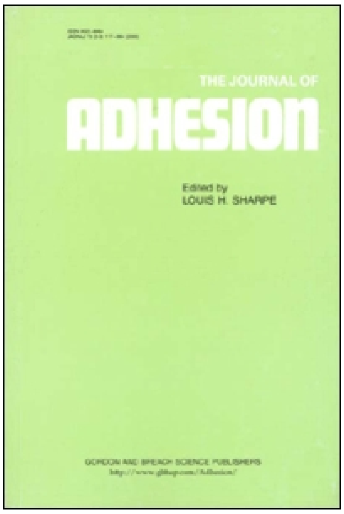

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EXPERIMENTAL STUDY OF ALUMINA PARTICLE REMOVAL FROM A PLANE SURFACE

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Micron and submicron alumina particles are often used for the mechanical polishing of the GaAs wafers processed in the microelectronic industry. A better understanding of the adhesion mechanisms is the key factor for the particle removal and for the optimisation of the industrial chemical cleaning. However, the nature and the strength of the complex interactions occurring between asymmetrical alumina particles and the surface remain unclear. Thus, an experimental study of the detachment of asymmetrical alumina particles in adhesive contact with a glass plate was done using a specially designed shear stress flow chamber. A series of experiments was performed to measure the shear stress necessary to remove individual alumina particles (of 3 and 0.3 μm nominal size) under various chemical solutions (diluted ammonia, surfactant and glycerol). Then the effects of the particle size, the resting time, the pH and the nature of the chemical solutions used for the removal of the alumina particles was characterised in terms of percentage of alumina particles detached. Results have shown that the longer the resting time, the more adherent the particles are. Moreover, it was found that the ammonia solution gives the best particle removal rate (80%) because of the strong repulsive electrostatic interactions between the alumina particles and the glass surface, both being charged negatively in a basic solution.

Keywords: Alumina particle; Adhesion; Removal; Chemical solutions; Electrostatic interactions

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INTRODUCTION

Particulate contamination of semiconductor wafers polished with slurry particles is a critical problem. Indeed, after polishing, a cleaning step is always required to remove the residual particles well adhered to the wafer surface. Several cleaning procedures are available for semiconductor substrates, such as chemical cleaning, thermal cleaning, sputter cleaning or hydrogen plasma cleaning; among them, chemical cleaning is the simplest and easiest to control [1, 2]. Most of the contaminating particles can be removed by cleaning procedures when the removal forces overcome the particle adhesion forces. However, 100% particle removal for post CMP (Chemical Mechanical Polishing) cleaning is very difficult to achieve. Statistically, there are always a few particles that penetrate into the oxide surface more deeply than the others due to the pressure of some extremely high polishing pad asperities [3]. Therefore, the particles remaining stuck to the surface after cleaning procedures are very problematic because they can cause damage and defects in successive deposited layers. Thus, the development of optimal methods for particle removal is a key factor for the industrial cleaning and in particular for the manufacture of high-quality semiconductor surfaces for technical applications. In particular, DI water cleaning is not sufficient for efficient removal and, in the case of alumina particles, the problem is accentuated because the adhesion force was found to be one order of magnitude higher than that for submicron silica particles deposited on silicon wafer surfaces [4]. The minimization of particle contamination during wet silicon wafer cleaning was studied by Itano et al. [5, 6]. They have demonstrated that it is essential that the wafer surface and the particles exhibit the same polarity of the zeta potential. This can be achieved by adding an anionic or cationic surfactant in solution. Indeed, in the case of polystyrene latex spheres, basic solutions are superior to acid solutions in terms of particle removal efficiency: particles are electrically repelled from the wafer surfaces due to the negative zeta potential of most particles in the basic solutions.

