

Particle Size Metrology: Comparison Between Aerosol Electrical Mobility and Laser Surface Light Scattering Techniques

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Abstract. Two different methods used to determine the size of polystyrene spheres were found to disagree by about 5 %. One of the methods, differential mobility analysis, measures particles in the aerosol phase, while the other, laser surface light scattering, measures them bound to a surface. It is believed that the observed discrepancy results from deformation of the particle when it is bound to the surface. The implications of such a deformation on scanning surface inspection system calibration is discussed.

Keywords: differential mobility, light scattering, and particle

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INTRODUCTION

Scanning surface inspection systems (SSISs) are used by wafer suppliers and integrated circuit manufacturers to inspect bare front-end silicon wafers for particle contamination, surface roughness, and other surface defects. These tools use diffuse light scattering to detect the presence of features. Accurate calibration of their response, using certified reference particles, is required in order to provide consistent compliance with wafer specifications. These particles are generally spherical, consist of polystyrene (PS), and have certified diameters ranging from 60 nm to 1 μm . Current standards call for relative uncertainties (with 95 % confidence) in these diameters of less than 3 % [1].

Each tool can classify particles and defects according to their light scattering equivalent (LSE) diameter. An issue arises when a different scattering geometry is used by each inspection tool. Two tools with different geometries can be calibrated to yield the same results for PS spheres; however, real contaminant particles will usually have different shapes and refractive indices, resulting in different light scattering distributions, and thus different LSE diameters measured on one tool versus that measured on another.

Recently, there has been interest in improving the calibration of SSISs by modifying the interpolation scheme between calibration points. Currently, interpolation is usually linear on a log-log scale. However, the calibration curve can change significantly if the set of reference particles changes, especially for larger particle sizes. As an alternative, a physical-model-based calibration, which accurately accounts for the scattering conditions (e.g., geometry, polarization, and wavelength), can effectively minimize this problem. The model must accurately describe the scattering function for the reference particles.

In this proceeding, we describe measurements of polystyrene sphere diameters performed using two independent methods. One of the methods is performed on the spheres dispersed in the gas as an aerosol, before the particles have been deposited onto a wafer, and the other is performed on the wafer *in situ*. The two methods are found to disagree, in a manner which is consistent with the particles being deformed on the wafer.

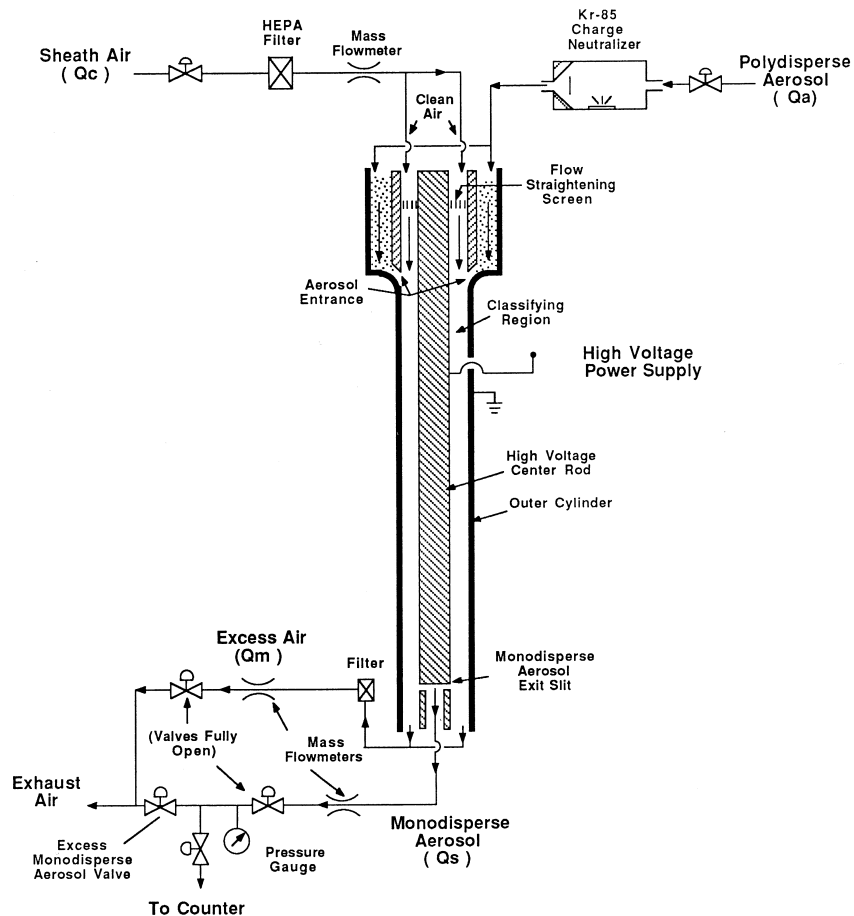


FIGURE 1. A schematic of the DMA.

