Can the Average Particle Section Size in a Metallographic Plane Be Larger Than the True Average Particle Size In a Three-Dimensional Microstructure?

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It is shown that the average particle section size observed in a metallographic plane can be larger or smaller than the true average particle size in the three-dimensional (3-D) microstructure. The ratio of the average particle section size in a representative metallographic plane to the true average particle size in the 3-D microstructure is linearly related to the square of the coefficient of variation (CV) of the size distribution function in the 3-D microstructure. For most of the size distributions encountered in material microstructures, the average particle section size in a metallographic plane is expected to be larger than the true average particle size in the 3-D microstructure. Experimental data on hollow spherical carbon particles in a polymer matrix composite are presented to illustrate the theoretical results.

I. INTRODUCTION

MATERIAL microstructures often contain particulates having a range of particle sizes. In such materials, the true average particle size in the three-dimensional (3-D) microstructure is an important parameter that affects the mechanical and physical properties; therefore, its estimation is of interest. Microstructures of opaque materials are usually characterized *via* observations on two-dimensional (2-D) metallographic sections through the 3-D microstructural space. Intersection of a particle with a metallographic sectioning plane can yield a particle section of any size less than (or at the most equal to) its true size, depending on its position and orientation with respect to the sectioning plane.[1,2] For this reason, it is often asserted that the average particle section size in a 2-D metallographic section is *always* lower than the true average particle size in the 3-D microstructure. In other words, the average particle section size in a 2-D metallographic section can be treated as the measure of a *lower bound* on the true average particle size in 3-D microstructure. It is the purpose of this article to prove that this is not correct: the average particle section size in a 2-D metallographic section can be smaller than, equal to, or *larger* than the true average particle size in a 3-D microstructure, depending on the nature of the 3-D particle size distribution. Further, for the majority of the size distributions encountered in practice, the average particle section size in a 2-D metallographic section is expected to be larger than the true average particle size in the 3-D microstructure. These results are useful in understanding and interpreting microstructural observations on metallographic sections. The theoretical derivation is presented in Section II. To keep the analysis simple, it is assumed that the particles can be modeled as spheres; however, the conclusions should be valid for particles of any simple con-

vex shape. Section III of this article discusses the experimental data on the population of hollow spherical carbon particles in a polymer matrix composite.

II. THEORETICAL DEVELOPMENT

Consider a microstructure containing a population of spherical particles of different sizes. Let the variable R represent the radii of the spherical particles. Let N_v be the number of spheres per unit volume of microstructure, and let $F(R)$ be the frequency distribution of the particles, such that $F(R) \cdot dR$ is equal to the fraction of spheres in the size range R to $(R + dR)$. It follows that

$$\int_0^\infty F(R) \cdot dR = 1 \qquad [1]$$

The arithmetic average particle size in a 3-D microstructure, $\langle R \rangle$, can be calculated from the frequency function $F(R)$ as follows:

$$\langle R \rangle = \int_0^\infty R \cdot F(R) \cdot dR \qquad [2]$$

Intersections of spherical particles with a sectioning plane yield circular particle sections. Let the variable r represent the radii of the particle sections observed in the sectioning plane. As mentioned earlier, a sphere of radius R gives circular particle sections of radii r less than or equal to R. Let $\langle r \rangle$ be the arithmetic average size of the circular particle sections observed in the metallographic sectioning plane. Let $f(r)$ be the size frequency of the circular particle sections in the metallographic plane, such that $f(r) \cdot dr$ is equal to the fraction of the particle sections in the size range r to $(r + dr)$. It follows that

$$\langle r \rangle = \int_0^\infty r \cdot f(r) \cdot dr \qquad [3]$$

It will be shown that $\langle r \rangle$ can be smaller than, equal to, or

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larger than $\langle R \rangle$, depending on the nature of the size frequency function $F(R)$. Let $\langle R^2 \rangle$ be the average value of the *square* of the radii of the population of spheres in 3-D microstructure. By definition,

$$\langle R^2 \rangle = \int_0^\infty R^2 \cdot F(R) \cdot dR \qquad [4]$$

Note that $\langle R^2 \rangle$ is not equal to the square of $\langle R \rangle$ unless all the spherical particles have the same radius. Further, $\langle R^2 \rangle$ and $\langle R \rangle$ are independent parameters related to the detailed nature of the size frequency function (Eqs. [2] and [4]). The variance of the population of spheres $\sigma^2(R)$ is defined*

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*Note that the variance can be defined for any distribution function; the distribution need not be normal (Gaussian).

