Cleaning 10nm ceramic particles by the supersonic particle beam

Inho Kim*, Kwangseok Hwang* and Jin W. Lee*

*Department of Mechanical Engineering, POSTECH
Hyoja 31, Pohang, South Korea, jwlee@postech.ac.kr

ABSTRACT

Cryogenic aerosol beam using micron-sized aerosol particles has long been successfully used to remove contaminant particles down to 50nm, and supersonic particle beam using particles smaller than 100nm lowered the limit of cleaning down to 20nm size. In this study, the supersonic particles beam technique was improved so that the cleaning limit could be lowered to 10nm size range for ceramic contaminant particles. Two different kinds of particles were generated using Ar, CO_2 gas with or without He carrier gas. Cleaning performance for 10nm contaminant particles was sensitive to the combined condition of the beam particle size and velocity. Optimum condition for particle size and velocity were sought by varying the nozzle contour and stagnation temperature and pressure. The best removal efficiency was about 90% for 10nm Al_2O_3 particle, which it is the best performance reported to date.

Keywords: supersonic particle beam, cryogenic particle beam, nano particle cleaning, supersonic nozzle

1 INTRODUCTION

Particle contamination seriously affects the manufacturing yield of submicron scale devices. Device feature for DRAM/flash memory is expected to decrease continuously, reaching 25nm by 2015, and together with it the critical defect size to decrease to 12.5nm by 2015 [1]. Various nanotechnology based devices with feature dimensions in the nanometer size range will also be marketed in due time, which may accelerate the decrease of the required killer defect size. Since the use of drag force becomes less efficient as the contaminant size is decreased, it is generally agreed that conventional techniques should work poorly for submicron particles, and state-of-the art of various cleaning technique stays around 50-90nm.[2, 3]

One promising technique applicable in the nanometer range is the cryogenic aerosol technique, where contaminated surface is bombarded by fine particles of volatile material at high velocity. Contaminant particles adhered on a surface can be removed when the energy transferred from the bullet particles is sufficient to overcome the adhesion energy between the contaminant and substrate. Lee et al.[4] showed that Argon bullet particles could remove contaminant particles effectively, and other studies reported the applicability of Argon aerosol technique to nano contaminant cleaning. Argon, nitrogen, carbon dioxide and water are the most common cleaning agents used, and each offers advantages and disadvantages over the others. In current technology gas is expanded through a simple nozzle like a cylindrical hole. During the cooling process part of the gas becomes liquid, and the nozzle expansion atomizes the liquid into fine droplets. Solidification follows through further expansion. Typical particle size generated in this way is a few microns and the velocity about 100m/s, which is effective for cleaning down to 50nm but cleaning efficiency drops very rapidly for smaller particles thereof.[5,6]

Recently, Yi et al.[7] showed by MD simulation that far smaller bullet particles have to be used for removing nano-sized contaminants. Removal efficiency for nano-sized contaminants depended more on the velocity of the bullet particle, and concluded that even at the same kinetic energy level a smaller particle moving at a higher velocity should give a better removal. It was also shown that when the bullet particle was too big compared to the target contaminant, by a factor of 10 or more, the fragmented atoms/molecules of the bullet particle after collision may even surround the contaminant particle, preventing it from leaving the surface. Thus the use of smaller bullets moving at high velocities is expected to have extra advantages in cleaning narrow trenches and in reducing the damage potential. Lee et al.[4, 8] succeeded in removing 20nm ceramic and metal particles using bullet particles of 100nm or smaller diameter.

This study aims to explore the possibility of using the same bullet particles smaller than 100nm for removing nano-sized contaminants down to 10nm. Argon and CO_2 particles were generated by homogeneous nucleation and growth during supersonic expansion through a Laval nozzle. Particle size and velocity were varied by varying the nozzle contour, gas composition, and stagnation pressure and temperature. Cleaning experiments were done for a flat surface contaminated with a variety of particles of size down to 10nm.

