, the relationship between the Re[13] and the pumping speed is required. For a constant volume vacuum chamber, the Re at the chamber pumping port can be derived as[6]

$$\text{Re} = -\frac{4V}{\pi\mu D}\frac{\rho_0}{P_0}\frac{\Delta P}{\Delta t}, \tag{4}$$

where: P is time dependent pressure in the chamber, V is volume of the chamber, t is time, D is diameter of the chamber pumping port, m is viscous coefficient of air, P_0 is air pressure at normal condition, and ρ_0 is air density at normal condition.

This relationship is used to calculate the Re during the rough pumping process. Figure 2 shows a typical result of particle count versus time dependent Re during a single pump down. It can be seen that once Re < 100 very few particles are counted; however, when Re > 2000, particle counts are at the sensor saturation level. The particle count increases with the Re in the transition region.

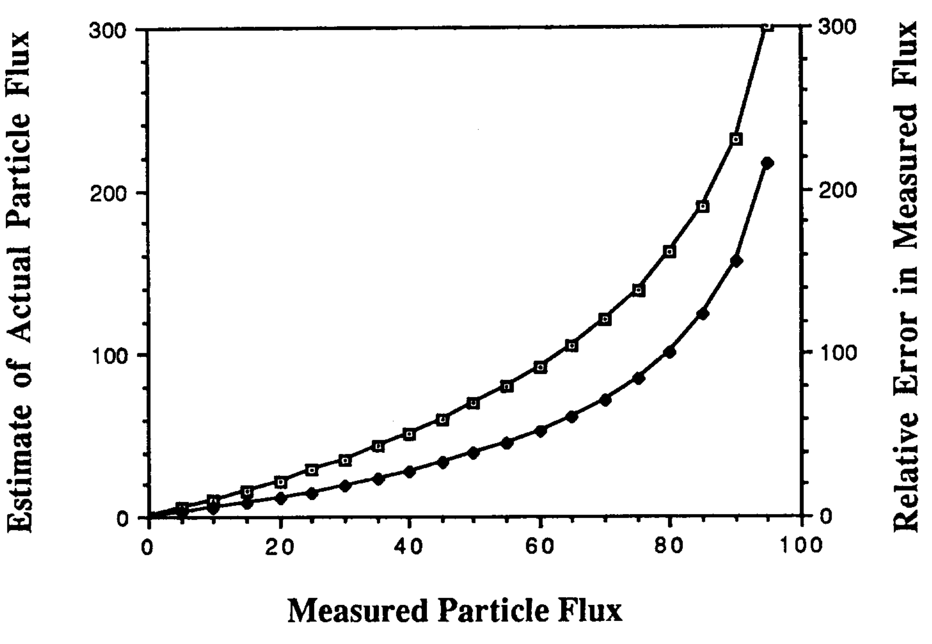

FIG. 1. The maximum likelihood estimation of the actual particle flux vs the indicated particle flux by the HYT particle monitor. -□- actual flux, -◆-error.

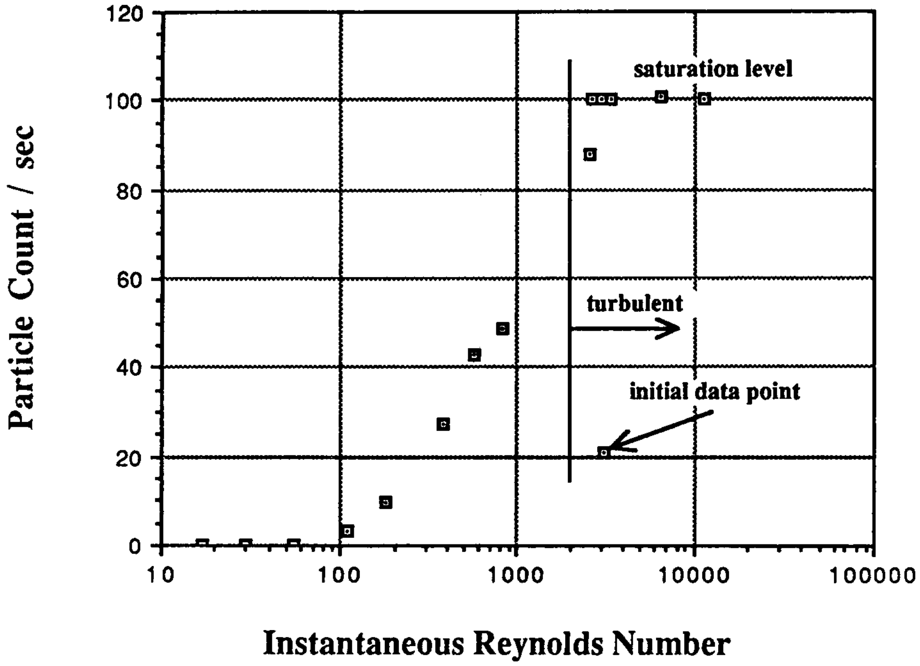

FIG. 2. Measured particle flux vs instantaneous Re during a typical pumpdown using fast pumping.