EXPERIMENTAL METHODS

We have performed measurements of particles both as an aerosol and bound to silicon wafers. The aerosol measurements use a differential mobility analyzer (DMA) to balance the Stokes drag force, which is related to sphere size, with an electrical force acting on the singly charged spheres. The measurement of particles after deposition on wafers is based upon angle-resolved laser surface light scattering (LS). For both measurements, theories exist to describe the physical process by which the diameter is determined, and those theories are accurate enough to estimate the uncertainty in the measurements. These methods are described in the following subsections.

Aerosol Electrical Mobility

The system includes a generator for producing a PS sphere aerosol, a differential mobility analyzer for

selecting a monodisperse size fraction of the aerosol, and then a chamber to electrostatically deposit the spheres onto wafers. Here we focus on the DMA, shown in Fig. 1. A brief description of the instrumentation and methodology is given below; a detailed description is given by Kinney, *et al.*[2].

Aerosol particles are generated from a dilute aqueous solution of nearly monodisperse polystyrene spheres by the electrospray method. Electrospray results in particles having little or no residue and very low multiplet concentration. The particles leaving the generator pass through a bipolar charger that produces a charge distribution that depends only on the size of the particles and not on their initial charge. For 100 nm particles, about 45 % of the particles are uncharged, about 20 % have +1 electron, another 20 % have -1 electron, and much smaller fractions have multiple charges. The aerosol then passes into the DMA, which consists of an inner cylindrical rod connected to a variable high voltage dc power supply and an outer annular tube connected to ground. Clean

sheath air flows through the axial region, while the charged aerosol enters through an axisymmetric opening along the outer cylinder. The positively charged PS spheres move radially towards the center rod under the influence of the electric field. Near the bottom of the classifying region, a fraction of the air flow consisting of near-monodisperse aerosol exits through a slit in the center rod.

The quantity measured by the DMA is the electrical mobility, Z_p , defined as the velocity a particle attains under a unit electric field. Knutson and Whitby [3] derived an expression for the average value of Z_p for particles entering the slit involving the peak electrode voltage, V , the sheath air flow rate, Q_c , the inner and outer radii of the cylinders, r_1 and r_2 , and the length of the central electrode down to the slit, L :

$$Z_p = \frac{Q_c}{2\pi VL} \ln(r_2/r_1) \quad (1)$$

This equation is valid provided the sheath air flow, Q_c , is equal to the excess flow, Q_m , leaving the classifier. They derived an expression for the transfer function, defined as the probability that a particle will leave the sampling slit. The transfer function is of great importance, because the monodisperse concentration exiting the DMA is proportional to the convolution of the transfer function with the particle size distribution function. The transfer function has a triangular shape with a value of 1 when the voltage at which the computed particle mobility using Eq. (1) is equal to the mobility of the particle. The ratio of the base of the transfer function triangle to the peak voltage is predicted to be $2(Q_s/Q_c)$, where Q_s is the flow of mono-disperse aerosol. From this one can see that the smaller the flow ratio the more monodisperse is the aerosol leaving the classifier.

The relationship between electrical mobility and particle size is obtained by equating the electric field force of a singly charged particle with the Stokes friction force,

$$Z_p = \frac{eC(D)}{3\pi\mu D}, \quad (2)$$

where μ is the dynamic viscosity of air, e is the electron charge, and D is the particle diameter. The Cunningham slip correction, $C(D)$, corrects for the non-continuum gas behavior of small particles.

The output of the DMA can be sent to a condensation nucleation counter, allowing the number concentration to be measured as a function of DMA voltage. Thus, the size distribution can be measured.

Alternatively, the voltage can be set to the peak of the size distribution, and the output stream of the DMA can be sent to a system that electro-statically deposits the particles onto a silicon wafer.

Laser Surface Light Scattering

For monodisperse spheres deposited onto silicon wafers, laser surface light scattering can be a very sensitive method for determining their size. Here, light from a 390 nm laser source (achieved with an intracavity-doubled Ti:sapphire laser) is incident onto the sample at a fixed incident angle of $\theta_i = 60^\circ$ with its electric field parallel to the plane of incidence. The distribution of the scattered light is then measured as a function of scattering polar angle, θ_s . An illustrative set of data is shown in Fig. 2.

