CO_2 laser-assisted particle removal from silicon surfaces

J.B. Héroux, S. Boughaba, E. Sacher, and M. Meunier

Abstract: A CO_2 laser particle removal system was built that enables the removal of 0.1 μm alumina particles from silicon substrates. This system has raster scan capabilities to clean large surfaces, which were analysed using a particle counter. After deposition and removal of 0.1 μm alumina particles, the final concentration is less than 25 particles cm^{-2} for particle clusters between 0.1 and 10 μm. The efficiency of particle removal is nearly independent of the laser fluence between 0.65 and 2.9 J cm^{-2} and drops suddenly below 0.65 J cm^{-2}.

Résumé : Un laser CO_2 a été utilisé pour la réalisation d'un dispositif de nettoyage de substrats de silicium, permettant l'enlèvement de particules d'alumine de 0,1 μm. Le dispositif a la capacité de traiter de larges surfaces par balayage du substrat; la caractérisation de telles zones nettoyées a été réalisée grâce à un compteur de particules. Après dépôt et enlèvement de particules d'alumine de 0,1 μm, la concentration finale est inférieure à 25 particules cm^{-2}, pour des agglomérats de particules de taille comprise entre 0,1 et 10 μm. Pour un flux laser compris entre 0,65 et 2,9 J cm^{-2}, l'efficacité du procédé demeure inchangée; elle décroît au-dessous de 0,65 J cm^{-2}.

1. Introduction

According to an industry survey published in 1994 [1], contamination by particles is one of the most important problems for the microelectronic industry. Particles can mask patterns during photolithography [2], generate defects in epitaxial growth, increase electromigration in metallic lines [3, 4], and lower the gate oxide breakdown voltage [5]. With today's design rules, particles as small as 0.1 μm in diameter are considered potential killer defects [6].

Traditional wet cleaning methods have several shortcomings; they are almost inefficient for 0.1 μm particle removal, inherently incompatible with cluster tools, expensive when using ultrahigh purity chemicals, and environmentally harmful. Therefore, new cleaning techniques that do not have these drawbacks are being developed [7, 8].

CO_2 laser-assisted particle removal (LAPR) is one of these promising new methods, developed by Allen and co-workers [9–13], to remove small particles from contaminated surfaces. In this technique, a film of water is first deposited onto the surface to be cleaned. A pulsed CO_2 laser emitting at 10.6 μm is then used to irradiate the surface. Its radiation is absorbed by the liquid film, inducing its explosive evaporation. The resultant acoustic wave produces forces of sufficient magnitude to expel particles from the surface.

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Allen et al. [9] demonstrated the removal of particles greater than 1 μm in diameter using a scanning electron microscope at a low magnification (whole wafer shown). To understand the phenomena involved in laser-assisted particle removal, they investigated the fluence thresholds for particle removal (2.2 J cm^{-2}) and shock wave generation (4.2 J cm^{-2}).

The work we report here carries on with the development of using CO_2 laser as an efficient cleaning tool. We investigated the removal of 0.1 μm alumina particles from silicon surfaces with water used as an energy transfer medium. By using an optical microscope in the dark field mode at 50× magnification as well as a particle counter, we were able to detect submicrmetre particles on large surfaces and provide quantitative results on the efficiency of particle removal as a function of the CO_2 laser fluence. The optical microscope was also used at 1000× magnification to detect possible damage to the surface and to measure the sizes of particle clusters remaining on the surface after cleaning.

2. Experiment

The experimental setup is shown schematically in Fig. 1. A Lumonics 840 CO_2 laser emitting at 10.6 μm in the pulsed mode was used. The output energy was 0.9 J and the pulse duration of 0.2 μs. The multimode beam was vertically deviated with a mirror at 45° and then focused on the sample surface using a converging germanium lens with a focal length of 30 cm. In the standard cleaning configuration, the incident fluence at the substrate was about 3 J cm^{-2}.

To deposit the water film in a reproducible manner only at the irradiated area, a home-made system was used. It consisted of a container, half-filled with deionized (DI) water that was heated to 40°C. Nitrogen gas flowed through the container to carry vapour in a copper nozzle near the surface to be cleaned. A pulse-timing unit permitted the water vapour to be deposited for periods between 0.1 and 2.5 s, resulting in the condensation of water at the colder target surface. This was immediately followed by the triggering of the laser.