B. The effect of pumping speed

We have also systematically studied, using an adjustable throttling system, how the particle count changes in relation to pumping speed. Figure 3 shows that total particle counts are reduced as the pressure curves are made more gradual, i.e., slower pumping speed.

The dependence of particle count on Re is an independent relationship that cannot be explained based on pressure or time alone. For example, in Fig. 3, at a pressure of 300 Torr, the particle count is markedly different over the three trials. Similarly, from the same figure, at a single point in time (e.g., 15 s after pumping begins) the particle count is different from trial to trial. While pressure or time alone cannot explain the relative particle count levels, a combination of pressure *and* time, as formulated through the Re, does seem to relate directly to the particle count.

Also in Fig. 3, an interesting effect has been demonstrated by using three pumping speeds. There is little difference between the medium and slow pressure curves, yet there is a major difference between the corresponding particle curves. The reason for this is that the Re corresponding to these two pressure curves are in the transient region, so a small change in the pressure curve will cause a major change in the particle count curve. Also, note that the particle curve for the fast pumping speed corresponds to a very large number of particles, especially if the saturation effect is taken into account.

IV. THE NUCLEATION PHENOMENA DURING PUMP DOWN

Having discussed the particle dynamics, we now concentrate on a phenomenon which is implied by the previous experimental results: nucleation during pump-down. We

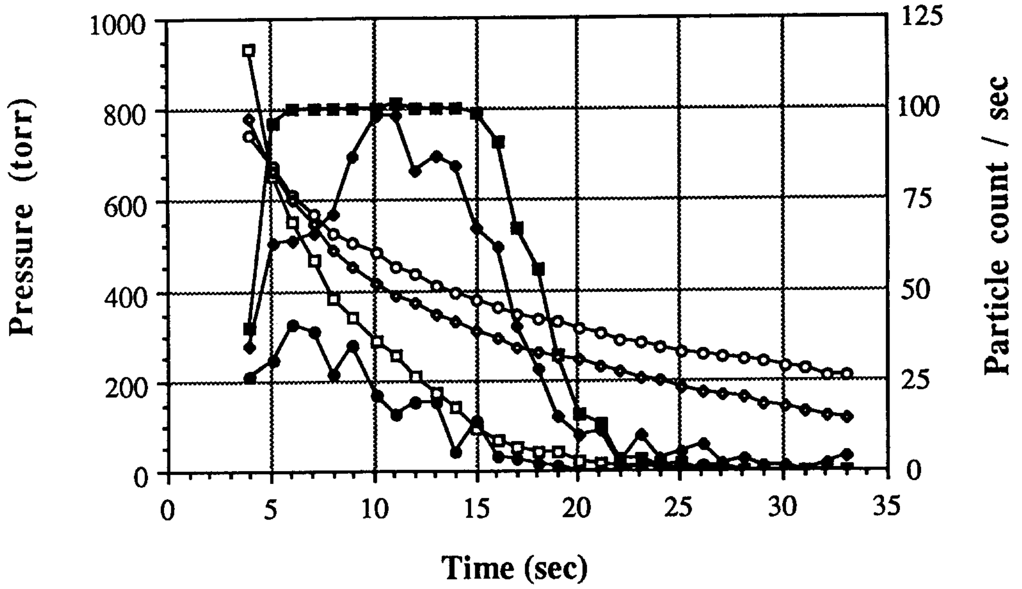

FIG. 3. Pressure and corresponding particle count vs time for three different pumping speeds. -□-pressure fast, -◇-pressure med, -O-pressure slow, -■-particle fast, -◆-particle med, -●- particle slow.

first reexamine the unexpectedly large number of particles observed during rough pumping and propose a nucleation hypothesis. Then, we present experimental results related to the investigation of this phenomenon.

