Communication

A Transfer Function for Relating Mean 2D Cross-Section Measurements to Mean 3D Particle Sizes

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It is common practice to estimate mean 3D particle and grain size of polycrystalline materials by multiplying 2D cross-sectional measurements by a multiplication factor. However, the most frequently used multiplication factors apply only to uniform or specific dispersions of particles, and therefore can provide misleading results. In the present work, empirical equations are developed to more accurately predict the mean 3D grain size of a lognormal spherical particle dispersion, regardless of the dispersion's width. The equations provide an improvement over scalar multiplier values by allowing the effects of particle size distribution to be accounted for using inputs that can be obtained by cross-sectional analysis.

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3D particle size is an important input parameter in many microstructure evolution models,[1–6] yet it can only be directly measured *in situ* for a very small percentage of materials. While serial sectioning can be used to obtain the 3D attributes of polycrystalline materials, it is either prohibitively expensive or impractical for the majority of applications. Often, the researcher must instead utilize stereology to estimate the 3D sizes from observations on a 2D cross-section. Stereologically appropriate 2D measurements are either lineal intercepts (the chord length of linear probe intersections with volumetric features) or section areas (the intersection areas of a planar probe cutting through volumetric features).[7] The lineal intercepts or section areas can then be used to predict the 3D caliper diameter distributions using either the Cahn–Fullman or Johnson–Saltikov methods, respectively.[8] However, both techniques suffer from relatively large error propagation issues (*e.g.*, as described in Reference 9).

In the absence of sufficient 2D distribution data, many researchers will instead resort to estimating the mean 3D particle size through the use of a scalar multiplication factor, denoted here as κ. These values are often based on geometric constants. For example, the diameter of a single sphere is equal to 1.5 times the average randomly oriented chord length through its volume. As such, multiplication of the mean 2D lineal intercept measurement by 1.5 to estimate the mean 3D particle size is an acceptable practice when the particles in a system can be reasonably approximated by a collection of uniformly-sized spheres. However, this multiplication factor is no longer accurate when the system contains spherical particles with a distribution of sizes. In fact, the correct value of κ will *always* be less than 1.5 when the particle sizes follow a lognormal, gamma, or Rayleigh distributions.[7,10,11] Additionally, it is worth noting that non-spherical shapes have different caliper-size-to-intercept ratios. Uniform distributions of regular polyhedrons can have values of κ ranging from 1.5 for a sphere to 2.25 for a cube,[7,12] with even higher values for shapes representing non-equiaxed grains: The proper multiplication factor for any distribution is therefore also a strong function of the expected particle shape and 3D distribution.

An alternative multiplication factor focusing on polycrystalline grain structures was proposed by Mendelson in 1967 and took into account the effect of both particle shape and distribution.[11] Mendelson began by determining the correct κ value for a uniform dispersion of tetrakaidecahedral particles, $\kappa_0 = 1.7756$, as this was expected to more accurately represent an equiaxed grain structure than a sphere. Mendelson then obtained the expected total surface area per unit volume of a lognormal distribution of particles with identical shapes and differing sizes. This function was given in terms of the lognormal shape parameter, σ, the statistical value that controls the variance of a lognormal distribution. Adjusting for the assumption of tetrakaidecahedral particles, this gave the following equation:

$$K = \frac{\bar{D}}{\bar{L}} = 1.7756 \times (e^{-2.5(\sigma^2)}) \qquad [1]$$

where $\bar{D}$ is the mean 3D caliper diameter, $\bar{L}$ is the mean 2D lineal intercept through the volume, and σ is the lognormal shape parameter of the 3D particle distribution. This approach is not limited to tetrakaidecahedra. It can also be applied to any particle shape assuming an appropriate adjustment is made to κ_0.