To understand the particle removal mechanism, considerable attention has already been devoted to the net adhesion force between spherical particles and a flat surface. A number of experiments and models were developed to understand and better describe the adhesion mechanisms [4, 7]. One of the most important and sensitive parameters in the relationship between adhesion force and wall shear stress at detachment is the radius of the contact area between the particles and the flat surface. When there are no specific interactions between the two surfaces in contact, the contact area may be related to the microroughness or to the adhesive deformation of the surfaces [8]. Knowing the radius of the contact area, the contribution of the non-specific interactions (*i.e.*, Van der Waals, double layer and structural interactions [9]) to the adhesion force and torque can be estimated. On the contrary, only few studies on the problem of asymmetrical particles in contact with a flat surface were done because of its high complexity. Indeed, the behaviour of non-spherical alumina particles is different from the behaviour of spherical ones in contact with a flat surface [10]. Due to the specific behaviour of these particles of complex shape, some parameters such as contact area, size, physico-chemical interactions, hydrodynamic forces and torque are very difficult to control and to modulate individually. Moreover, the contamination of semiconductor surfaces by non-spherical alumina particles is a real problem that is still not well understood. So it was of interest to propose an original approach concerning the study of their removal from the surfaces. To this end, we have used a specially designed shear stress flow chamber to study the effects of some chemical solutions on the detachment of non-spherical alumina particles of 3 and 0.3 μm nominal size from a glass plate, a model for a semiconductor wafer. A particular effort was made to control, individually, all the experimental parameters. First, the particles are dispersed in the chemical solutions prepared under optical control and static conditions. Then, they are put in adhesive contact with the glass plate allowing for physico-chemical interactions to take place between the surfaces. In addition, the shear stress flow chamber is carefully designed to give a fully-developed, laminar, two-dimensional Poiseuille flow, resulting in the accurate knowledge of the wall shear stress acting on the individual particles. A series of experiments are performed to measure the wall shear stress necessary to remove a population of individual particles under the influence of different chemical products (surfactant, glycerol and ammonia) diluted in distilled water and to investigate their role in the detachment mechanisms.

MATERIALS AND METHODS

Alumina Particles

Experiments were performed with 3 and 0.3 μm alumina particles used especially for the mechanical polishing of the GaAs surfaces. The alumina particles have a special shape, elongated with plane facets. Thus, with their complex shape, they are perfectly parallel to the polished surface and, consequently, the abrasion rate is higher than in the case of spherical particles. Figure 1 shows a scanning electronic

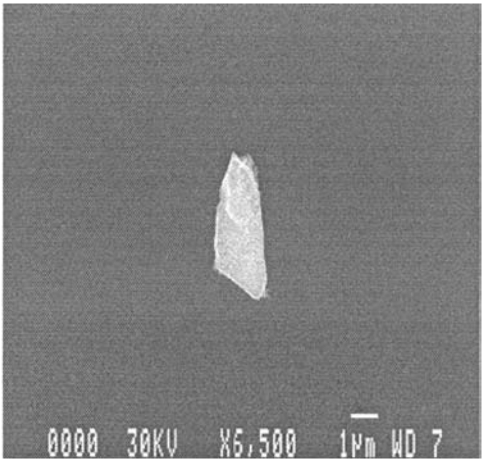


FIGURE 1 Alumina particle of 3 μm size before mechanical polishing.

microscopy (SEM) observation of a 3 μm particle before polishing. After polishing, these particles can form well adhered aggregates that can act as large particles, scratching the surface. Moreover, some particles are embedded in the surface and it is very difficult to remove them by rinsing or chemical cleaning. SEM observations made on 0.3 μm alumina particles have showed that they have a shape more spherical than the 3 μm particles. However, it was practically impossible to observe an individual alumina particle of 0.3 μm because they form many aggregates.

The population of particles used in the experiments is far from monodispersed. In particular, particle size distribution of the 3 and 0.3 μm alumina particles, measured by laser granulometry (Malvern Mastersizer), is not Gaussian and is not symmetric around the mean value. Indeed, we observe for the size distribution of the 0.3 μm particles expressed in volume percentage that there are some particles, probably aggregates, larger than 1 μm as can be seen in Figure 2.

The Chemical Solutions

We dispersed 50 g of the 3 and 0.3 μm alumina particles separately in each chemical solution to obtain a concentration of 1 g/l. Chemical solutions used and studied were distilled water, wet solutions containing surfactant and glycerol diluted respectively in distilled water at 0.05 and 0.1 ml/l (surfactant) and 20 ml/l (glycerol). We also used basic solutions of ammonia diluted in distilled water (1 ml/l and 10 ml/l).