A. The nucleation hypotheses

In examining Fig. 2 and the "particle fast" curve in Fig. 3, we notice that a large number of particles (well over 1000) are pumped out during the beginning stage of rough pumping when there is a large air flow and turbulence is expected. Furthermore, Fig. 1 shows that when the measured particle flux is near the saturation level, the actual flux is much higher. Thus, the total number of particles pumped out may be several thousand. In these experiments, the small chamber (~ 0.5 ft^3) is initially filled with air from the Class 100 clean room whose cleanliness is verified using a laser spectrometer. Thus, only 50 or fewer particles larger than 0.5 μm are expected to be present in the chamber. However, since the HYT particle monitor has a low counting efficiency, only a few particles are expected to have been counted by the monitor. Thus, a discrepancy exists. Furthermore, repeated experiments have shown that this phenomenon will occur consistently as long as the same backfill source and the same fast pumping speed are used. Thus, it is important that we explain the discrepancy and understand the underlying mechanism.

One possible theory is that the unexpectedly large particle count is due to particles dislodged from chamber walls by turbulence. If this is true, a lot of particles should be counted during the first pumping cycle, and a decreasing number during subsequent pumping cycles. This is not the case. No significant change in particle count is noted over successive pump-down cycles as long as the same air source is used in venting. Furthermore, attempts to clean the chamber walls do not change the results. Another theory, that of multiple counting, is also unlikely. As noted, the number of detectable particles expected is very small (~ 5). To achieve a count of more than 1000 particles, these several particles would have to be suspended in a cyclic motion pattern near the detecting light net without being carried away by the strong pumping airflow. This situation is also deemed unlikely.

The proposed theory is that during pump down, the proper conditions exist for particles to be generated by means of nucleation. The number of "fines" (particles < 0.5 μm) is very large, and fines as small as 0.005 μm are capable of serving as condensation nuclei.[14] During rough pumping, extraction of the air from the chamber is, in a sense, equivalent to undergoing a sudden volume expansion. According to thermodynamics, this sudden expansion will cause a temporary temperature drop in the air, which will in turn cause the relative humidity in the chamber, if originally filled with moist air, to increase to saturation level. Thus, the water vapor from the clean room air will tend to condense onto the fines through heterogeneous condensation (which is much more likely than homogeneous condensation with the presence of fines as nuclei). The presence of turbulence will trigger the nucleation quickly, since these wetted fines will collide and coagulate with each other, thus growing to sizes that are detectable by the HYT monitor.

It should be emphasized that turbulence is playing an important role in the particle generation. Turbulence and moisture are two necessary conditions. In our case, the whole rough pumping process is relatively short (~ 20 s). In the case of laminar flow, condensation begins slowly so that nucleation will not occur in such a short time. But, if the air is turbulent, nucleation could happen in 1–2 s. If the turbulence stops, the nucleation will also stop within seconds. The total time that the turbulence lasts will determine the total number of particles nucleated. The previous results have revealed a strong relationship between particle count and turbulence. The key point is that moisture is a necessary condition and turbulence triggers the nucleation.

In the following subsections, we will describe some related experimental results to support the above hypothesis.

B. The effect of backfilling with dry nitrogen

In an attempt to reduce particle contamination during pumping and to prove or disprove the nucleation hypothesis, many experiments were performed using various backfill gases with different moisture contents. In one experiment, it was found that backfilling with dry nitrogen dramatically reduces the particle count compared to backfilling with clean-room ambient air.

In the experiment, the venting port was connected to a dry nitrogen source through a control valve. The chamber, which was initially filled with clean-room air, was closed and pumped down (see Fig. 4). After a pressure of about 10^{-4} Torr was reached, the pumping valve was closed and the chamber was vented with dry nitrogen until atmospheric pressure was reached. It was then pumped again from a dry nitrogen-filled state. In the experiment, this pumping sequence was immediately repeated, i.e., after pumping down the nitrogen-filled chamber to a certain pressure, the chamber was vented with clean-room air and pumped down, and then vented with nitrogen and pumped down again. The maximum pumping speed was used during each trial to ensure that turbulence was created (this is a basis for the experiments in the rest of this paper). In doing this, it was verified that: The experimental results have good reproducibility, and the large number of particles detected with clean-room-air backfill and the reduction of particle count with nitrogen backfill have little to do with the pumping history. Figure 6 shows how particle counts change greatly (from a total number of > 1000 to < 10 particles) when a dry nitrogen backfill is used.