2 EXPERIMENT

The experimental system is schematically shown in Fig.1. Gas mixture is passed through a liquid-N2 dewar and cooled to a required temperature by a temperature controller. Pre-cooled gas mixture is then introduced into a stagnation chamber, and then expands through a Laval nozzle into a vacuum environment. During a supersonic expansion through the Laval nozzle, tiny condensation nuclei are formed and grow in size, where the final size can be

controlled by the stagnation pressure and temperature, back pressure of the vacuum chamber, and the shape of nozzle.

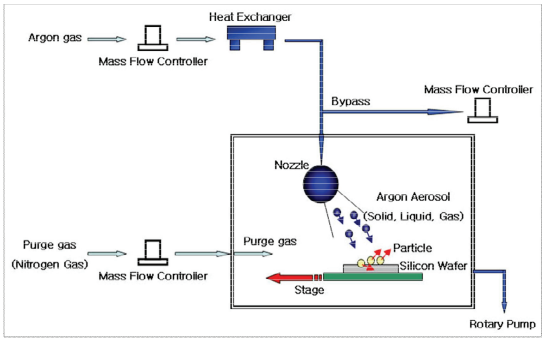


Figure 1: Schematic of the experimental setup.

In order to generate bullet particles of require size and velocity, four different gas mixtures - Ar, Ar/He, CO_2, CO_2/He - were used. Nozzle contour and nozzle dimensions were so determined that the Mach number at the nozzle exit is higher than 5.0 over the whole range of experimental conditions. Nozzles are nearly conical but rapidly expanding near the exit in order to prevent shock formation.

Silicon wafers coated with Al_2O_3 particles were exposed to the bullet particle beam for variable period of time up to 2-3minutes. Size of the contaminant particles was in the range of 5~80nm with nominal size of 10nm. Particles were dispersed in alcohol using an ultrasonic stirrer, and then dripped and spin-coated on a Si wafer. Coated wafers were dried with dry N_2 gas flowing for 1-7 days. SEM pictures of the wafer surfaces were taken before and after cleaning. Distance between the nozzle and the wafer and the angle of the particle beam were variable. Chamber pressure was also varied in the range of 10~200torr.

2.1 Cleaning with Ar and Ar/He

Cleaning experiment was first tried for Al_2O_3 particles on a flat Si surface. When pure argon was expanded at 1500torr, cleaning effect was almost zero. When the pressure was raised to 1850torr which was high enough for removing 20nm contaminants, particle removal was almost negligible (Fig.2). Aerosol particles generated at 1500torr and 1850torr are formed via homogeneous nucleation, but the size or the velocity is not good enough to remove 10nm contaminant particles. In order to vary the bullet size and velocity over a wide range via variation in stagnation pressure, mixtures of Ar and He were used, where Argon partial pressure was controlled so that no liquid formation occurs before the nozzle throat and particles are formed entirely by homogeneous nucleation and growth downstream of the throat. In order to generate bullet particles of required size and velocity, three different mixtures of 1:1, 2:8, 1:9 Ar/He were used, and stagnation pressure and temperature were varied in the range of 3000~37500 torr and 118~127K, respectively.