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as follows:

$$\sigma^2(R) = \int_0^\infty [\langle R \rangle - R]^2 \cdot F(R) \cdot dR \qquad [5]$$

Equations [2], [4], and [5] yield

$$\sigma^2(R) = \langle R^2 \rangle - [\langle R \rangle]^2 \qquad [6]$$

Since the variance cannot be negative (Eq. [5]), it follows that for any arbitrary size distribution, $\langle R^2 \rangle$ is always larger than or equal to $[\langle R \rangle]^2$.

Let N_A be the total number of circular particle sections observed per unit area of the metallographic sectioning plane. It can be shown that[1,2]

$$N_A = 2 \cdot \langle R \rangle \cdot N_V \qquad [7]$$

Let S_v be the total surface area of all the spherical particles contained in a unit volume of microstructure The surface area of one spherical particle of radius R is equal to $4\pi R^2$. There are $N_V \cdot F(R) \cdot dR$ particles in the size range R to $(R + dR)$ per unit volume of microstructure. It follows that

$$S_V = 4\pi \cdot N_V \int_0^\infty R^2 \cdot F(R) \cdot dR \qquad [8]$$

Equations [4] and [8] yield

$$S_V = 4\pi \cdot \langle R^2 \rangle \cdot N_V \qquad [9]$$

Let L_A be the total perimeter of all the circular particle sections observed in a unit area of the metallographic sectioning plane. The perimeter of a particle section of radius r is equal to $2\pi r$, and there are $N_A \cdot f(r) \cdot dr$ number of particle sections in the size range r to $(r + dr)$, per unit area of metallographic plane. Thus one can write,

$$L_A = 2\pi \cdot N_A \int_0^\infty r \cdot f(r) \cdot dr \qquad [10]$$

Equations [3] and [10] give the following result:

$$L_A = 2\pi \cdot \langle r \rangle \cdot N_A \qquad [11]$$

The total particle surface area per unit volume, S_v, and L_A are related through the general stereological equation[1,2] given subsequently.

$$S_V = 4 \cdot L_A / \pi \qquad [12]$$

Substituting Eq. [11] for L_A into Eq. [12] leads to the following result:

$$\langle r \rangle = S_V / (8 \cdot N_A) \qquad [13]$$

Substituting Eqs. [7] and [9] into Eq. [13] gives the following expression:

$$\langle r \rangle = \pi \cdot \langle R^2 \rangle / (4 \cdot \langle R \rangle) \qquad [14]$$

Equations [6] and [14] lead to the following result:

$$\frac{\langle r \rangle}{\langle R \rangle} = \frac{\pi}{4} \cdot \left\{ 1 + \frac{\sigma^2(R)}{[\langle R \rangle]^2} \right\} \qquad [15]$$

In statistical terms, the square root of the ratio of the variance $\sigma^2(R)$ together with the square of the true average particle size $[\langle R \rangle]^2$ is called the coefficient of variation of the distribution function, and it is denoted by CV. Thus, Eq. [15] can be written in the following alternate form:

$$\frac{\langle r \rangle}{\langle R \rangle} = \frac{\pi}{4} \cdot \{1 + CV^2\} \qquad [16]$$

where

$$\text{where } CV^2 = \sigma^2(R) / [\langle R \rangle]^2 \qquad [16a]$$

The left-hand side of Eq. [16] is the ratio of the average particle section size in the metallographic section $\langle r \rangle$ and the true average sphere size in the 3-D microstructure $\langle R \rangle$. Whether this ratio is less than one or greater than one is determined by the CV of the sphere size distribution $F(R)$. Inspection of Eq. [16] reveals the following.

(1) If all the spheres are of the same size, then the CV is equal to zero (minimum possible value of CV). In such a case, $[\langle r \rangle / \langle R \rangle]$ is equal to $[\pi/4]$, which is the minimum possible value for this ratio. Thus, for a collection of mono-dispersed spheres, the average particle section size in the metallographic section is smaller than the true sphere size in the 3-D microstructure, as expected.
(2) When a range of sphere sizes exist but the distribution function is such that $0 \leq CV^2 < [(4/\pi) - 1]$, the ratio $\langle r \rangle / \langle R \rangle$ is less than one; hence, the average particle section size $\langle r \rangle$ is smaller than the average sphere size in the 3-D microstructure, $\langle R \rangle$.
(3) For the class of size distributions where CV^2 is equal to $[(4/\pi) - 1]$, the average particle section size in the metallographic section $\langle r \rangle$ is *equal to* the average sphere size $\langle R \rangle$ in the 3-D microstructure.
(4) If CV^2 is larger than $[(4/\pi) - 1]$, then the average particle section size in the metallographic plane $\langle r \rangle$ is greater than the true average sphere radius $\langle R \rangle$ in the 3-D microstructure. This precisely identifies the size distributions for which $\langle r \rangle$ is expected to be larger than $\langle R \rangle$. The CV is the measure of the spread of the particle sizes around the mean $\langle R \rangle$. The wider size distributions have a higher value of CV. The majority of the size distributions encountered in the material microstructures are not narrow; hence, in the majority of cases, the average particle section size $\langle r \rangle$ is expected to be larger than the true average particle size $\langle R \rangle$ in the 3-D microstructure.