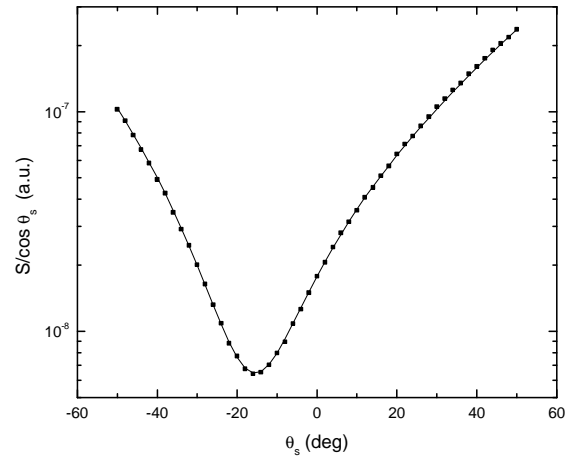


FIGURE 2. Light scattering data (points) and a best fit to the model (curve) for 100.7 nm PS spheres deposited on a silicon wafer. The non-linear least squares fit yielded a diameter of 95.6 nm with a type A contribution to the uncertainty ($k=2$) of 0.1 nm.

Bobbert and Vlioger [4,5] developed an exact solution to the scattering of light by a uniform spherical particle above a planar substrate. This theory can be extended to include coatings on the substrate and the sphere, and can be further extended for slightly nonspherical, but axisymmetric particles. The experimental results are fit to the data, allowing the diameter of the particles and the surface number density of particles to be free parameters. The diameter of the particles affects the shape of the curve, while the number density affects only the overall scaling of the data. Other parameters, such as the indices of refraction of all of the materials, the thickness of the substrate surface oxide, and the wavelength of the light are held constant, and

uncertainties in these values are included in the uncertainty for the particle diameter. Figure 2 shows a best fit of the model to measured data.

A previous study reported a measurement of the 100.7 nm (SRM[®] 1963) spheres and found the diameter to be 99.7 nm ± 1.7 nm. [6] The largest source of uncertainty was due to the sample-to-sample variation generated by the presence of doublet particles produced by pneumatic nebulization. By using the electrospray method to generate the particles, the number of doublets was significantly reduced, alleviating the need to attempt to correct the data for the presence of those doublets and reducing the statistical fluctuations which were due to the limited number of doublets and their random orientations. Furthermore, examination of a number of literature values for the indices of refraction of the substrate and the substrate oxide, reduced other sources of uncertainty.

RESULTS

Polystyrene spheres with diameters ranging from 56 nm to 269 nm were measured using the two different techniques. Figure 3 shows results of the comparison between the diameter measured by the aerosol method, D_{DMA} , and that measured by the light scattering technique, D_{LS} . The uncertainty for each point was determined by combining the uncertainties from each of the two methods:

$$u = (u_{DMA}^2 + u_{LS}^2)^{1/2} \quad (3)$$

The difference between the two methods is not only outside the uncertainties in the measurement, but they are outside the limit imposed by the 3 % requirement. The difference appears to be approximated by a linear function:

$$D_{LS} - D_{DMA} \cong 0.053D_{DMA} \quad (4)$$

DISCUSSION

The most likely explanation for the difference is deformation of the particle due to its adhesion onto the surface. Polystyrene spheres having diameters ranging from 1.5 μm to 12 μm and deposited on silicon substrates have been observed to have contact radii given by approximately [7]

$$a = 0.18(D/\mu m)^{1/2} \mu m. \quad (5)$$

If one were to assume that this expression could be extrapolated to the sphere sizes studied here, one would find that spheres having diameters less than 125 nm would have contact diameters larger than their sphere diameter. So, it is reasonable to assume that the spheres deform significantly, although Eq. (5) is probably a gross overestimate in the size regime studied here.

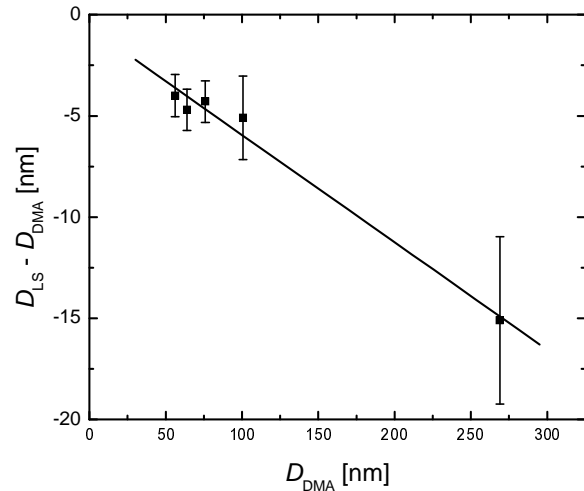


FIGURE 3. Comparison between DMA and LS measurements.