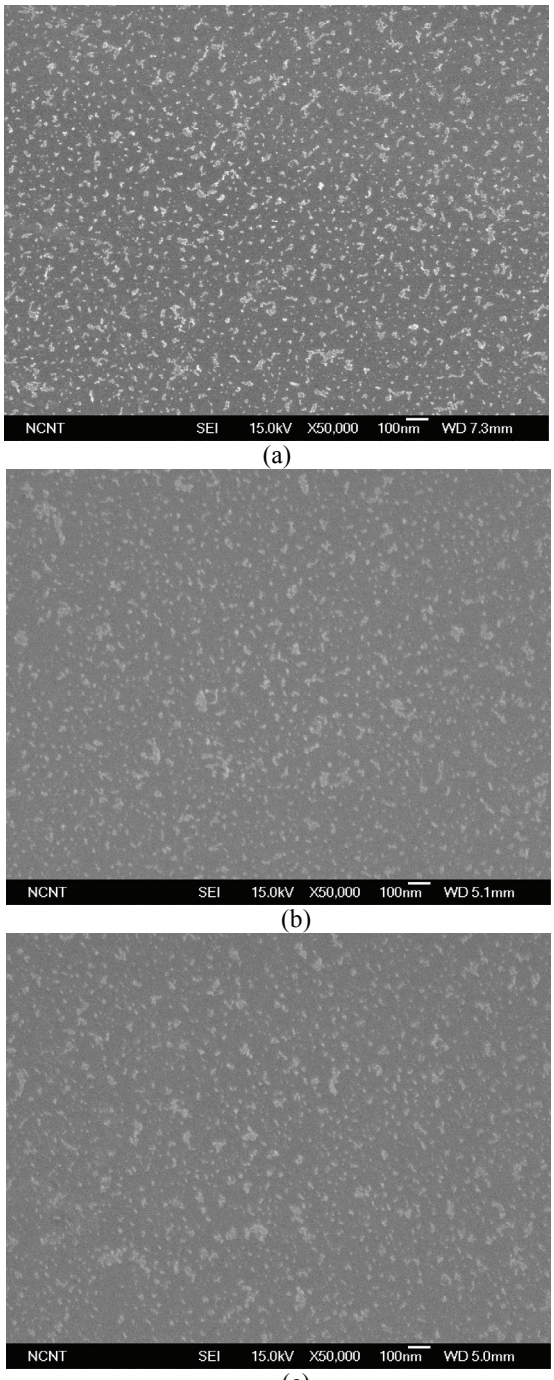
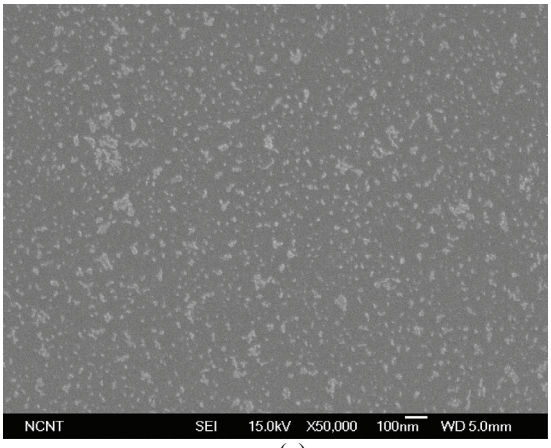


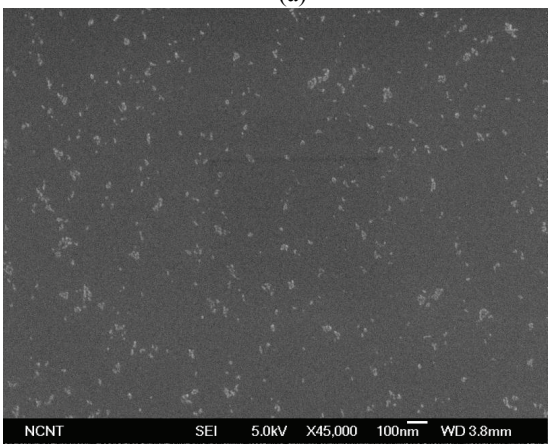
Figure 2: SEM images (a) before and after cleaning of 10nm Al_2O_3 particle using pure Argon at (b) 1500torr and (c) 1850torr, respectively.

Al_2O_3 particles were not cleaned when a 1:1 Ar/He mixture was expanded at 3000torr and 127K. (Fig. 3(a)) When 2:8 mixture gas was used at 8000torr and 118K, large Al_2O_3 particles were removed, but very small particles remained uncleaned - 10nm Al_2O_3 particles are still observed after cleaning (Fig. 3(b)). When 1:9 Ar/He

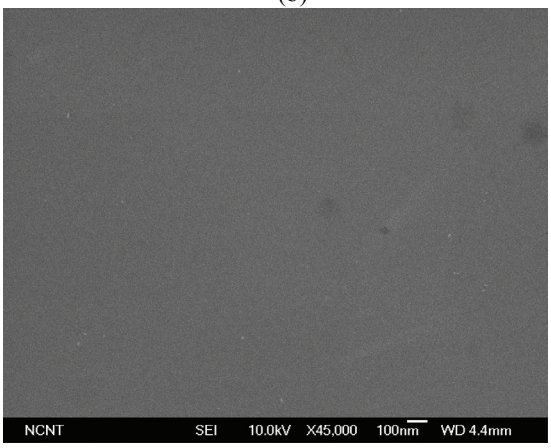
mixture was used at 37500torr and 123K, Al_2O_3 particles were completely removed (Fig. 3(c)). The use of Helium carrier increased particle velocity through increased sonic speed and also enhanced particle growth by removing condensation heat through collisions on growing bullets.