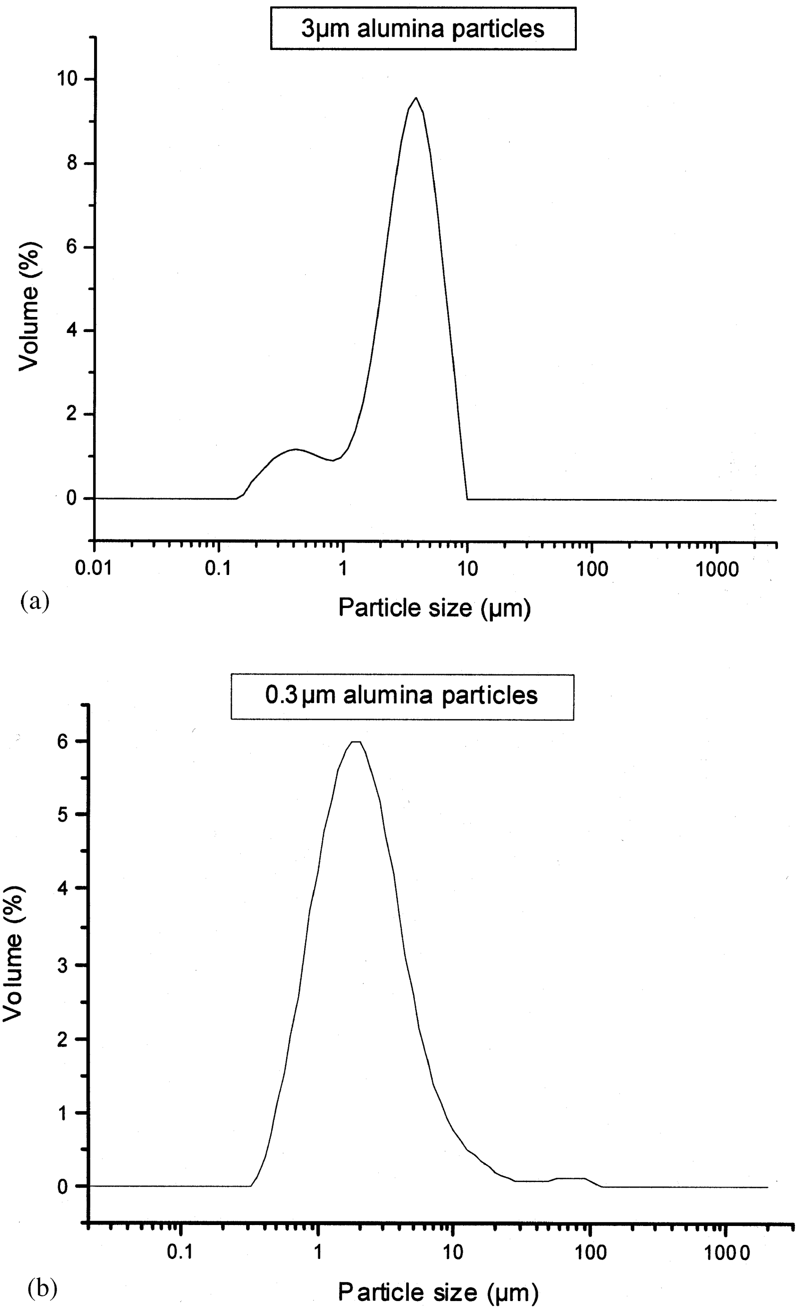


FIGURE 2 Size distribution of 3 and 0.3 µm alumina particles.

Shear Stress Flow Chamber

The shear stress flow chamber and the associated experimental devices used in the present study were derived from two other chambers used in other studies concerning the adhesion of particles on surfaces. In these studies, Elzo et al. [11] have characterized particle/membrane interactions during drinking water production and Lorthois et al. [12, 13] have quantified the fibrin/fibrin specific molecular interactions in blood clot fragmentation.

The chamber is composed of a bottom glass plate (210 × 90 × 4 mm) onto which alumina particles are deposited, an upper Plexiglas® plate (210 × 90 × 10 mm) and a hollowed-out stainless steel shim (210 × 90 × 0.2 ± 0.0025 mm) for channelling the fluid flow. The three plates are held together with aluminium clamps. The fluid enters the chamber through a 1 mm wide slit pierced perpendicularly in the upper plate and it exits the chamber through a 2 mm diameter hole. A third orifice topped by a syringe valve is used to inject the suspension of alumina particles. The rectangular flow channel follows a diverging-converging channel, in order to ensure a uniform flow at the entrance of the channel part where particles are deposited. More details concerning the flow in the chamber are reported by Lorthois et al. [12], Figure 3.

