Contamination Measurement and Control in the Semiconductor Industry

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ABSTRACT

The semiconductor industry routinely uses instrumentation capable of detecting particles as small as 0.01 μm in the air; 0.08 μm in water, and 0.1 μm on polished silicon wafer surfaces, all at relatively low concentrations. Gravity and diffusion processes dominate particle transport in atmospheric air, while electrical double-layer effects are important in liquid systems and depend on particle and surface properties. Particle removal from surfaces also depends on particle and surface properties and, while most removal techniques employ wet chemicals at present, optimum methods remain controversial and dry cleaning methods are becoming more common. These results can be extended to application areas other than the semiconductor industry.

INTRODUCTION

Particulate contamination is now a major threat to product yield in semiconductor manufacturing. Over the next few years, as the next generations of devices with smaller, submicrometer line widths and even larger chip areas come on line, the particulate threat is likely to become even more serious in spite of the measures already taken to minimize or eliminate particle-related chip losses. This paper reviews some of the contamination measurements and control technologies of the semiconductor industry with the goal of promoting technology exchange.

PARTICLE DEFINITION AND PROPERTIES

A particle is a stable cluster of molecules forming a discrete liquid or solid unit of matter, nominally 2 nm to 1 mm in size. This size range specification, spanning nearly six orders of magnitude, is somewhat arbitrary Molecular clusters smaller than about 2 nm are usually unstable. Clusters larger than 1 mm usually have other names and few of the small particle properties. Indeed, size is the primary property distinguishing particle behavior from the behavior of larger objects of the same composition. Settling velocity, diffusion rate, adhesion, light scattering, chemical reaction rate, and other properties depend strongly on size.

For example, terminal settling velocity (V_t), determined by the velocity through a gas at which Stokes drag force on a spherical particle is equal to gravitational force, is given by:

$$V_t = \left[\frac{d_p^2 (\rho_p - \rho_f)}{18\eta} \right] g = \tau g$$

where

d_p = particle diameter
p_p = particle density
p_f = fluid density
η = fluid viscosity
g = gravitational acceleration
τ = particle relaxation time

Equation (1) shows settling velocity varying directly with particle diameter squared, meaning small particles settle less rapidly than large particles. When gravitational settling is the dominant particle deposition mechanism, exposing a surface to equal concentrations of aerosol particle sizes would skew the particle distribution collected on that surface toward the larger particles—equal concentration of 0.5 μm and 5 μm particles in the surrounding air would result in higher concentrations of 5 μm particles on the surface after a given exposure.

Gravitational settling is not the only important mechanism whereby aerosol particles deposit on a surface, however. Brownian diffusion—the motion of small particles brought about by molecular collisions—is also size dependent. Particle diffusion coefficient varies inversely as the first power of sphere diameter. Thus, a surface held vertically (deposition by diffusion only—no gravitational settling) in an aerosol made up of equal concentrations of aerosol particle sizes would collect more small particles than large particles—equal concentrations of 0.5 μm and 5 μm particles in the surrounding air would result in higher concentrations of 0.5 μm particles on the vertical surface after a given exposure time.

Still another size-dependent property of importance: van der Waals adhesion forces to a planar surface depend directly on

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particle diameter (~ d_p), meaning that the adhesion force per unit mass (~ d_p^3) varies inversely with the second power of particle diameter. This conclusion means that small particles are more difficult to remove than larger particles. Gravitational forces, varying with sphere diameter cubed, exceed van der Waals adhesion forces for large particles—a BB falls off a glass slide when turned upside down—but not for small particles—a 0.1 mm dust particle remains attached to a glass slide when inverted.

PARTICLE MEASURING INSTRUMENTATION

Control of particles in the wafer manufacturing environment presupposes adequate capability for measuring particles in all the manufacturing environments—air, water, and processing chambers, including subatmospheric—and also on the surface of product wafers, initially highly polished and nearly optically flat but ultimately patterned and nonplanar. The underlying principle used in measuring particles in all these environments and conditions is optical scattering, although the instrument design for each environment or particle state differs significantly. This section reviews each use.

COUNTING PARTICLES IN AIR

Optical particle counters (OPCs) can count and size aerosol particles as small as 0.05 μm. Minimum detectable size varies with sample flow rate, the lowest size detection limits being achieved only at the lowest flow rates. In the typical OPC design, the aerosol being measured is aerodynamically focussed into a narrow stream by surrounding it with particle-free sheath air. The aerosol stream passes through the laser beam from which light is scattered and detected. A photodiode, or other light sensor, receives the scattered light and generates an output pulse, counted and classified according to size by a pulse height analyzer. A calibration curve converts the size and number data into a size distribution.