As pointed out by Mendelson in the original work,[11] Eq. [1] is of little practical use because the σ for the 3D distribution must be known *a priori*. At the time (prior to automated serial sectioning), very little reliable data

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existed for reasonable estimation of expected ranges of σ based on the composition or thermal and mechanical history of a specimen. Mendelson used experimental data from Feltham[13] to predict that a typical equiaxed polycrystalline material would likely have a σ value of 0.2291, giving a κ value of 1.558. As a verification, he found a similar result, $\kappa = 1.570$, using Hillert's distribution function for steady state growth in place of a lognormal distribution.[11,14]

To date, Mendelson's multiplication factor is the most generally accepted method for estimating 3D particle dimensions from lineal intercept data, as evidenced by inclusion in the appendix of ASTM E112.[15] However, it cannot be assumed that a multiplication factor in the range of 1.5 to 1.6 would be appropriate for all particle or grain size measurements in microstructures because not all particle distributions have identical values of $\sigma = 0.2291$.[16,17] Additionally, as has been noted many times elsewhere, Hillert's distribution skews the opposite direction from what is typically observed in material microstructures.

From inspection of Eq. [1], it appears that it should be possible to obtain an improved estimate of 3D particle size using an empirical function relating a measurable 2D metric of dispersion to κ for a given particle shape and distribution type. In the present work, we develop such a transfer function computationally by using simulated particle size distributions to relate the standard deviation of lineal intercepts and section areas (which are measurable in 2D) to the multiplication factor, κ.

For this study, synthetic microstructures were generated containing random lognormal dispersions of spherical particles. In each simulation, a lognormal dispersion of spheres was packed into a cubic voxel array measuring 1500 voxels on each side. This was done by randomly sampling particle diameters from a lognormal probability density function with a fixed mean of $\bar{D} = 30$ and a given lognormal shape parameter σ as shown in Eq. [2], until the sum of the volumes of the spheres reached 25 pct of the total volume of the cubic array.

$$f_V(D) = \frac{1}{D \times \sigma \times \sqrt{2\pi}} \times \exp\left(- \frac{\left(\ln\left(\frac{D}{\bar{D}}\right) - \frac{\sigma^2}{2} \right)^2}{2\sigma^2} \right) \quad [2]$$

To ensure these lists accurately represented a lognormal distribution, the mean of each list was restricted to less than 1 pct variation from 30, and the Shapiro/Wilk test for normality[18] was required to return a p value of 95 pct or greater on the log of the values in the list. If either of these criterion was not met, the list of particle volumes was rejected and a new random list was generated from the distribution.

Once these lists of particle diameters were obtained, spheres of corresponding volumes were randomly placed inside the synthetic microstructures, starting from largest and going to smallest with centers at floating point coordinates to avoid any grid-based bias. Spheres were not allowed to come within 1 voxel of another sphere or extend outside the simulated volume boundaries, in order to prevent edge effects. An example of a resulting microstructure with $\sigma = 0.99$ can be seen on the left hand side of Figure 1.

Every simulated microstructure was given an initial σ between 0 and 0.99 at intervals of 0.01, and enough simulations were run at each value of sigma to result in at least 100,000 spheres. This resulted in a total of approximately 11.12 million individual spheres across 735 simulated microstructure, grouped into 100 families based on the initial sigma chosen to generate the spheres.

Once all simulations were complete, a probability plot was made from the diameters for each family of data, as a final assurance of lognormality. Figure 2 shows all 100 probability plots in a single plot, grouped by color into sets of 10 to help aid the eye, with the lightest color lines in each series corresponding to $\sigma = 0.0, 0.1, 0.2,\ldots,0.8, 0.9$. This figure validates the claim of lognormality, while also highlighting problems related to resolution, sample size, and common misunderstandings about the lognormal distribution. In terms of resolution, it is often necessary when imaging real materials to define a minimum particle size below which an apparent feature has a high likelihood of being a "false positive" due to imaging noise, and is effectively no longer observable. Note in Figure 2 that that all plots with $\sigma > 0.68$ have 1 pct or more of their particles represented by a diameter of three or fewer voxels, as indicated by the dashed lines. An analysis with a minimum particle size of three pixels would not register the contribution of these particles to the distribution, thereby shifting down the rest of the plot and distorting the observed mean value. The fraction of particles excluded by a given minimum particle size requirement varies with σ. An oft-quoted rule-of-thumb is that sufficient analytical resolution for particle size measurement requires the mean particle diameter to be the equivalent of at least 10 pixels. Three voxels is 1/10th of the nominal mean 3D particle diameter used in all of our simulations. Considering that Feltham's estimation of σ for equiaxed polycrystalline grains[13] is significantly less than 0.68, the rule-of-thumb appears appropriate for most applications.