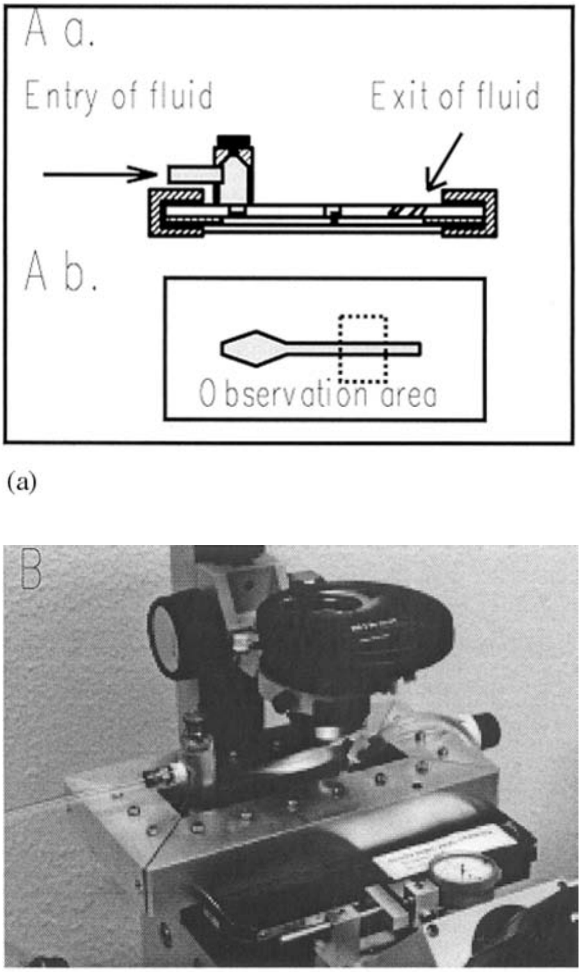
The existence of a laminar unidirectional Poiseuille flow in the rectangular flow channel was demonstrated by preliminary experimental verifications of the theoretical relationship between pressure drop (ΔP) and flow rate (Q) for plane two-dimensional Poiseuille flows [13] showing the non-deformation of the flow chamber even at the higher pressures applied. For this type of flow, the wall shear stress, τ_w, is uniform except in a short entry region and in the boundary layers confined near the channel side walls. Outside, *i.e.*, in the region of interest where particles are deposited (see the observation area shown in Figure 3), τ_w can be written as:

$$\tau_w = \frac{3\mu Q}{4h^2 l} \tag{1}$$

where h is the flow channel half thickness, μ is the dynamic viscosity of the fluid and l is the flow channel half width.

Detachment Mechanisms

Let us consider the ideal case of an individual spherical particle in contact with a plate under a linear shear flow. The particle experiences the hydrodynamic drag, D, torque, C, and lift, L, given by [14, 15]



(a)

(b)

FIGURE 3 A view of the shear stress flow chamber: A. Scheme, B. Photograph.

$$\begin{cases} D = 32\tau_w a^2 \\ C = 0.38aD \\ L = 9.257\tau_w a^2 \mathrm{R_{ep}} \end{cases} \tag{2}$$

where a is the particle radius. In this expression $\mathrm{R_{ep}}$ denotes the particle Reynolds number. For convenience, the velocity of the fluid at the particle centre is replaced by $a\tau_w/\mu$ assuming a linear velocity profile close to the wall as a is negligible compared with h. It can be noticed that R_{ep} is always lower than 1. Lift may, thus, be neglected compared with drag in the detachment process as was verified by Lorthois et al. [12] using the expressions (2). Earlier workers already assumed that lift may be neglected in the theory of detachment of particles from flat surfaces by a laminar shear flow [8, 16].

Let us now consider the real case of a three-dimensional laminar flow past an array of spherical particles in contact with the bottom side of a rectangular channel. If the particles are sufficiently spaced (typically the distance between particles greater than $5a$) and the ratio of the particle radius, a, to the half channel height, h, is less than 1/15, Equations (2) still hold [17]. Both conditions are fulfilled in the experiments reported in the present paper. Unfortunately, real particles such as the alumina particles used in the experiments are not spherical. Therefore, it is not possible to interpret quantitatively the results in terms of adhesion force between particle and surface using Newton's second law and Equations (2) as was previously done by other workers [11, 12].

Detachment Experiments

The chamber is assembled, placed on the stage of an inverted phase contrast microscope (Nikon Diaphot) and coupled to a CCD camera with a video image-processing system for the visualization and counting of particles. The observation area is located far downstream of the rectangular channel entry, in order to avoid entrance effects. The flow chamber is filled with distilled water mixed in each case with the chemical products. Alumina particles in suspension in the chemical solutions are slowly injected into the flow chamber through a syringe valve. They are settled under gravity for a minimal time of 20 minutes, resulting in particle separation of about 5 particle diameters and allowing them to adhere perfectly. This separation is chosen to minimise the artifacts caused by hydrodynamic interactions between particles, such as shielding of the shear field as mentioned in the previous section. After that resting time, the flow rate in the channel is increased step by step (a typical step is three minutes). Flow rates ranging from 1 ml/min through 4.5 ml/min are generated by gravity, controlling the height of a constant head vessel located upstream from the chamber. Flow rates greater than 4.5 ml/min are obtained using a gear pump. At the end of each flow rate step, the number of particles remaining at the glass surface are counted using the video image processing system. The shear stress applied is calculated using Equation (1) from the value of the flow rate measured by weighing on an electronic balance.