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Fig. 1. Schematic of the CO_2 laser-assisted particle removal setup.

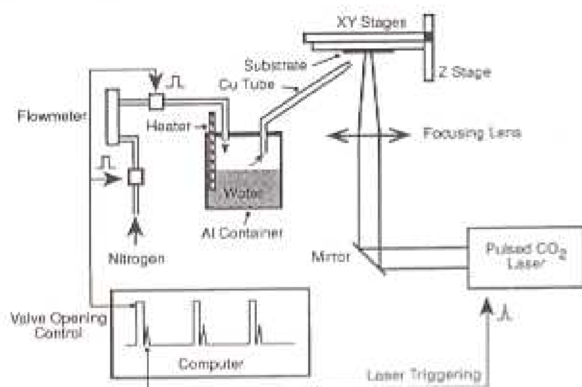
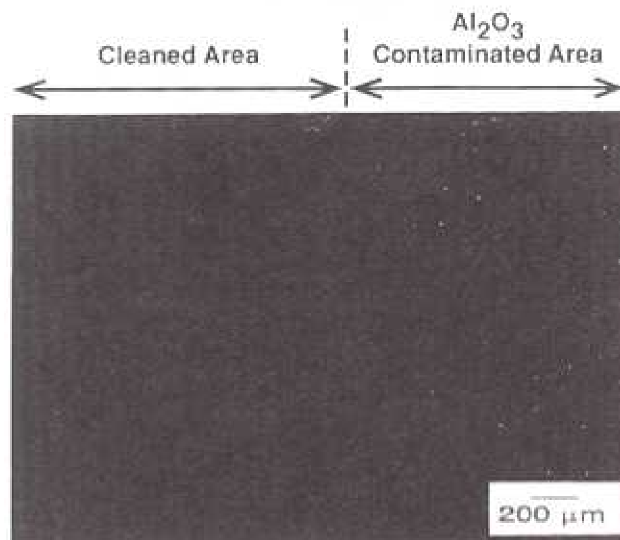


Fig. 2. Dark-field optical micrograph (50×) of a silicon surface contaminated with 0.1 μm alumina particles. The edge of the cleaned region is shown. Laser fluence is 2.9 J cm^{-2}.



The sample was mounted on an *XYZ* computer controlled stage. Displacement perpendicular to the optical axis (*XY*) was used to scan the wafer surface to clean large areas, whereas displacement along the optical axis (*Z*) was used to vary the laser fluence.

To reduce the probability of contamination by ambient air, the sample was held face down and the experimental setup was placed in a class 500 cleanroom under a laminar flow hood that provided a class 10 working area.

The substrates used were 100 mm <100> silicon wafers not subjected to any special treatment. The alumina particles (Beta Diamond Corp.), of certified 0.1 μm size, were deposited by mixing about 0.01 mg of particles with 100 ml DI water. Drops of this colloidal solution were deposited on the surface, which was then spun at 4000 rpm; the particles remaining on the surface exhibited a nearly uniform concentration.

To observe the particles, an optical microscope, which can be operated in either bright- or dark-field modes at magnifications ranging from 50× to 1000×, was used. Bright-field illumination was used to detect damage to the surface and film contamination, whereas dark-field illumination served to detect submicrometre particles over areas of about 9 mm^2. A laser scanning particle counter (SAS 3600, Particle Measuring Systems Inc.) was used to obtain quantitative data on larger surfaces. This instrument classifies particles according to their size from 0.1 to 10 μm and is calibrated with spherical polystyrene latex (PSL) spheres. Since the shape and optical properties of alumina contaminants differ from those of spherical PSL particles, the sizes given are PSL-sphere equivalents. It must be emphasized that we have verified, using a particle generator to deposit 0.1 μm Al_2O_3 particles at a very low surface concentration, i.e., without formation of large size clusters, that these PSL-sphere equivalent sizes correctly account for the absolute size of the alumina particles.