The choice of sampling methodology can also affect the large particle side of the distributions. Despite there being over 100,000 spheres sampled in each dataset, the right-side tails of all datasets exhibit deviations from lognormality, especially points representing diameters of 300 or greater. This is because it is very difficult to sample enough values from a widely dispersed population to properly characterize the right-side tail. Failing to do so can throw off the calculated mean. As such, when collecting data from cross sections, it is important to sample enough values to accurately represent the population. Since the impact of feature resolution and sample size on accuracy are both non-linear functions of σ, there is no minimum requirement for either that is applicable to all situations.

Finally, it is worth noting that even though every distribution has a $\bar{D}$ between 30.3 and 29.7, most plots

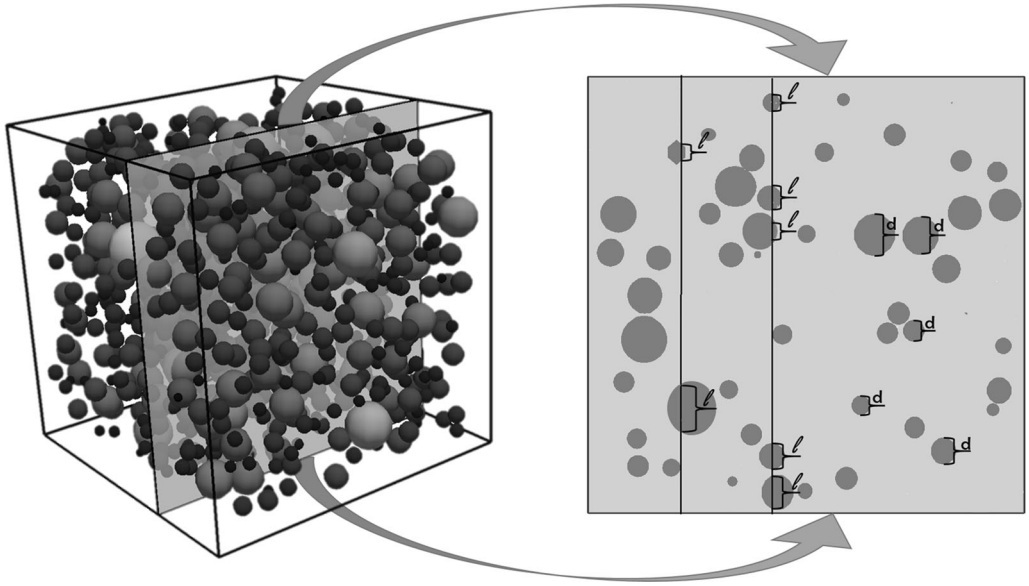

Fig. 1—A simulation used in this investigation is shown on the left, along with a single cross-sectional cut on the right. For each cross section, all sphere sections were recorded to find the circle-equivalent diameters, whereas each column and row was also analyzed independently to determine the linear intercepts.

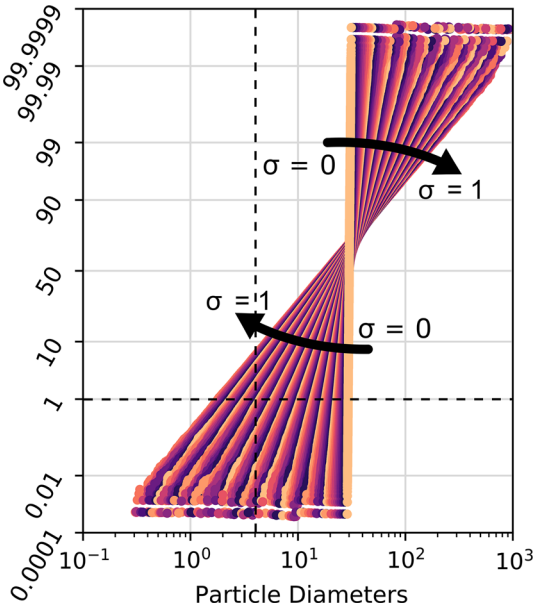

Fig. 2—A lognormal probability plot showing the spherical volumes for each analyzed dataset of different expected σ from 0.0 to 0.99 in steps of 0.01. Data conforming to a lognormal distribution will follow a straight line and intercept the 50th percentile at e^{μ}. Coloring cycles from dark to light in groups of ten, such that 0.0, 0.1, 0.2, 0.3, and so on each correspond to a light set of points.