RESULTS AND DISCUSSION

The number of particles initially present in the observation area is typically between 100 and 150. After each experiment, results obtained are carefully checked and validated. The number of particles remaining in both halves of the observation areas at each flow rate step are plotted as a function of the number of particles remaining in the whole observation area (Figure 4). In the ideal case (no variability of the particle radius, perfect contact area...), the curve obtained should be the first bisecting line. Therefore, results are eliminated in the case when the deviation from the bisecting line is greater than 15%, Figure 4.

For convenience, the results of all the detachment experiments have been classically plotted as the percentage of particles remaining attached to the surface as a function of the wall shear stress applied.

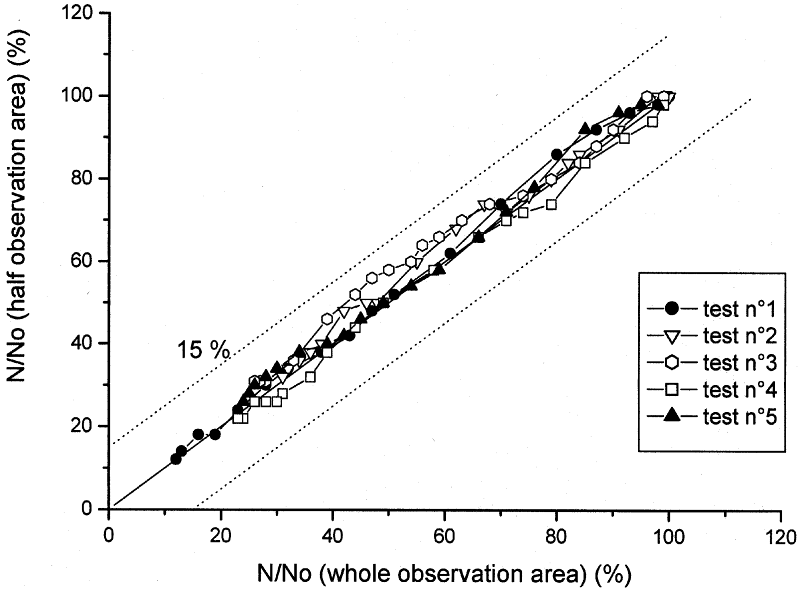


FIGURE 4 Number of particles remaining in both halves of observation areas *versus* number of particles remaining in the whole observation area.

A typical result can be examined in Figure 5. A first wall shear stress threshold under which no particle removal occurs is evidenced. A second wall shear stress threshold over which no particle removal occurs is also evidenced. These two phenomena can be explained by the non-spherical shape of the alumina particle (Figure 1) which leads to a large contact area between the particles and the plate. Consequently, the adhesion force is increased.

We have used only the 3 µm alumina particles to study the effect of the particle size on the removal, the other experiments were done with the submicron particles of 0.3 µm. These solutions of diluted surfactant, glycerol and ammonia were used to study the effects of wettability and basic pH on the removal of the particles. We performed, for each set of parameters, 5 experiments except in the case of the resting time of 14 hours (set of 2 experiments). Each result presented in the paper is the mean of five experiments.

Influence of the Resting Time

To determine the minimum resting time of the alumina particles in contact with the glass plate, we made a series of experiments with several resting times (5, 10, 15, and 20 minutes). We observed that under 20 minutes, the 3 and 0.3 µm particles did not adhere on the glass surface. So, 20 minutes is the minimal time for the particulate sedimentation. To determine the influence of resting time on the removal of the particles, we left the 0.3 µm particles to rest on the glass surface for 20 minutes, 40 minutes and 14 hours.

From Table 1, for the maximum value of $\tau_w (= 10\,\text{Pa})$, we observe that more particles remain stuck to the glass plate after 14 hours of sedimentation (65%), than for 40 minutes ($< 55\%$) and 20 minutes ($\cong 35\%$). As could be expected, the longer the resting time, the more particles remained adhered. Once particles approach the surface under the gravity effect, hydrodynamic interactions take place which govern the classical viscous repulsion. This well known phenomenon drastically delays the contact between the particles and the surface, Table 1.