3. Results and discussion

Figure 2 shows a dark-field optical micrograph of the edge of a cleaned area, which is 7 × 7 mm^2, using a fluence of 2.9 J cm^{-2}. The right side shows the originally contaminated surface, whereas the left side is a part of the cleaned area. The nitrogen flow was 5000 mL min^{-1} and the vapour pulse duration was 1 s, so that the volume of nitrogen mixing with the vapour in the container was 80 mL and the thickness of the water on the sample was estimated to be about 6 μm. Three laser pulses (1 s between each pulse) were fired 0.2 s after the vapour burst. The reason for using three pulses instead of one is to be sure that all the water was evaporated. This cycle (one vapour burst and three laser pulses) was repeated five times for a complete static cleaning (no scanning of the surface).

Figure 3 shows results obtained with a fluence of 2.9 J cm^{-2} using the particle counter, by scanning the sample to clean large regions. The vapor pulse duration was 2 s with a flow rate of 5000 mL min^{-1}, giving a volume of nitrogen of 160 mL. After one burst of vapour and three laser pulses, the sample was moved 3 mm along a line 24 mm long. This cycle was repeated to clean a square of 24 × 24 mm^2. The region analysed by the counter is a circle 20 mm in diameter inside the square, so that the results were not biased by the edges of the square.

Before particle deposition, the originally clean surface, Fig. 3*a*, already contained 80 particles, all below 1 μm, in the circle analysed. After deposition of DI water, deliberately not contaminated, followed by spinning, there was only a slight increase of the number of particles, Fig. 3*b*. After particle deposition, a large quantity of these particles remained on the wafer, Fig. 3*c*, the largest number of them corresponding to a size of 2 μm. This is due to the formation of clusters during deposition. Moreover, the concentration of these clusters is high enough to veil or screen the particles that appear on the surface before deposition. As a consequence, the laser counter is not able to detect them and a reduction in the number of 0.1 μm particles is observed after the Al_2O_3 deposition, Fig. 3*c*. After one cleaning scan Fig. 3*d*, some particles are removed, the clusters become smaller and the largest number of particles corresponds to a size of 0.2 μm. After three scans, Fig. 3*e*, the distribution of the particle sizes has its maximum at 0.1 μm. Following seven scans, Fig. 3*f*, the number of particles reaches a lower limit essentially equal to that before deposition. These