Aerosol particles smaller than 0.05 μm can be counted using a condensation nucleus counter (CNC). A CNC is an OPC with an upstream particle growth stage. Particle growth takes place by condensation of vapor in a super saturated chamber. The aerosol stream being measured initially flows through a chamber saturated with vapor at a given temperature. When this saturated aerosol stream then enters a chamber of lower temperature, the stream becomes supersaturated and condenses on all available nucleation sites, enlarging their diameters by factors of up to 1000. At these greatly expanded sizes, particles initially invisible to the OPC become easily detected and counted. However, size information is lost in the enlargement process so that the CNC output is just a total number count greater than a specific size, typically 0.003 μm to 0.02 μm.

COUNTING PARTICLES IN LIQUIDS

Light scattering is also the principle used for counting and sizing particles in liquids. Designs virtually identical to those used for measuring aerosols but adapted for liquid streams dominate the contemporary instrumentation. The higher indices of refraction characteristic of liquid media reduce the light intensity scattered from a given particle.

Another factor of some practical importance in counting particles in liquids is that air bubbles also create a region of differing index of refraction that scatters light and thus produces a pulse in the instrument detector that can be mistaken for a particle.

In spite of these complications, contemporary instruments can detect polystyrene latex (PSL) spheres in water as small as 0.08 μm. This size sensitivity is achieved at low volume flow rates.

COUNTING PARTICLES IN VACUUM SYSTEMS

Aerosol particle counters rely on aerodynamic focusing for confining the aerosol stream to the region illuminated by the laser light source. In vacuum sytems, this option no longer exists so conventional aerosol particle counting instrumentation is not usable in the low-pressure environments characteristic of vacuum coating operations. Optical scattering principles, however, still apply and indeed perform even better in that a primary source of background noise in the conventional aerosol counter—air or vapor molecules—is present in greatly reduced numbers. And commercial instrumentation, based on optical scattering, does exist by which unattached particles in vacuum systems can be detected and sized. These instruments are referred to as particle flux meters in that they measure particle transit through a plane defined by a fixed light beam. In general, no built-in focus or beam control is part of this instrumentation which relies instead upon the particle transport mechanisms present in the system being evaluated. The performance and capabilities of this type equipment are discussed in detail elsewhere in these Proceedings [1].

COUNTING PARTICLES ON A SURFACE

Optical scattering is also the basis of sophisticated instrumentation, called laser scanners, for counting particles on surfaces. When these are planar and optically flat, the smallest PSL sphere size detectable can be near 0.1 μm.

The addition of a reflecting plane adjacent to a transparent sphere makes the scattering problem much more complicated than scattering from a sphere alone. The common calibration of the relationship between sphere size and scattering intensity is experimentally determined by depositing monodisperse PSL spheres of known size on otherwise clean surfaces and measuring the response. Figure 1 shows such a relationship for a specific laser scanner, measuring PSL spheres on polished silicon wafers. The relationship is non-monotonic in the region where the sphere size is comparable to the wavelength of the incident light. Thus, particle sizing by pulse height analysis alone cannot be carried out in this size range for this sphere-surface system.

Figure 1 probably represents a worse case in that the scattering particles are all transparent spheres and the scattered light is collected over a small, solid angle. With absorbing particles of irregular shape and scattered light collected over large solid angles, the relationship between particle size and scattering intensity becomes more monotonic. Simply flattening the PSL spheres by heating them briefly to their softening temperature eliminates much of the oscillation seen in the Figure 1 curve.

Designing a scanner that illuminates the surface with two laser beams, one polarized perpendicular to the scattering plane and one polarized parallel to the plane provides a means of obtaining a monotonic size-scattering relationship even for transparent spheres [2].

The optical properties of the surface are also an important variable affecting the scattering intensity from a surface particle. Simply changing the surface reflectivity alters the scattering cross section of a particle resting on the surface. In Figure 2, the scattering from various sized PSL spheres deposited on silicon wafers coated with various thicknesses of oxide is shown to vary with the surface reflectivity. Any coating on a flat surface that changes reflectivity also changes the calibration curve between scattering intensity and particle size.