To compare the clean-room air and the dry nitrogen, a laser spectrometer particle counter was used to verify that the two gases indeed have a similar particle distribution for diameters 0.12 μm and above. Following a careful calibration, the particle counter was programmed to record the particle counts for each of ten different particle size channels. First, the clean-room air was sampled for 2 min, which was repeated several times to check the reproducibility. The dry nitrogen was then sampled for 2 min. When sampling the nitrogen, a T-junction was used, with one opening connected to the sampling probe, the second to the nitrogen source,

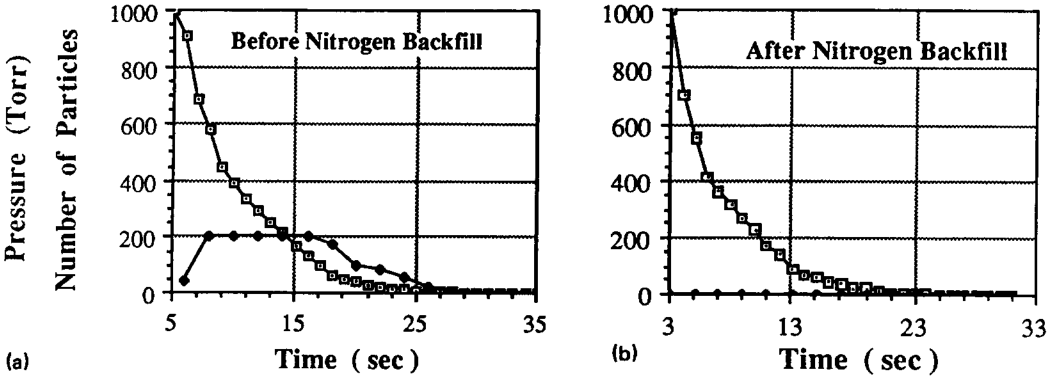

FIG. 4. Pressure and corresponding particle count vs time, comparing (a) clean-room air backfill with (b) dry nitrogen backfill -□-pressure, -◆-particle.

which is releasing more nitrogen than the probe can take, and the third exposed to atmospheric pressure for venting the excess nitrogen. This was done to ensure the same sampling pressure for both gases. Reproducibility was also checked. These experimental results are shown in Fig. 5.

From the figure, we can see that nitrogen and clean-room air have very similar particle contents. But there is a major difference in moisture content: The nitrogen is from a high pressure source and it is dry, but the clean-room ambient air is comparatively wet, having a relative humidity of over 70%. Thus, it is probable that the generation of the large number of particles when pumping ambient air is related to the moisture content in the air.

From the results of this experiment, a recommendation regarding the operation of vacuum systems can be made. In a system in which pumping and venting occur frequently, such as in a load-lock system, it is beneficial to use dry nitrogen every time venting is necessary in order to reduce particle contamination.

C. Experiments using a molecular sieve

After studying the previous experimental results, it becomes more clear that moisture content is playing an important role in particle generation during rough pumping. To investigate this more directly, the moisture content is removed from the same clean-room ambient air, using a molecular sieve. The molecular sieve used, which is a very efficient and fast drier, is of type 4A, with a cylindrical shape and a length of 1.6 mm. Since the chamber volume is known, the amount of molecular sieve can be precalculated to bring the relative humidity down to below 1%, assuming an initial relative humidity of 70% and temperature of 25 °C. The desired low humidity is reached only after a sufficient waiting period, since it requires time for the sieve to absorb the moisture. Since the waiting period is directly related to the amount of moisture removed, a correlation should exist between waiting period and particle reduction if the nucleation hypothesis is true. Thus, we should be able to observe a gradual reduction in the total number of generated particles as a function of the waiting time. When the waiting time is sufficiently long, so that most of the moisture content has been absorbed by the sieve and hence no nucleation is possible, this function should approach five, the expected number mentioned before.

