Quantifying the dispersion of mixture microstructures

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Key words. Composite, dispersion, fibre, inclusion, microstructure, mixture, multiphase, particle, quantification, section.

Summary
A general method to quantify the inclusion dispersion of mixture microstructures has been developed. The dispersion quantity, $D$, is defined as the probability of inclusion particle free-path spacing falling into a certain range of the mean spacing $\mu$, according to the particle spacing data frequency distribution. Two quantities, $D_{0.1}$ and $D_{0.2}$, are proposed, which are the probabilities of the particle free-path spacing falling into the ranges of $\mu \pm 0.1 \mu$ and $\mu \pm 0.2 \mu$, respectively. Both normal and lognormal distributions are discussed, and in both cases, the quantities $D_{0.1}$ and $D_{0.2}$ are specified as monotonous increasing functions of $\mu/\sigma$, where $\mu$ and $\sigma$ are the mean particle free-path spacing and standard deviation, respectively. Examples of composite are presented to quantify the dispersion of foreign reinforcements based on the proposed method.

Introduction
Quantitative measurements of material microstructures are beneficial for a better understanding of correlations between material processing-microstructure-property characteristics (Higginson & Sellars, 2003). Mixture microstructures, such as microstructures with multiphases, precipitation in the matrix, chemical reactants and products and foreign reinforcements in composites are commonly studied (Higginson & Sellars, 2003; Chung, 2005). Developments involving image-processing techniques have made it possible to quantify the particle dimensions (diameter, perimeter and areas), mass centre locations ($x$ and $y$ positions) as well as the fractional concentrations based on these measurements. These processing techniques are basically determined by the threshold of a certain image grey level to select the particles (Russ, 1991).

To study the dispersion of the mixture microstructure, the ‘dispersion value’ is an important characteristic. Generally, a good dispersion of the inclusions is favourable to the materials properties, whereas large aggregation with poor dispersion reduces or compromises the properties. Moore (1970, 1972a, b) pointed out that the dispersion of the inclusion particles could be characterized using the following three characteristics:

1. Variability index (VI): The variability index is defined as

\[
VI = \frac{\sigma_{\text{obs}}}{\sigma_p},
\]

where $\sigma_{\text{obs}}$ is the standard deviation of the material, and $\sigma_p$ is the Poisson standard deviation from a uniformly random mixture containing the same number of particles.

2. Anisotropy ratios: The anisotropy is specified by the ratios of mean intercept size ($l_1/l_2$) (on the particles or the matrix) as measured along these two different principal directions.

3. Degree of patterness (slice index): This characteristic of slice index (SI) is used to indicate the severity of laminated or fibrous nature of the microstructure. Lower SI corresponds to a better dispersion. It approaches zero for a uniform distribution of the particles.

The above three characteristics only reflect the dispersion grade in different ways, but they do not provide quantitative definitions of the dispersion. Recently, Tscheschel et al. (2005) studied the dispersion of high-level silica-filled rubber using a variance $\sigma^2$ and a median range $h_{0.5}$. The former one, $\sigma^2$, is a measure of the grade of the micro-dispersion of filler particles (smaller values of $\sigma^2$ imply higher grade of dispersion); the later one, $h_{0.5}$, is related to the size of the object (smaller values of $h_{0.5}$ correspond to smaller objects and thus a higher grade of dispersion).

In this publication, a quantitative definition of the inclusion dispersion is given. A fundamental principle of stereology
says that the volume fraction $V_V$ of a certain phase is measured from the point counting ($P_P$), area analysis ($A_A$) and lineal analysis ($L_L$)-related measurements (Moore, 1970; Underwood, 1972; Weibel, 1979; Stoyan et al., 1995; Howard & Reed, 1998; Higginson & Sellars, 2003), that is,

$$V_V = P_P = A_A = L_L,$$

where $P_P$ is the fraction of points falling in the interested phase in a random array of a total number of points, and $A_A$ and $L_L$ are the corresponding area and length fractions, respectively (for details, see Higginson & Sellars, 2003). This equation serves as the fundamental relationship in quantitative measurements.