(a)



(b)

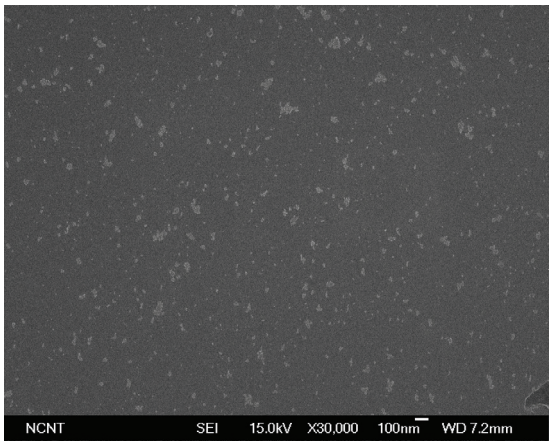


(c)

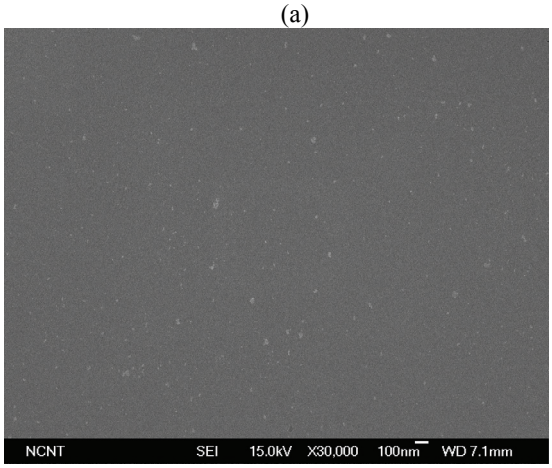
Figure 3: SEM images after cleaning of >10nm Al_2O_3 particles using (a) 1:1 Ar/He at 3000torr/127K, (b) 2:8 Ar/He at 8000torr/118K, and (c) 1:9 Ar/He at 37500torr/123K.

2.2 Cleaning with CO2 and CO2/He

In order to further increase the bullet particle velocity, CO_2 particles were generated starting from room temperature, almost three times as high as that used for Ar particle generation. To control the bullet particle size and velocity, stagnation pressure and CO_2/He mixture gas were used. When pure CO_2 gas was expanded at 10bar, cleaning effect was almost zero. When the stagnation pressure was raised to 30bar, a substantial fraction of the contaminant particles were removed (Fig. 4(a)). When the stagnation pressure was raised further to 50bar, Al_2O_3 particles were completely cleaned (Fig. 4(b)).



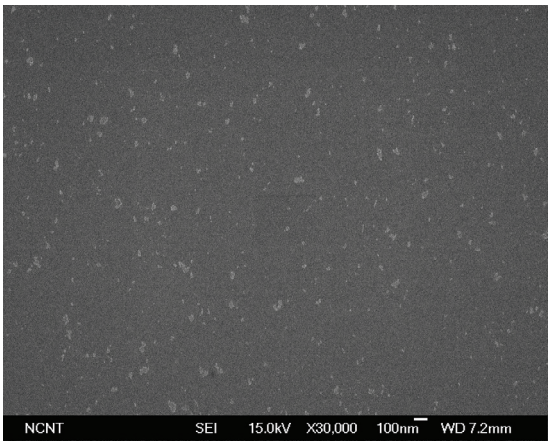
(a)