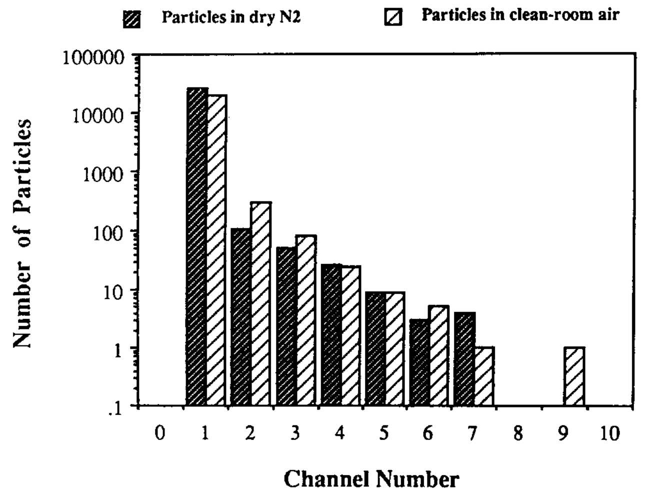

FIG. 5. Comparison of initial particle size distributions before pumping of clean-room ambient air and dry nitrogen. Channel size (μm) (1) 0.12–0.17, (2) 0.17–0.27, (3) 0.27–0.42, (4) 0.42–0.62, (5) 0.62–0.87, (6) 0.87–1.17, (7) 1.17–1.52, (8) 1.52–1.92, (9) 1.92–2.37, (10) over 2.37.

In the following experiments, the molecular sieve is wrapped with lint-free paper. The paper is not airtight, so that water vapor can penetrate the paper and be absorbed by the sieve. On the other hand, the paper efficiently prevents particles in the sieve from entering the chamber air. When the moisture is absorbed by the sieve, some heat will be released. Most of this heat will remain in the sieve or be transferred to the chamber floor, causing some temperature rise. Although some of the heat will be transferred to the air, we assume that we can neglect its effect on nucleation, since this part is relatively small.

First, the effect of using the molecular sieve inside the chamber was tested. Each time about 50 g of molecular sieve, which is more than twice the amount needed to absorb all the moisture in the chamber, was placed in the middle of the chamber wrapped with lint-free paper. For comparison, a preliminary trial was run with no molecular sieve, recording the total number of particles. Next, the molecular sieve was placed inside the chamber, and the chamber was quickly pumped down and vented with the same clean-room ambient air, but no data was taken. As soon as atmospheric pressure was reached, the chamber was pumped down again, this time recording the total number of particles generated during rough pumping. The entire process was repeated twice more. In the first trial, after the chamber was vented to air pressure with a molecular sieve inside, there was a 5 min wait before the chamber was pumped down again and data taken. In the second trial, the waiting time was increased to 20 min. In each trial, a new molecular sieve was used to ensure proper comparison.

These experimental results are shown in Fig. 6. It should be noted that in each experiment the actual absorbing time is the waiting time plus the preceding venting time. Since the absorption during venting is always changing and is different from that during waiting, it is not appropriate to add the two to get the effective absorbing time. Thus, the graph is not made to a linear time scale. It can be seen from the figure that the particle count is reduced by more than half even with no waiting time. This means that a significant amount of moisture is already removed by the sieve during venting. It is important to notice that the particle count does reach four when the waiting time is 20 min.

After these experiments, we then tested the total particle count by using the molecular sieve in the venting line. In this way, air was forced through the sieve during venting, with some of the moisture absorbed by the sieve. Thus, less moisture enters the chamber. In the experiments, the chamber was first pumped down to vacuum. Then, after some molecular sieve was put in the venting line, the chamber was vented to air pressure and pumped down again with the total number of particles recorded. Two trials were taken. In the first trial, 20 particles were observed, and in the second one 57. At first glance, the two trials resulted in a widely different number of particles. But in comparison with the number (1233) obtained when no molecular sieve was used, these two numbers are in fairly good agreement.

From these tests with a molecular sieve inside the chamber or in the venting line, we conclude that if moisture is removed from the air in the chamber, the total number of particles observed during pumping will be reduced accordingly. This conclusion strongly supports the nucleation hypothesis.

D. Other nucleation experiments

To further verify the effect of moisture content on particle generation during rough pumping in the presence of turbulence, other experiments were also performed. In one set of experiments, we studied the particle generation by employing backfilling gases with systematically changing moisture content. In each experiment, the chamber was first pumped down, vented with gas of a different moisture content, and then pumped down again, with the total number of detected particles being recorded.

These results are shown in Fig. 7. The dry gases were obtained from pressurized gas sources. One method of introducing extra moisture into the chamber is to bubble the air through water, and collect and use the wet air for venting. The principle concern in doing this was that the particle content might be changed since the water might serve as a particle filter or the impurities in the water might be carried into the air. Instead of bubbling, we introduced extra moisture (more than that in the clean-room ambient air) into the chamber by creating a local humid climate for venting. In the experiment, a water surface was temporarily introduced near the chamber, creating a local area of higher humidity right above the water surface. By changing the temperature of the water, a changing local humidity can be achieved. Then the venting probe was placed about half an inch above the water surface. Thus, air of high moisture content could be used to vent the chamber without changing the particle content of the air. A test similar to that done for the comparison of clean-room air and dry nitrogen was also performed to verify that all the venting gases had a similar original particle content.