**Quantification method**

The dispersion quantity is related to the free-path spacing between the inclusion particle surfaces (rather than the particle centre spacing), regardless of their shape or size. The more uniform the spacing between these inclusion surfaces, the higher the dispersion grade will be. If all of the inclusion particles are distributed at an equal free-path distance, the dispersion is defined as 100%.

Figure 1(a) depicts schematically a solid sample with various irregular inclusions (in dark colour) in a matrix. A section is made, as shown in Fig. 1(b), where these inclusions are seen in a 2-dimensional (2D) plane view. Because the inclusion particles have very irregular shape and are in reality three-dimensional (3D), it is impossible to calculate the spacing between them. However, one may use a random line to intercept these particles to obtain the free-path spacing measurements between these inclusions, $x_1, x_2, \ldots, x_i, \ldots, x_N$, as shown in Fig. 1(b). If two particles attach together on this line, their spacing is counted as $x = 0$. These measurements are then divided into groups with an interval of $\Delta x$, and the frequency density $f_i$ of each group is thus

$$f_i = \frac{n_i}{N \Delta x},$$

where $n_i$ is the number of data falling into this group, and $N$ is the total number of the measurements. Therefore, a histogram can be constructed to exhibit the spacing data distribution, as shown in Fig. 1(c). If the interval $\Delta x$ is small enough (usually divided into 10–100 classes) and the number of measurements $N$ is sufficiently large (usually over 100), then one can obtain a smooth curve of the frequency function, that is, the probability density function, $f(x)$, as shown in Fig. 1(d). It has a mean value of $\mu$ and a standard deviation of $\sigma$. It is well known that the standard deviation $\sigma$ is a measurement of the data dispersion, that is, smaller $\sigma$ indicates more data closer to the mean $\mu$. However, $\sigma$ does not quantify the dispersion ‘value’. It can be noted that the area below the curve is the probability of a certain range of the spacing data. For example, the probability of the spacing data falling into the range of $a - b$ is $\int_a^b f(x) dx$.

Certainly, $\int_0^\infty f(x) dx = 1$. The probability of a certain range of spacing around the mean $\mu$ can be defined as the dispersion $D$. In the range of $\mu \pm 0.1 \mu$, the dispersion $D_{0.1}$ is

$$D_{0.1} = \int_{0.9\mu}^{1.1\mu} f(x) dx.$$  (3)

Similarly, in the range of $\mu \pm 0.2 \mu$, $D_{0.2}$ is

$$D_{0.2} = \int_{0.8\mu}^{1.2\mu} f(x) dx.$$  (4)

The dispersion quantities of $D_{0.1}$ and $D_{0.2}$ are the shadowed areas below the $f(x)$ curve as shown in Fig. 1(d), corresponding to the possibilities of the spacing data falling into the range of $\mu \pm 0.1 \mu$ and $\mu \pm 0.2 \mu$, respectively. Higher $D$ values indicate more spacing data closing to the mean $\mu$, and thus more uniform distribution of the inclusion particles. In this way, one may define the dispersion in other data ranges around the mean spacing $\mu$, such as $D_{0.3}, D_{0.4}, D_{0.5}$, etc. It is evident that for smaller $\sigma$, the probability $f(x)$ around $\mu$ is higher, and thus, $D_{0.1}$ and $D_{0.2}$ are also higher for the smaller $\sigma$, although the Eqs (3) and (4) do not contain $\sigma$ directly.