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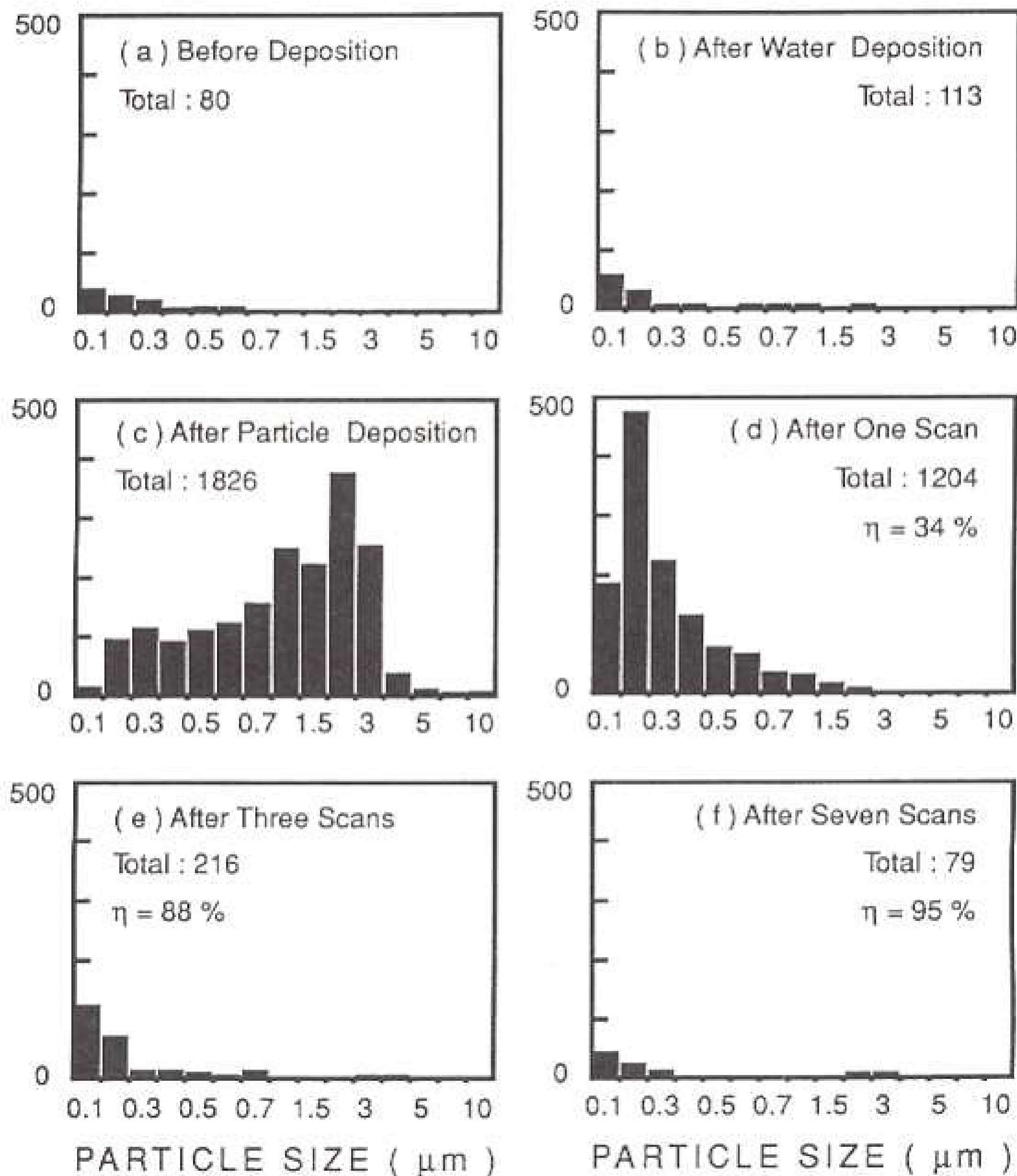
Fig. 3. Histograms obtained with the particle counter, showing 0.1 μm alumina particles on a silicon substrate (*a*), before treatment; (*b*), after deposition of DI water only; (*c*), after deposition of particles; (*d–f*), after cleaning by laser rastering. Particle removal ratios, η, are also given.



results clearly show that laser cleaning is efficient in removing alumina particles as small as 0.1 μm. The efficiency of the cleanliness is defined, in a useful way, using the particle removal ratio, η, which is the ratio of the number of removed particles to that at the starting point. All the sizes (0.1–10 μm) are considered in defining η.

The variation of the cleaning efficiency was also investigated as a function of the laser fluence. Figure 4 shows the particle size histograms obtained after four laser cleaning scans for a fluence ranging from 0.6 to 2.9 J cm^{-2}. The vapor pulse duration and the flow rate were, respectively, 2 s and 5000 mL min^{-1}, giving a volume of nitrogen of 160 mL. The starting size distributions, after particle deposition, were similar to that shown in Fig. 3*c*. At 0.6 J cm^{-2}, the cleaning was totally inefficient, the removal ratio being within the particle counter error range. Between 0.65 and 2.9 J cm^{-2}, the cleaning process was greatly improved and was almost independent of the laser fluence. Beyond 2.9 J cm^{-2}, ripples on the surface were observed with the optical microscope. Such surface defects are similar to those widely reported in laser processing works [14–17] and probably result from interference phenomena between incident and scattered radiation at the surface.

A fluence threshold between 0.6 and 0.65 J cm^{-2} was confirmed by analysing several experiments by optical microscope. This threshold, which corresponds to an incident peak power of 1.6 MW cm^{-2}, is probably due to a physical change