Moreover, in the case of non-spherical particles, shape is another factor accounting for the difficulty of their removal. Busnaina *et al.* [18] have demonstrated that for submicron particles such as Si_3N_4 deposited on silicon wafers, there are more contact areas than for spherical particles, leading finally to greater adhesion force and, subsequently, to a smaller removal efficiency. In the present case of asymmetrical alumina particles, the phenomenon is surely similar and the adhesion force is also increased. These results show that the

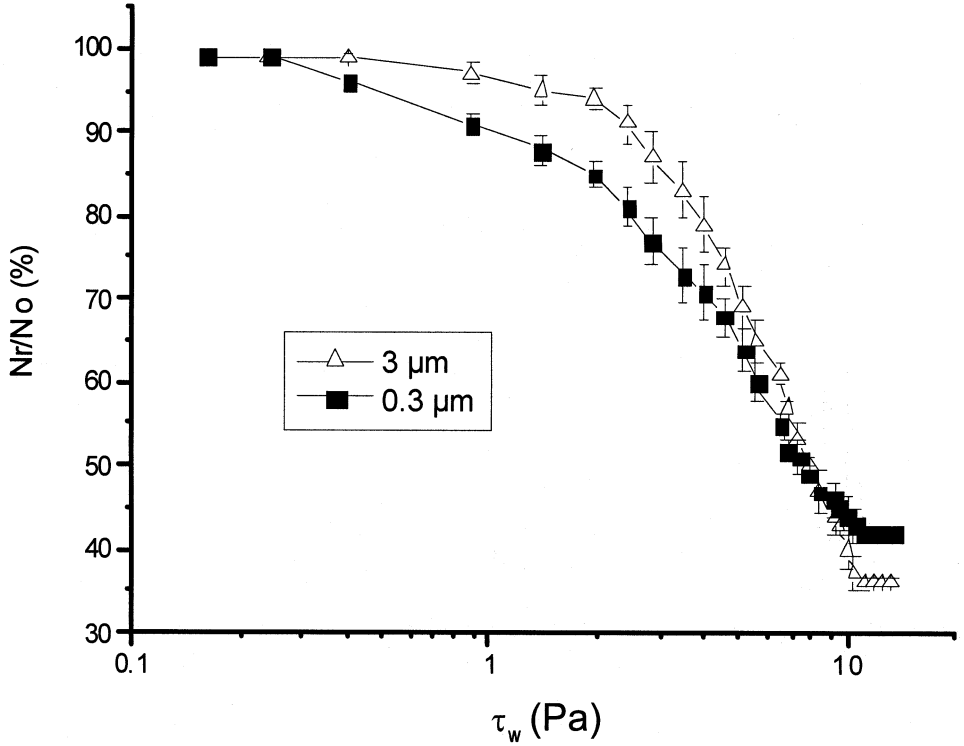


FIGURE 5 Number of 3 and 0.3 μm particles remaining in the observation area.

TABLE 1 Number of 0.3 μm Particles Remaining in the Observation Area for Different Resting Times ($\tau_w$max = 10 Pa)

Resting times	20 minutes	40 minutes	14 hours
Nr/No (%)	35	55	65

longer the resting time, the more difficult it becomes to remove the submicron particles on the surface. Thus, in the microelectronics industry, semiconductor surfaces should be quickly rinsed and cleaned after the polishing step to avoid an increase of the adhesion force.

Influence of the Particle Size

We have used alumina particles of 3 and 0.3 μm nominal size diluted in distilled water (pH = 6) and rested for 20 minutes on the glass surface. As can be seen in Figure 5, the smaller 0.3 μm particles appear to be more easily removed than the larger 3 μm ones. However, a larger percentage of 0.3 μm particles remained adherent to the surface at the end of the experiment, *i.e.*, beyond τ_w of the order of 8 Pa.

At this stage, it is useful to recall that the alumina particles are not spherical, as can be seen in Figure 1. Therefore, the effect of particle size on alumina particle removal is significantly different from what can be expected on spherical particle removal. To simplify, let us assume that the alumina particles are cubical. In general, the contact area may be related to the microroughness or to the elastic deformation (negligible here) of the surfaces [8]. The radius of the contact area is then proportional to the particle side (cubical particle) or proportional to the square root of particle radius (spherical particle, [12]). Let us also assume that the adhesion force is proportional to the contact area between the particle and the surface ($F_{\mathrm{ad}} = k\pi R^2$), even if we know that it is not rigorously true. Let us consider the classical torque balance on a particle performed by many workers, for instance by Hubbe [8], as follows:

$$F_{\mathrm{ad}}R = aD + C \tag{3}$$

where F_{ad} is the net adhesion force and R is the radius of the contact area. Using (2), it can easily be found that the wall shear stress necessary to remove spherical particles from the surface increases as the particle radius decreases. On the contrary, if we keep the expression (2) for the hydrodynamic effects on a cubic particle, the wall shear stress required to remove cubic particles does not vary as a function of the particle size. In the real cases presented in Figure 5, the improvement of particle removal as the particle size decreases is probably due to the more complex shape of alumina particles and to the subsequent modified hydrodynamic effects compared with the ones exerted on spherical particles [19]. However, at high wall shear stress, the residual adherent particles of smaller size are more difficult to remove because they are more sensitive to microroughnesses which are relatively higher for 0.3 μm particles than for 3 μm particles. This probably explains the results obtained in Figure 5.