do not pass though the 50th percentile at $D = 30$. That is because the lognormal distribution is asymmetric, and as such the mean and median are not generally the same value. The two are linked by Eq. [3], but the implications of this distinction are often overlooked when discussing lognormally distributed particle data.

$$Median = \bar{D} \times \exp\left(\frac{\sigma^2}{2}\right) \quad [3]$$

After verifying lognormality, exhaustive cross sectional analysis was performed on each simulated microstructure. Every column in the X, Y, and Z directions was analyzed, and the length across every intersected grain recorded to simulate the most likely dispersion of chord lengths (ℓ) from a random cross sectioning. Each synthetic microstructure was also cross sectioned across every plane of voxels in the X, Y, and Z directions and the 2D section area a of each exposed particle section was recorded for each plane. This gave the most likely distribution of cross sectional areas expected from a random cross sectioning for each simulation. These values were then converted into circle-equivalent-diameters (d) according to Eq. [2]:

$$d = 2 \times \sqrt{\frac{a}{\pi}} \quad [4]$$

An example of a slice through a synthetic volume with $\sigma = 1$, along with the measurements performed on the slice, can be seen on the right hand side of Figure 1.

The mean chord length ($\bar{\ell}$), mean circle-equivalent-diameter ($\bar{d}$), and the normalized standard deviation of both values ($S(\ell/\bar{\ell})$ and $S(d/\bar{d})$) were calculated and recorded for each of the 100 families of data. Normalized standard deviations were determined by taking a set of data, dividing each value by the mean of that dataset, and then finding the standard deviation of the new set of values. Finally, the multiplication factors for converting between either $\bar{\ell}$ or $\bar{d}$ and D were calculated for every dataset, giving κ_l and κ_d, respectively.

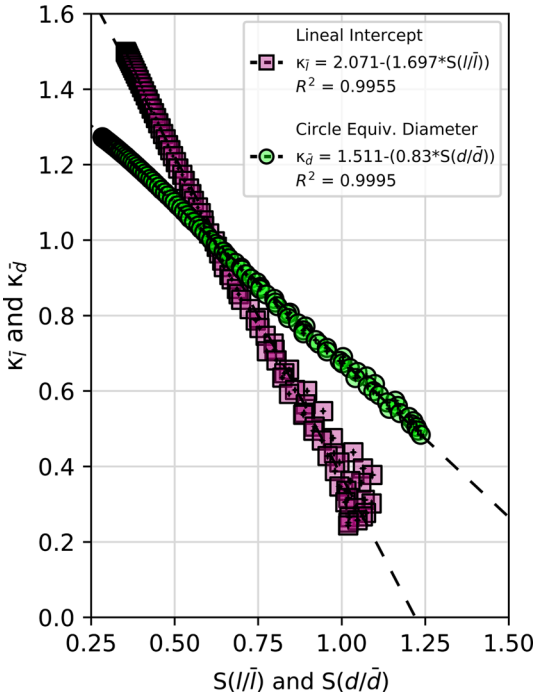

Fig. 3—Plot showing the linear relationship between κ and the normalized standard deviations.

The results of this work are summarized in Figure 3, which plots κ_l against $S(\ell/\bar{\ell})$ and κ_d against $S(d/\bar{d})$. The area of the points at the center of each marker represent the 99.99 pct confidence interval for values of both the normalized standard deviation and the calculated multiplication factor. Since κ_l and κ_d are mean values, their confidence intervals are determined using the standard error of the mean, whereas $S(\ell/\bar{\ell})$ and $S(d/\bar{d})$ are standard deviations, and therefore their confidence intervals are determined using the Chi-squared distribution.

It was found that a strong linear relationship exists between the normalized 2D standard deviations $S(\ell/\bar{\ell})$ and $S(d/\bar{d})$ and both κ_l and κ_d, as shown in Figure 3. Both linear equations of fit have R^2 values of 0.995 or greater, and the data for κ_d in particular shows variation of less than 1 pct for every point tested.