From Fig. 7, we can see that higher humidity in the chamber will result in more particles during pumping. A systematic reduction in the moisture content will give a systematic reduction in the total particle count. With a virtually dry gas content, there will be no extra particles being generated by rough pumping. That is, the total particle count will be limited to the expected minimum amount even if the fastest pumping speed is used.

Another set of experiments involved the use of liquid nitrogen to cold-trap the water vapor inside the chamber before pumping. In each trial, after the chamber was filled with

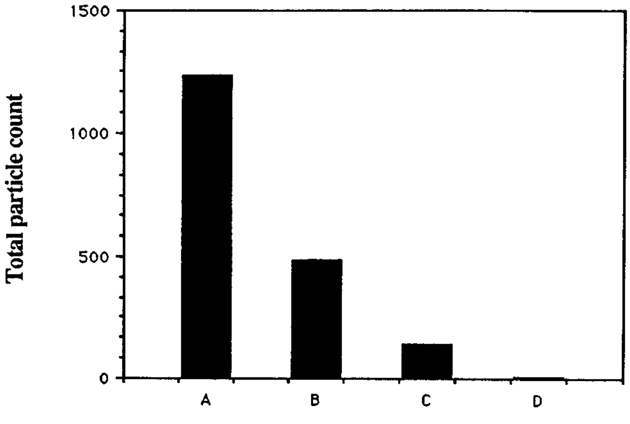

FIG. 6. Particle count during each pumping obtained in experiments using molecular sieve dryer in the chamber: (A) normal pumping without use of sieve, (B) sieve in the chamber during venting, followed immediately by pumping, (C) sieve in the chamber, pumped 5 min after venting, (D) sieve in the chamber, pumped 20 min after venting.

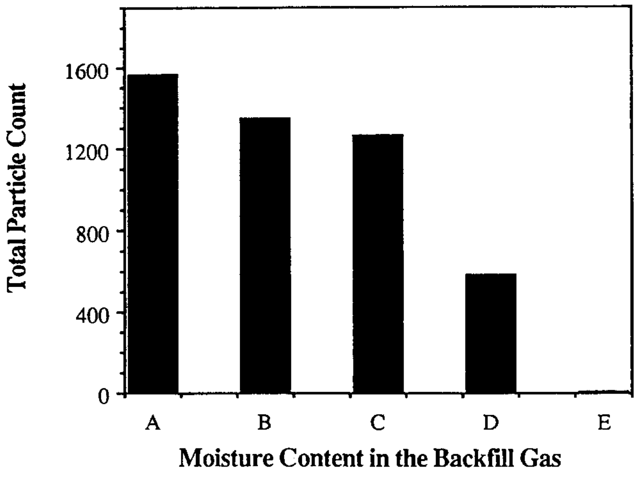

FIG. 7. Particle count during each pumping using backfill gases of different moisture contents: (A) air drawn near hot water surface, (B) air drawn near cold water surface, (C) normal clean-room air, (D) compressed air, (E) high-pressure-dried air.

clean-room air and tightly closed, a proper amount of liquid nitrogen was put into the cold trap near the back of the chamber. After waiting for a certain time for the trap to attract the water vapor from the air, the chamber was pumped down and the total particle count was taken. Venting followed and the system was allowed to recover to its original condition for further trials. This testing was repeated many times and an example of the results is shown in Fig. 8.

Some problems exist in relation to these tests using liquid nitrogen cooling: (1) In a physical vacuum system, this cooling may not be appropriate. (2) Even if allowed, when the chamber is large (e.g., the SCARF[5] chamber at the CRSM), it becomes a very expensive practice, especially if the chamber is frequently vented and pumped. (3) By using cooling, the number of particles is reduced during pumping, but the water vapor has become ice and it remains in the chamber. This makes it very difficult to pump the chamber to high vacuum, because the ice keeps evaporating. Furthermore, this residual gas of water vapor may not be good for some physical or chemical processes. (4) By using a liquid-nitrogen cold trap, the temperature of the air inside the chamber is reduced, making it more difficult to evaluate the study of the nucleation. Besides, this cooling causes the air inside the chamber to contract and the pressure to decrease. Thus, to have the same initial pressure before pumping, the venting valve should be slightly open so that some outside air can enter to maintain atmospheric pressure. Therefore, the amount of air to be pumped in this case is a little more than that in normal pumping. The longer it is cooled, the more air must be pumped. Another important point is that in order to get good comparable data, one has to wait long enough for the system to completely recover to its original condition before the next experiment can be done, which increases the experimental time and increases difficulties to have good reproducibility.