When the surface is nonplanar, additional scattering centers are present which interfere with the identification of surface particles. In certain cases where regular, predictable surface geometries are present, the interfering scattering can be minimized by (1) orienting the sample surface with respect to the scattering plane; (2) increasing the angle of incidence of the primary beam (measured from a perpendicular to the illuminated surface) to near 90° (the incident light strikes the surface at a very low grazing angle); (3) signal processing to eliminate all signals appearing on adjacent chips and counting only those pulses unique to one chip or the other; and (4) filtering known pattern signals in the Fourier plane and reconstructing a holographic image containing the unfiltered signals. Commercial equipment, based on one or the other of these approaches, is in use in the semiconductor industry for silicon wafer inspections at various stages of production. These instruments typically cost in the quarter million to a million dollar plus range.

PARTICLE DEPOSITION ON SURFACES

Particles in the product environment per se do not degrade product quality. It is those particles that end up on the product surface before, during, or after processing that cause problems. How particles travel from a generating source to the product surface depends on the environment.

In air at atmospheric pressure, the two primary transport mechanisms are gravity and diffusion. Particle size plays an important role. Large particle (PSL spheres > ~0.2-0.3 μm) deposition is dominated by gravitational settling; small particle (particle diameter $\leq$ 0.1-0.2 μm) by diffusion. A typical set of calculated values of particle deposition velocity ($\equiv$ particle flux/ particle concentration) is shown in Figure 3.

Electrical forces increase deposition velocity over the diffusion branch (see Figure 4) while thermal gradients decrease deposition velocity in the transition region between the two branches.

In liquids other mechanisms of particle deposition become more important than gravity or diffusion. Particles in solution become surrounded by charged ion sheaths depending on solution pH. Ions adhere to the particle and drift with it in the presence of electric fields. By measuring the drift velocities of a particle in a known electric field, particle potential, called the zeta potential, can be determined. Particles and surfaces of the same sign of zeta potential repel each other so that contact between the two is prevented. This effect, called double-layer repulsion, dominates many particle-surface systems in liquids, keeping the surfaces freer of particles than would be true if the repelling electric forces were not present. Thus, understanding particle deposition in liquids requires knowledge of the charge states of both the particle and the surface in those liquids. In liquids, electrical charges with the right sign and magnitude can prevent product contamination by particles.

Particle deposition in vacuum systems would seem to be a simpler problem dependent only on gravitational settling—rather than diffusion and flow field. At high vacuum this conclusion is true, but during pumpdown and venting of vacuum systems, flow fields can be important. Pumping rate is also important in affecting temperature drops within a process chamber which can cause super saturation and condensation of species in the chamber atmosphere.

Much like the action of the condensation nucleus counter previously described, condensation collects on the small particles present. Alternatively, condensation can occur homogeneously. In both cases particles form in the chamber. These particles can fall onto work surfaces before vaporizing, sometimes leaving an objectionable residue or a nucleating center on the product surface. In effect, the condensation action has simply enhanced the transport of contamination to a surface.

Electrical forces are also important particle collection mechanisms in vacuum systems. Many particles generated in a vacuum system are electrically charged. The fields created by individually charged particles and ensembles of charged particles precipitate particles on surfaces in the system, including product surfaces. Elimination of electrical charge is a desirable condition for minimizing particle deposition, but electrical forces can also be used to remove particles from the chamber environment by depositing them on collecting surfaces inserted in the system expressively for that purpose.

PARTICLE REMOVAL

In spite of all precautions to prevent particle deposition on work surfaces, some particles inevitably will end up on a product surface and require removal. Particle removal implies some technique for overcoming particle adhesion to a surface.

Particle adhesive forces consist of not only the ever present van der Waals forces alluded to earlier but can also include electrostatic, capillary, and chemical bonding. Regardless of the source of particle adhesion, particle removal requires the application of forces to the particles that exceed the adhesive forces.

The most common methods for removing particles in the semiconductor industry involve wet chemistry at present. In liquids, van der Walls forces are lower than in air, and capillary forces are offset by liquid immersion. In addition, liquids are more dense and hence a more suitable medium for applying drag forces to a particle.

Three basic forces for removing particles are (1) centrifugal force, (2) hydrodynamic drag, and (3) vibrational force.

Centrifugal forces are those created by radial acceleration as in a rotating spinner. Hydrodynamic drag is the force exerted on a stationary object by a fluid flow. Vibration force refers to the force created by an oscillatory motion and the attendant accelerations perpendicular to the surface.