As a further verification of the validity of this experiment, mathematical proofs can be used to show that the distributions of spheres with $\sigma = 0$ should have κ_l and κ_d values of precisely 3/2 and $4/\pi$ respectively. These proofs are provided in the Supplementary Material. The simulated data adheres precisely to these values, whereas the trendlines vary by 3 pct for κ_l and 0.14 pct for κ_d. This comparatively large error in κ_l is due to the fitted data not actually being linear, but in fact very slightly convexly curved away from the origin. The true equation of fit for both equations would have to be an exponential function of some form, and deriving them from geometrical statistics is beyond the scope of this paper. However, the linear equations included here provide much higher accuracy than a constant single-value multiplication factor, while also retaining simplicity in application.

In addition to proving that the correct multiplication factor is a strong function of particle size dispersion, this work also demonstrates experimentally how both κ_l or κ_d can fall below unity for broad distributions. This has been previously suggested and subsequently observed by Louis and Gokhale, and contradicts the logical lower boundaries suggested by Mendelson.[7,8,11,19] Despite this, the observed linearity *must* break down at very broad particle size dispersions because it is physically unreasonable for κ to drop below zero. As such, it would be expected that κ must asymptotically approach zero as $\sigma \to \infty$. Nevertheless, for any cross sectional analysis where $S(\ell/\bar{\ell})$ and $S(d/\bar{d})$ are less than unity, which includes all but the most extreme cases of abnormal grain growth, the provided transfer functions can provide a much higher degree of accuracy than a constant multiplication factor.

It should be emphasized here that the volume of a particle of the mean diameter does not have the same meaning or value as the mean volume. The two values would be calculated as follows:

$$V_{\bar{D}} = \frac{\pi}{6}\left(\frac{\sum_{i=1}^{n} D_i}{n}\right)^3 \quad [5]$$

$$\bar{V} = \frac{\sum_{i=1}^{n} V_i}{n} \quad [6]$$

and for lognormal dispersions of spherical particles, are related by the following equation:

$$\bar{V} = V_{\bar{D}} \times \exp(3\sigma^2) \quad [7]$$

The importance of this distinction has been previously noted for 2D section diameters by Coutinho *et al.*[20] The mean particle volume cannot be calculated using only the mean particle diameter, even if the particle shape is also known. As with Mendelson's equation, some *a priori* knowledge must exist about σ, or a new method for determining σ from the cross sectional data must be established.

While the present work has only been concerned with lognormal distributions of spherical particles, it is likely that linear emperical equations could be fit to dispersions of other particle shapes, such as cubes or tetrakaidecahedra. It is also possible that a similar approach could be applied to any other particel size distribution, such as Rayleigh or gamma distributions. The origin of linearity in these emperical relationships, the prediction of σ from cross sectional data, the expected distribution of the 3D particle volume, and the effect of voxel resolution and sample size on the accuracy of derived values will be explored in subsequent papers.

In summary, it has been found that 3D particle size can be estimated to a high accuracy using a linear function of normalized standard deviation of the 2D measurement. The multiplication factor to convert mean

2D particle size to the mean 3D caliper diameter can be calculated as:

$$\kappa_l = 2.071 - 1.697 \times S\left(\frac{\ell}{\bar{\ell}}\right) \quad [8]$$

$$\kappa_d = 1.511 - 0.83 \times S\left(\frac{d}{\bar{d}}\right) \quad [9]$$

These two transfer functions can be used to accurately estimate the mean 3D diameter of any particle dispersion, assuming the following three conditions are met:

(1) The particles can be assumed to be spherical in shape;
(2) The volumes of the particles can be assumed to follow a lognormal distribution;
(3) The standard deviation of the stereological data is less than its mean.

Under these conditions, Equations [8] and [9] provide a substantial improvement over the previously mentioned scalar multiplier values for κ, which either fail to properly account for the effects of a dispersion of particle sizes or require information that cannot be obtained from cross sectional analysis.

ELECTRONIC SUPPLEMENTARY MATERIAL

The online version of this article (https://doi.org/10.1007/s11661-018-4808-8) contains supplementary material, which is available to authorized users.

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