Despite all these problems, the results in Fig. 8 do clearly indicate a consistent particle reduction due to longer cooling. It is of interest to notice a phenomenon that occurs when the waiting time is insufficient after a cooling test. We repumped, without using liquid nitrogen, while the chamber was still cool and 1666 particles were counted: about 30% more than in a normal fast pump cycle. Our explanation for this is as follows. Since no liquid nitrogen was used, no icing would occur and no moisture would be removed from the air. Thus, when the clean-room air came into the chamber, the relative humidity of the air would increase due to the cool chamber. This would make the nucleation process occur more easily and thus more particles were generated.

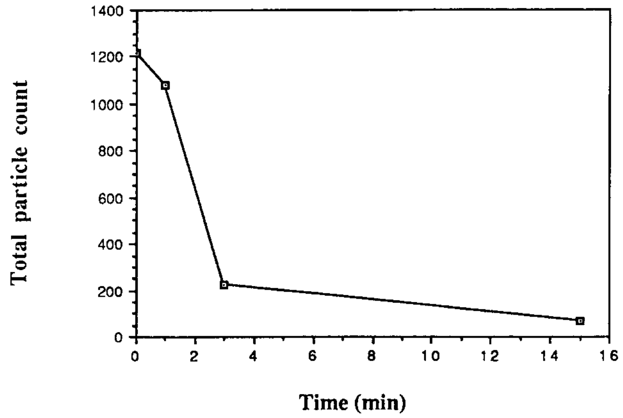

FIG. 8. Particle count during each pumping vs the corresponding cooling time before each pumping.

E. Final remarks on nucleation

From the above results, one common conclusion is that nucleation during rough pumping is highly possible. All the experiments strongly support the hypothesis, although these can only serve as indirect verification. To directly prove the occurrence of the nucleation requires direct *in situ* observation of the nucleation process.

An improved verification of the nucleation hypothesis may be performed through more quantitative experiments, e.g., by obtaining more quantitative data on relative humidity, temperature, and pumping speed. It would be very useful to obtain a detailed quantitative description of particle count as a two variable function of both relative humidity and pumping speed. Then, we would be able to choose the maximum pumping speed based on the measurement of the relative humidity in order to avoid the generation of a large number of particles and at the same time achieve the fastest pumping possible. This is desirable, especially when the chamber is large. Computerized automatic control of the pumping can then be used to ensure the optimal pumping speed.

Of course it is important to clarify the occurrence of nucleation, but it is even more important to study the relation between nucleated particles and wafer defects. It can be visually observed that these nucleated particles will contaminate wafer surfaces. In Hoh's paper,[8] photographs clearly show numerous particles on a wafer surface. However, it is difficult to study how these particles differ from normal particles in causing fatal defects in integrated circuits.

It would also be beneficial to study the various physical properties, such as the optical characteristics, of the nucleated particles for better detection and control. Another topic of interest is a study of the reevaporation of the moisture after the chamber has been pumped to vacuum, i.e., a study of residual gases in the chamber.

In summary, with the simultaneous presence of both turbulence and high relative humidity, a violent nucleation process is likely to happen during rough pumping. The appearance of this phenomenon needs to be avoided due to its negative effects: (1) Nucleation is observed to generate a large number of particles during pumpdown. These nucleated particles can contaminate a wafer surface. (2) Due to their wet surfaces, these particles are more likely to stick to a wafer surface. Due to their different nature, they may increase the difficulty of wafer defect studies. (3) Even if the water around the deposited wet particles evaporates in a vacuum, the residuals may still cause defects. Furthermore, due to coagulation, these residuals are usually much larger than the fines. (4) The use of vacuum as a clean environment is based on the fact that all the particles will be pumped out. However, when nucleation occurs, fine particles grow into much larger ones and become much more difficult to pump out. Instead, they are more likely to fall down and remain in the chamber, making the chamber less clean. (5) This deposition of large, water surrounded particles will keep more water in the chamber, which is undesirable in most cases and which makes the chamber more difficult to pump. (Water vapor is one of the most difficult gases to pump.)