Spin rinse/dryers often use a combination of both centrifugal force and hydrodynamic drag from a water spray. Water sprays used alone, with pressurized liquids or as air jets, rely on hydrodynamic (or aerodynamic) drag to dislodge a surface particle. Ultrasonic and megasonic cleaners are both examples of vibratory cleaners, although ultrasonic cleaning in liquids is often dominated by cavitation—the formation and collapse of bubbles in the liquid with the accompanying high release of energy to surface particles.

Choice of liquid is important in hydrodynamic cleaning and vibrational cleaning. A high-density liquid is desirable for maximizing drag force, but a low viscosity is also wanted to reduce the boundary layer thickness and allow the high drag forces to be exerted on smaller particles than would be possible with fluids of higher viscosity.

The choice of liquid also influences the value of van der Waals force, as do the composition of the particle being removed and the surface being cleaned. An optimum fluid is one that minimizes van der Waals adhesion and at the same time maximizes hydrodynamic drag and minimizes boundary layer thickness.

Trial and error governs most selections at present.

Dry cleaning methods such as plasma cleans, uv ozone, and various frozen gases (CO_2, Ar, H_2O) are gaining in popularity, especially for in-situ cleaning in vacuum chambers. This area remains one of active development.

CONCLUSIONS

The semiconductor industry employs sophisticated instrumentation for measuring particulate contamination in manufacturing and has developed some useful control strategies for minimizing particulate damage. While this technology continues to evolve, some of the established methods may be of value in vacuum coating.

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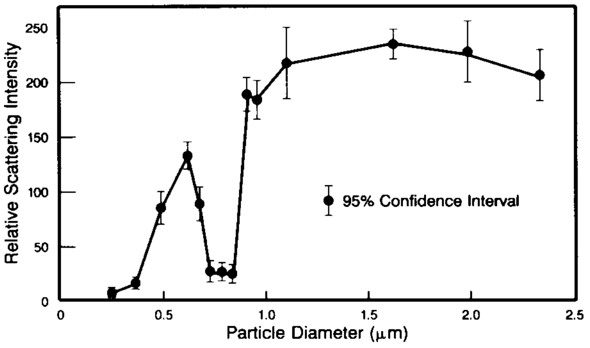

Figure 1. Relative scattering intensity of PSL spheres on silicon.

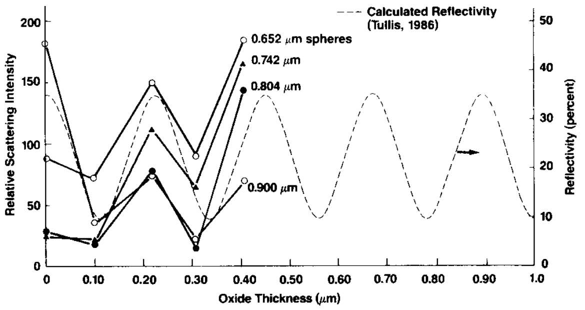

Figure 2. The effect of surface reflectivity upon scattering [3].

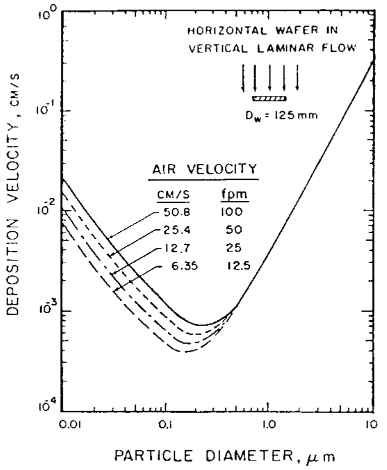

Figure 3. Mean deposition velocity for a wafer in a VLF clean room [4].

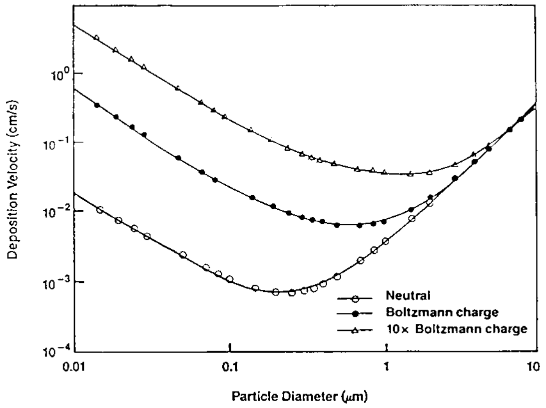

Figure 4. Deposition velocity including electric field effects [5].