Influence of the Nature of the Cleaning Solution

For all the experiments, we have let the 0.3 μm particles settle on the surface for 40 minutes.

Surfactant

Surfactant is a large molecule with a long carbon chain and ionic active heads at its extremities. Used in dilute solution in industrial cleaning processes, surfactant can decrease or neutralise the charge of the surfaces and make steric interactions leading to the detachment of the particles from semiconductor surfaces during rinsing.

Table 2 shows that the particle removal rates under distilled water with or without diluted surfactant (0.05 ml/l) are almost the same; but, for a double concentration of 0.1 ml/l, the removal rate is only slightly improved, Table 2. Thus, the results show that a very diluted surfactant has no significant efficiency in particle removal because the concentration chosen is surely lower than the micellar concentration. In fact, previous works on surfactant effects on particle adhesion and removal [11] have shown that there is also a critical concentration below which particle removal is not improved. Indeed, the zeta potential of the 0.3 μm alumina particles in the presence of surfactant in

TABLE 2 Number of 0.3 μm Particles Remaining in the Observation Area After Dispersion in Distilled Water and Added Surfactant Solutions of 0.05 and 0.1 ml/l) and for $\tau_w$max = 10 Pa

Solutions	Distilled water (DW)	DW with surfactant (0.05 ml/l)	DW with surfactant (0.1 ml/l)
Nr/No (%)	56	56	53

solution (0.1 ml/l) has been measured (Malvern, Mastersizer); it is equal to –15 ± 5 mV. The absolute value of the zeta potential is relatively low indicating that there are not strong repulsive electrostatic interactions between the particles and the glass surface. As can be deduced by the classical DLVO theory [9], the adhesion force should be increased by the creation of additional chemical bonds between the particles and the glass surface. A diluted surfactant can increase the particulate removal but it has to be used in carefully controlled conditions. In our case, the diluted surfactant has no positive effect on the detachment. Further studies using another concentration should confirm these results.

Glycerol

For the final polishing step of the GaAs wafers, chemical products containing glycerol are often used to protect the surface by a hydrophilic oxide layer to prevent air contamination. We have used this product in our experiments to specify and to determine its role in particle removal.

Results have shown that the removal rate of the glycerol solution is lower than that for distilled water only. Effectively, 60% of particles remain stuck to the glass surface using glycerol in distilled water *versus* 55% with pure distilled water, as reported in Figure 6. It can be deduced that the glycerol is not an adequate product for the removal of the alumina particles on the glass surface. This result is confirmed by the measurement of zeta potential of the 0.3 μm particles suspended in the glycerol solution, which gives –11 ± 5 mV. The quite low absolute value of the zeta potential indicates that the adhesion force is higher in the diluted glycerol solution. The results obtained show that glycerol has a negative effect on the particle removal.

Influence of pH

To study the effect of pH on alumina particle removal, we have used 1 ml/l and 10 ml/l of concentrated NH_4OH diluted in distilled water to obtain various basic solutions ($9 < pH < 10$). For the concentration of 1 ml/l, 40 minutes was the minimum time necessary to achieve the complete sedimentation of the 0.3 μm particles and their adhesion on the glass surface. At a short resting time, the observations on the microscope show that the particles follow a chaotic motion. They are subjected to Brownian diffusion at a very small distance from the glass surface, leading to the suspicion that they are still not adherent to the surface. A longer resting time, *i.e.*, a longer settling time, is required to