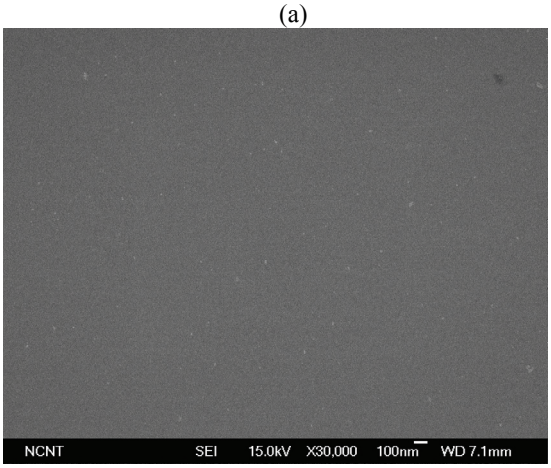
(b)

Figure 4: SEM images after cleaning of >10nm Al_2O_3 particle using pure CO_2 at (a) 30bar and (b) 50bar.

In order to further increase the velocity of the bullet particles, 1:1 CO_2/He mixture gas was used at various stagnation pressures. When this 1:1 mixture gas was used at 30bar, large particles were perfectly cleaned, but there still remained 10nm Al_2O_3 particles uncleaned (Fig. 5(a)). Al_2O_3 particles of any size were completely cleaned only when 1:1 CO_2/He mixture gas was expanded at 50bar (Fig. 5(b)). Cleaning result was a little better with CO_2/He mixture than with pure CO_2 gas.



(a)



(b)

Figure 5: SEM images after cleaning of >10nm Al_2O_3 particle using 1:1 CO_2/He at (a) 30bar and (b) 50bar.

3 CONCLUSIONS

Nano-particles on a flat surface were cleaned by bombardment with supersonic particle beam. Particle beams were generated by supersonic expansion through contoured Laval nozzles, such that the final particle velocity was in the supersonic range. Particle size was varied by controlling the stagnation pressure/temperature, nozzle contour and gas composition. Cleaning was almost complete for various ceramic particles down to 10nm, which was attributed mostly to the small size and increased velocity of the bullet particles.

4 ACKNOWLEDGMENT

This work was supported by the Korea Science and Engineering Foundation (KOSEF) grants funded by the Korea government (MOST) (No.ROA-2008-000-20045-0).

REFERENCES

[1] SEMATECH, International Technology Roadmap for Semiconductors (ITRS), 2007

[2] K. Bakhtari, R.O. Guldiken, P. Makaram, A.A. Busnaina and J.G. Park, "Experimental and Numerical Investigation of Nanoparticle Removal Using Acoustic Streaming and the Effect of Time", Electrochem.Soc., 153(9),G846, 2006

[3] H.K. Lim, D.D. Jang, D.S. Kim, and J.W. Lee, "Correlation between Particle Removal and Shock-wave Dynamics in the Laser Shock Cleaning Process", Applied Phys., 97(5), 1, 2005

[4] J.W. Lee, K.H. Hwang, K.H Lee, M.Y. Yi and M.J. Lee, "Removing 20nm particles using a supersonic argon particle beam generated with a contoured Laval nozzle", Adhesion Science and Technology, 23, 769, 2009

[5] J.M. Lauerhass, J.F. Weygand, and G.P. Thomes, "Advanced cryogenic aerosol cleaning: application to damage-free cleaning of sensitive structured wafers", IEEE/SEMI Advanced Semiconductor Manufacturing Conference, 2005

[6] H.Lin, K. Chioujones, J. Lauerhaas, T. Freebern and C. Yu, "Damage-free Cryogenic Aerosol Clean Processes", IEEE Trans. Semiconductor manufacturing,20(2), 2007

[7] M.Y. Yi, D.S. Kim, J.W. Lee and J. Koplik, "Molecular dynamics (MD) simulation on the collision of a nano-sized particle onto another nano-sized particle adhered on a flat substrate, Aerosol Science and Technology, 36(12), 1427, 2005

[8] J.W. Lee, K. Kang, K. Lee, M. Yi, and M. Lee, "Removing 20nm ceramic particles using a supersonic Argon particle beam from a contoured Laval nozzle", Thin Solid Films, 517(14), 3866, 2009