To avoid nucleation, we recommend using computer controlled pumping to avoid turbulence, using dry gas (e.g., dry nitrogen) backfill, using a drying agent in the chamber, or using an efficient moisture filter in the venting line to remove moisture.

V. SUMMARY AND CONCLUSION

Following a discussion of data interpretation for the HYT particle monitor and a review of our previous results, we have explored the explanation for the observed, unexpectedly large, number of particles at the rough pumping stage when the chamber is backfilled with clean-room ambient air. A nucleation hypothesis has been proposed as follows: During pumping, moisture in the air will tend to condense onto fines, and the presence of turbulence will trigger and enhance the condensation process, causing the fines to quickly grow into particles of supermicron sizes. This is supported by the following experimental results. (1) When the chamber is prepared with moist clean-room air, there is a clear relationship between particle count and the Re, indicating that turbulence corresponds to the generation of a large number of particles. Completely avoiding turbulence results in a large reduction in particle count. (2) Backfilling with dry nitrogen leads to a great reduction in particle count, although the nitrogen used has a very similar particle distribution to that of the clean-room air. (3) The use of a molecular sieve indicates that the reduction in particle generation is clearly dependent on how long the sieve is in the chamber, i.e., dependent on how much moisture has been removed from the chamber. (4) Finally, a systematic change in total particle count has been achieved by backfilling with air of variable humidity content. The results of the use of liquid-nitrogen cold trap also agree with the nucleation hypothesis.

While these results all support the nucleation hypothesis, more quantitative experimental data and direct experimental observation of the nucleation process will be pursued in future work.

ACKNOWLEDGMENTS

The authors would like to thank Dr. T. Seidel of Sematech (formerly of the CRSM), S. Belinski of CRSM, Dr. E. Hu of UCSB, and Dr. T. C. Smith of Motorola for stimulating technical discussions. The authors would also like to thank B. McGinley of High Yield Technology for technical assistance, and Sputtered Films, Inc., for providing the load-lock system and the necessary support information. Special acknowledgment goes to Hideharu Zenpo of Yaskawa Electric Co., in Japan for assistance with nucleation experiments while he was a visiting researcher at the CRSM. The work described here was carried out under National Science Foundation support Contract No. 8421415.

[1] J. Berger and D. Bahr, Surface Particle Detector Monitors Work Station Air Quality, Microelectronic Manufacturing and Testing, Contamination Control Report, February 1988, Vol. 11, No. 2, p. 19.

[2] T. Hattori, Total Contamination Control for ULSI Wafer Processing, Microelectronic Manufacturing and Testing, Contamination Control Report, April 1988, Vol. 11, No. 5, p. 31.

[3] S. Belinski and S. Hackwood, Automated Manufacturing in a Vacuum Environment, in proceedings of IEEE International Electronic Manufacturing Technology Symposium (IEEE, New York, 1987).

[4] S. Belinski, W. Trento, R. Imani, and S. Hackwood, Robotic Design for Vacuum Environment, in proceedings of NASA Workshop on Space Telerobotics, Pasadena, CA, June 1987, p. 95.

[5] T. Seidel, Self-Contained Automated Robotics Factory (SCARF), CRSM Internal Memo, 1987 (unpublished).

[6] D. Chen, T. Seidel, S. Belinski, and S. Hackwood, J. Vac. Sci. Technol. A 7 3105 (1989).

[7] R. A. Bowling and G. Larrabee, Particle control for semiconductor processing in Vacuum Systems," in Microcontamination Conference Proceedings Canon Communications, Inc., Santa Monica, CA, 1986, p. 161.

[8] P. D. Hoh, J. Vac. Sci. Technol. A **2**, 198 (1984).

[9] J. O'Hanlon, J. Vac. Sci. Technol. A **5**, 2067 (1987).

[10] W. Weisenberger, Semiconductor International **11**, 188 (1988).

[11] P. G. Bordon, Y. Baron, and B. McGinley, Microcontamination **5**, 30 (1987).

[12] D. Chen and S. Hackwood, Probabilistic Modeling and Data Interpretation for Vacuum Particle Flux Monitors, presented at the 1989 American Association for Aerosol Research Conference, Reno, Nevada, Oct. 9–13, 1989 (unpublished).

[13] E. U. Condon and H. Odishaw, *Handbook of Physics*, 2nd ed. (McGraw–Hill, New York).

[14] C. H. Murphy, *Handbook of Particle Sampling and Analysis Methods* (Verlag Chemie International, Weinheim, 1984).