Deformation of a sphere, consistent with surface adhesion, would be expected to shift the center of mass of the particle closer to the substrate. While the exact profile of the deformed sphere is unknown, calculations of the scattering by a number of different possible deformation models suggest that the most important parameter determining the shape of the curve in Fig. 2, and thus a particle's apparent size, is the distance the center of mass lies from the substrate. Deformation thus reduces the apparent size of the sphere as seen by angle-resolved light scattering, consistent with the findings shown in Fig. 3.

Since integrated light scattering is the primary method for inspecting wafers, reference particle deformation may have a substantial consequence on inspection system calibration. One philosophy is that the measure of the LSE includes deformation of the sphere, and that as long as the spheres are accurately sized before deposition, they represent a well-defined point in the calibration of a scanner. However, problems are known to arise, especially for larger particles, when one changes the set of calibration particles. These problems occur due to the use of a non-physical response curve, which is often taken to be piecewise linear on a log-log graph, in a size regime where the scattering has significant structure.

Efforts are underway to improve the calibration procedure by using physics-based response curves, using accurate models for the scattering. By using a physics-based response curve, the instrument calibration should be less susceptible to changes in the calibration particle set. However, for this goal to be realized, particle deformation might need to be included in the scattering model.

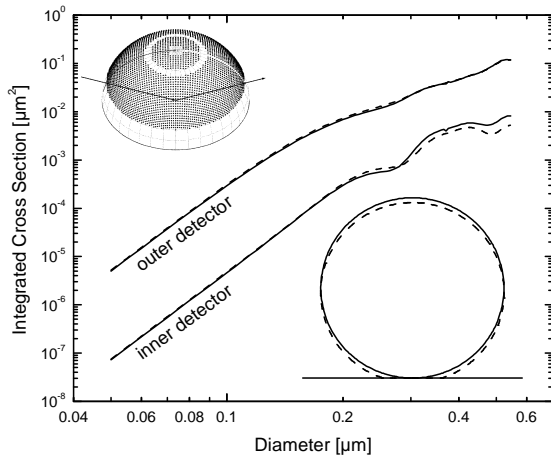


FIGURE 4. Calculated response for two scattering channels as a function of particle diameter for spherical particles (solid curves) and deformed particles (dashed curves). The profiles of the 0.05 μm particles are shown in the lower right of the figure. The scattering geometry is shown in the upper left of the figure. The upper pair of curves corresponds to the outer detector, while the lower pair of curves corresponds to the inner detector.

To assess the importance of particle deformation on the calibration curve, a simulation was performed assuming 488 nm p-polarized light incident at 70° and light collection over two channels, an inner one collecting light between 6° and 20° of the surface normal and an outer one collecting light between 25° and 70° of the surface normal. The integrated cross sections for spherical and deformed particles were calculated as a function of sphere diameter. The profile of the deformed particles was that of a sphere displaced into the surface by a distance $d = 0.027D$, consistent with Eq. (4). The overall sizes of the deformed particles were renormalized so that their volumes matched those of their spherical counterparts. While we have no details for the actual shape, something close to this particular shape is plausible, and the calculations are expected to yield an approximate amount of error that one might expect to observe.

Figure 4 shows results of this simulation. For particles smaller than about 200 nm, the result of deformation is small. The integrated intensity, in

contrast to the angle-resolved intensity, is much less sensitive to the shape than to the volume of the particle. The approximately 1 % error in particle diameter can easily be corrected by a uniform vertical shift in the modeled curve. For larger diameters, the effect is much more noticeable, especially for the inner channel. For these larger particles, the response curves cannot be mapped onto one another by a simple vertical shift (constant factor). Thus, without further information about the particle shape and degree of deformation, consistent model-based calibration of SSISs may be difficult.

CONCLUSIONS

Angle-resolved laser surface light scattering consistently yields smaller diameters by about 5 % than differential mobility analysis, when a spherical particle model is used as a basis for analyzing the light scattering results. This finding is consistent with deformation of the particles when they are bound to the substrate. Such a deformation should have little effect on model-based calibration of SSISs for diameters less than about 200 nm, but may be an important factor to consider for larger particles.

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