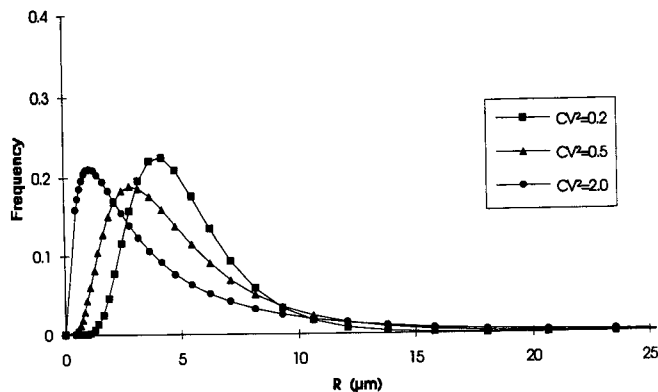The conclusion that the average particle section size can

Fig. 1—Log-normal frequency distribution function for different CV^2 and the same mean radius ($\langle R \rangle$ = 5.3 μm).
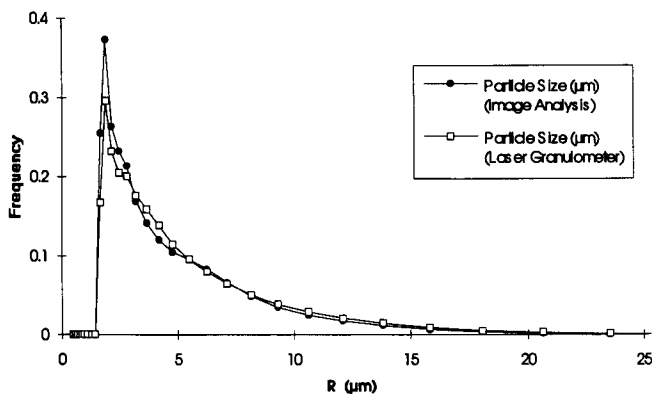

Fig. 2—3-D frequency distribution function of the carbon particles ($\langle R \rangle$ = 5.3 μm; CV^2 = 0.58).

Fig. 3—Microstructure of composite specimen observed at magnification 200 times.

be larger than the true average size in the 3-D microstructure appears to be counter-intuitive. One may argue that if the circular particle sections from any sphere cannot be larger than the sphere itself, how can the average section size be larger than the average sphere size? This paradox can be resolved by recognizing that the probability of a sphere intersecting the sectioning plane is directly proportional to its size.[1,2] Thus, the number of particle sections from the spheres of given size is directly proportional to their number density in the 3-D microstructure and to their size. In other words, the sampling by metallographic planes is biased toward larger particles: the planes sample (intersect) more larger spheres than they do smaller ones, for the same number densities in the 3-D microstructure. In the metallographic plane, there are more particle sections originating from larger spheres (than from the smaller ones), and on the average, the sizes of these particle sections are also larger; hence, they contribute more to the average value of the particle section size as compared to relatively smaller sections from the smaller particles. This can lead to a higher average particle section size as compared to the true average 3-D particle size. The coefficient of variation of the size distribution function governs the relative magnitude of the average particle section size as compared to the true average particle size in the 3-D microstructure. If the size distribution is narrow (low CV), then the average particle section size is less than the true average size. For wider size distributions (high CV), as usually observed in material microstructures, the average particle section size is
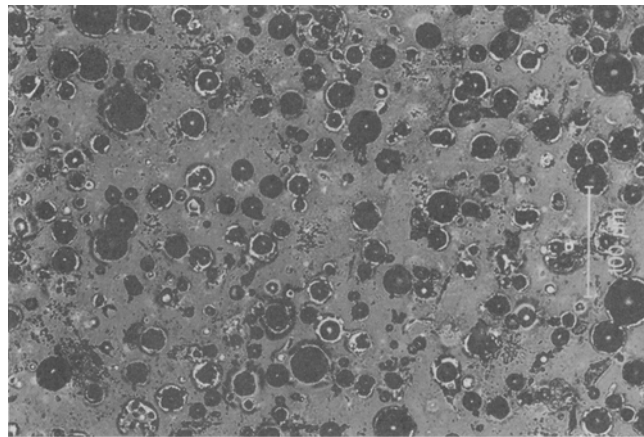
larger than the true 3-D average particle size. Figure 1 shows three log-normal size distributions having the same value of average size but different values of CV. For the distribution, with CV^2 equal to 0.2, the average particle section size in the metallographic plane is expected to be smaller than the true average size in the 3-D microstructure ($\langle r \rangle/\langle R \rangle$ = 0.94); however, for the distribution having CV^2 equal to 2.0, the average particle section size in the metallographic plane is more than two times the true average size in the 3-D microstructure ($\langle r \rangle/\langle R \rangle$ = 2.35).