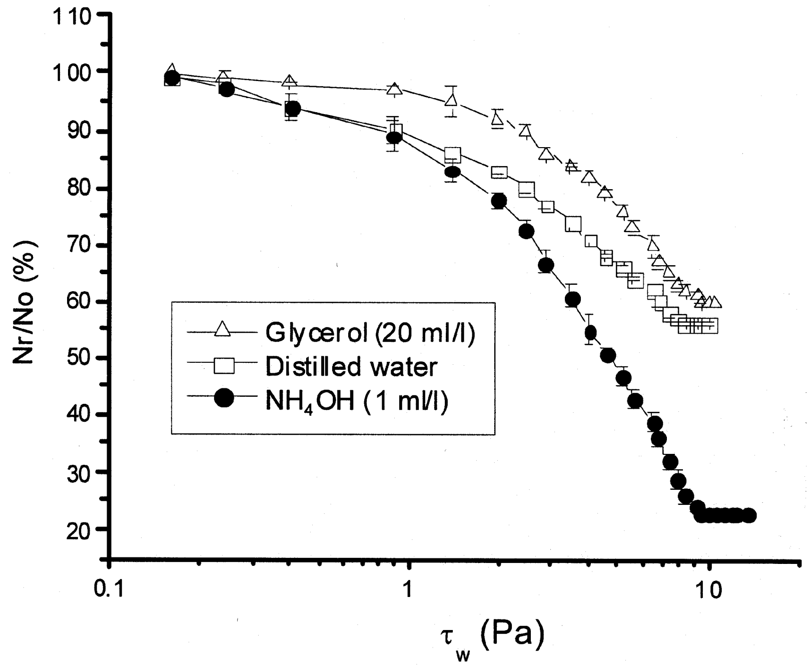


FIGURE 6 Number of 0.3 μm particles remaining in the observation area after dispersion in diluted glycerol and ammonia solutions.

overcome the energy barrier due to strong repulsive electrostatic effects at basic pH. Moreover, in the case of the 10 ml/l solution, it was impossible to start the measurements because the particles never adhered to the surface, whatever the resting time. In this latter case, the energy barrier due to very strong repulsive electrostatic effects cannot be overcome by the gravity force.

The removal efficiency of the 0.3 μm alumina particles obtained with the use of a basic diluted ammonia solution is shown in Figure 6. The results are in good agreement with the DLVO theory, which describes the energy of interaction between two charged surfaces in a polar medium, as previously found by other authors [11, 12]. According to the classical DLVO theory, the net energy of interaction is the summation of the attractive Van der Waals energy and the repulsive electrical double layer energy due to the negative charge of both alumina particles and glass surface. The effect of pH is based on the variations of the zeta potential of particles and surface. The absolute value of the zeta potential is lower at low pH.

Therefore, the electrical double layer repulsion is weaker, resulting in stronger adhesion at low pH, in accordance with the experimental results of Figure 6. In our experiment, the zeta potential of the 0.3 μm particles in the ammonia solution (1 ml/l) is found to be $-32 \pm 5\,\mathrm{mV}$. So, the absolute value of the zeta potential is high at basic pH, increasing the repulsive electrostatic forces between the alumina particles and the glass surface, both negatively charged. Particles are electrically repelled from the glass surface due to the higher magnitude of the negative zeta potential in the basic solution. We can see in Figure 6 that 80% of particles have been removed by the ammonia solution. This important result shows that the use of a basic solution containing ammonia (NH_4OH) to remove submicron alumina particles can be recommended. Moreover, in the case of semiconductor surfaces, besides increasing the electrostatic forces between the particles and the surface, ammonia can also etch the wafer surface to reduce the contact area, further reducing the particle adhesion force, roughly proportional to the contact area, and resulting in improving still more the effective cleaning of the semiconductor surface.

CONCLUSION

Suspensions of micron and submicron alumina particles are used for the mechanical polishing of the GaAs surfaces. However, some alumina particles remain stuck to the surface after the polishing step and it is very difficult to remove them by a simple rinse with DI water. Therefore, a post CMP cleaning step is often required to decrease this particulate contamination. A series of experiments using a shear stress flow chamber was performed to gain a better knowledge of adhesion forces and mechanisms acting between non-spherical particles and a plane surface. To this end, we have performed experiments with special chemical solutions to observe their effects on the modification of the physical and chemical forces existing between the alumina particles and the glass surface used as a model for a semiconductor surface.

The results have shown that the higher the resting time, the more difficult it is to remove the small particles stuck to the glass plate because of the increase of the adhesion force. Moreover, the effect is increased because of the non-spherical shape of the particles. Thus, in the microelectronics industry, semiconductor surfaces should be rinsed and cleaned quickly after the polishing step to avoid an increase of the adhesion force between the particles and the surfaces. Glycerol is an oxidizing product creating an oxide layer on semiconductors wafers.

It has no efficiency on the particle removal itself so it has to be used only when the surfaces are very clean.

This work has shown that diluted ammonia solution can be recommended for the removal of alumina particles on the glass plate because of the presence of high repulsive electrostatic interactions between the particles and the glass surface, both negatively charged in basic solution. Particles are electrically repelled from the glass surface due to the higher magnitude of the negative zeta potential in this solution. The diluted ammonia is also known to be effective for particle removal from semiconductor surfaces (Si and GaAs). Regarding the chemical cleaning processes applied in the microelectronics industry, such a study as reported here is helpful in understanding the removal mechanisms of submicron particles in contact with a flat surface.

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