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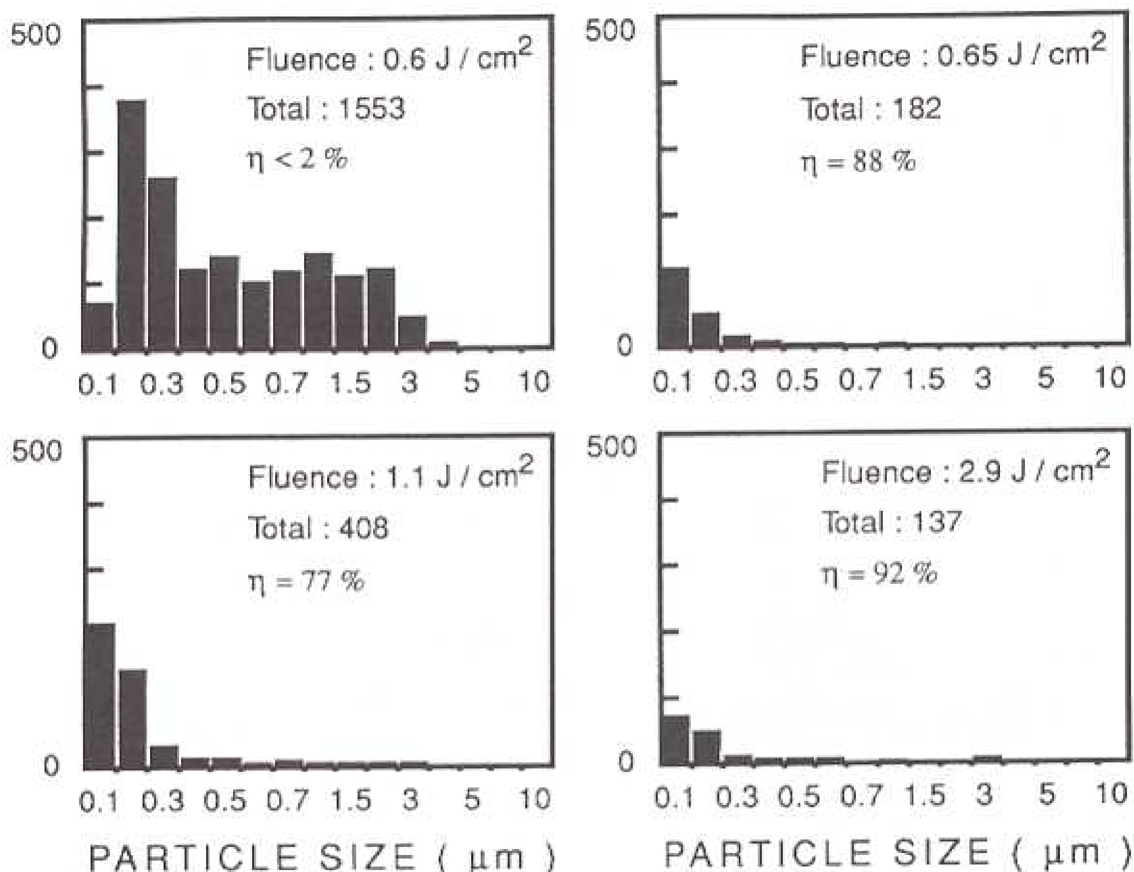
Fig. 4. Influence of the laser beam fluence on the efficiency of the cleaning process, characterized using the particle removal ratio η. Initial contamination of the substrates was 0.1 μm Al_2O_3 particles. Vapor pulse duration, 2 s; flow rate, 5000 ml min^{-1}; volume of nitrogen, 160 ml.



in the explosive evaporation process that generates the removal forces. This threshold may correspond to that for the water film to reach its maximum temperature (and pressure) without boiling, which is given by $T_{max} = 0.9T_c = 309°C$, where T_c is the critical temperature of the water [18]. It may also correspond to the minimum fluence necessary to generate an acoustic wave at the front of the film that would be reflected by the substrate, thus ejecting particles. Finally, this threshold may also be the limit of uniform heating of the film; below 0.65 J cm^{-2}, the temperature elevation would occur mainly at the front of the film.

4. Conclusion

A CO_2 laser particle removal system was built. It permits the removal of 0.1 μm alumina particles from silicon substrates over large areas due to its raster scan capabilities. A particle counter was used to obtain quantitative results after cleaning. The final concentration of particles was less than 25 particles cm^{-2} for particle clusters between 0.1 and 1μm and less than 3 particles cm^{-2} for clusters between 1 and 10 μm. The efficiency of particle removal was nearly independent of the laser fluence between 0.65 and 2.9 J cm^{-2}, dropping suddenly below 0.65 J cm^{-2}.

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