III. EXPERIMENTAL DATA

To support the theoretical calculations, experimental measurements were performed on a microstructure having known 3-D particle size distribution. The measurements were performed on a polymer matrix composite containing hollow spherical carbon particles. Figure 2 shows the size frequency function of the as-received carbon particles obtained from laser granulometry. The true average carbon particle radius $\langle R \rangle$ is 5.3 μm. Substituting the frequency distribution data of Figure 2 into Eq. [5] yields the value of $\sigma^2(R)$ equal to 16.4 μm^2. Substituting these data into Eq. [15] yields $\langle r \rangle/\langle R \rangle$ equal to 1.24. Thus, if this carbon powder is dispersed in a 3-D microstructure, and a random metallographic sectioning plane is observed through that 3-D microstructural space, then the average particle section size in the metallographic plane should be about 24 pct larger than the true average radius of the carbon particles in the 3-D microstructure.

The polymeric resin (araldite) and hardener (supplied by Ciba Geygi, Germany) and the hollow spherical carbon particles (supplied by Carbospheres, Inc., USA) were mixed in the appropriate proportions. The mixture was heated in vacuum to de-gas the material and to decrease the viscosity of the polymer. It takes about 10 hours for this polymer to harden completely; hence, to minimize the particle segregation due to gravitational effect, the molds were slowly rotated during the hardening of the polymer.

The specimens were cut, mounted, and polished on a series of SiC polishing papers followed by polishing on diamond cloths using automatic polishing machines. Figure 3 shows a typical microstructure of this composite observed in a metallographic plane. In Figure 3, the dark regions are
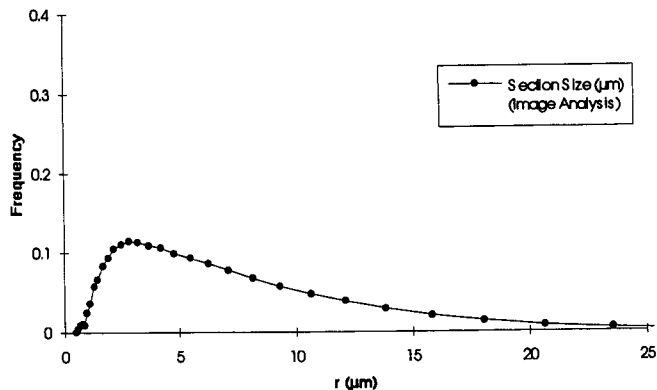
Fig. 4—Experimentally measured frequency distribution function of the carbon particle sections observed in 2-D metallographic section ($\langle r \rangle = 6.7$ μm).

the "hollows" or cavities created by the intersections of hollow carbon particles with the sectioning plane. The bright regions are due to near tangential intersections between the particles and the sectioning plane.

The particle section size measurements were performed on a VIDAS (Zeiss) image analyzer at magnification 500 times. The measurements were performed on 455 fields of view at magnification 500 times. The particle section radii of about 12,000 particles were measured to obtain a statistically reliable estimate of the average section size. Figure 4 shows the frequency distribution function of the particle section radii calculated from these measurements. A simple calculation (Equation [3]) yields the average particle section radius $\langle r \rangle$ equal to 6.7 μm. Thus, the experimental value of the average particle section size in the metallographic section is larger than the true average sphere size $\langle R \rangle$ (equal to 5.3 μm), as predicted from Eq. [15] and [16]. Further, the experimental value of the ratio $\langle r \rangle/\langle R \rangle$ is equal to 1.26,

which is in excellent agreement with the value 1.24 predicted by Eq. [15].

IV. CONCLUSIONS

The arithmetic average particle size in a representative 2-D metallographic section can be less than, equal to, or greater than the true arithmetic average particle size in a 3-D microstructure. Thus, the average particle section size cannot be regarded as a lower bound on the true average particle size in the 3-D microstructure. The ratio of the average particle section size to the true average particle size in the 3-D microstructure is linearly related to the square of the coefficient of variation of the particle size frequency function in the 3-D microstructure. For wider size distribution (CV^2, $> (4/\pi) - 1$), the average particle section size is larger than the true average particle size in the 3-D microstructure.

ACKNOWLEDGMENTS

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