The quantity of the dispersion $D$ is related to the accurate measurement of the spacing $x$. If the inclusions have regular spherical shape and their locations $(x, y, z)$ are known, the spacing between particles 1 at centre position $(x_1, y_1, z_1)$ with radius $r_1$, and particle 2 at $(x_2, y_2, z_2)$ with radius $r_2$ is

$$x = \sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2 + (z_2 - z_1)^2} - r_2 - r_1.$$  (5)

For some soft samples, such as some biological and polymeric specimens, the dissector method can be applied to assist the spacing measurements (Gundersen, 1986; Howard & Reed, 1998), for example, a physical dissector using two physically separated sections with known thickness as prepared by microtome (two parallel sections in Fig. 1(a) with known thickness), or an optical dissector using two images obtained at two different focal planes by a confocal microscope (Howard & Reed, 1998; Gundersen, 2001). However, these approaches may not be applicable to most hard materials, such as metal- and ceramic-matrix composites (Luo et al., 2001; Chung, 2005) and some polymer-matrix composites, depending on their hardness and transparency (Koo et al., 2003). Sectioning these hard materials, such as by the ion milling method as described later, loses the thickness control during the preparation process. In this case, isotropic uniform random sampling should be used to make the microstructure representative (Howard & Reed, 1998). In fact, under the mild condition that the inclusions are isotropic in the matrix, the mean free path in 2D is an unbiased estimator of the mean free path in 3D (Underwood, 1970, pp. 82–83).

It should be pointed out that in this publication, the spacing is measured directly. If one calculates the fraction of $x_i/l_i$,
Fig. 1. (a) A solid specimen contains various irregular inclusions (dark colour) in the matrix; (b) a section from the solid specimen, where free-path spacing of the inclusions are measured; (c) construction of histogram; (d) definition of dispersion $D_{0.1}$ and $D_{0.2}$ quantities from the frequency function $f(x)$.

where $l_j$ is the length of the random $j$th line in Fig. 1(b), the fraction of the matrix space can be obtained according to Eq. (1). In fact, Moore (1970, 1972a, b) used the ratio of the mean spacing along two different directions to specify the anisotropy character, whereas here we define the dispersion from the data scattering, which can be measured along any direction, that is, the dispersion can be quantified in all directions. Therefore, one may use the dispersion $D$ along different directions to specify the material anisotropy.

Consider the situations of particle distributions as discussed by Russ (1991). If the particles are regularly distributed, as shown in Fig. 2(a), the frequency curve only shows one peak, as schematically shown in Fig. 2(b), with a mean $\mu$ and a standard deviation $\sigma$. A normal and lognormal distribution curves are superimposed, with the same mean $\mu$ and standard deviation $\sigma$, as shown in thin line in Fig. 2(b). The normal distribution is defined as

$$f(x) = \frac{1}{\sigma \sqrt{2\pi}} e^{-\frac{1}{2} \left( \frac{x - \mu}{\sigma} \right)^2}, \quad (x > 0)$$

and

$$0 \quad (x \leq 0)$$

and the lognormal distribution is defined as

$$f(x) = \begin{cases} 
\frac{1}{x n \sqrt{2\pi}} e^{-\frac{1}{2} \left( \ln \frac{x}{m} \right)^2} \quad (x > 0) \\
0 \quad (x \leq 0)
\end{cases}$$

where

$$m = \ln \frac{\mu^2}{\sqrt{\mu^4 + \sigma^2}}$$

and

$$n = \sqrt{\frac{\ln \mu^2 + \sigma^2}{\mu^2}}.$$

One may choose the normal or lognormal distribution to fit the real frequency distribution depending on which one gives the best fit. However, when clusters are present, as shown in Fig. 2(c), the frequency distribution curve exhibits two peaks of bimodal distribution (or more peaks depending on the cluster
distance distribution), as shown in Fig. 2(d). The first one with \((\mu_1, \sigma_1)\) reflects the distribution inside the clusters with shorter spacing, and the second one with \((\mu_2, \sigma_2)\), the distribution of these clusters with larger spacing. If one uses these two peaks to quantify the dispersions \(D_1\) and \(D_2\), and then take the average of them for the sample dispersion \(D\), this dispersion maybe overestimated, because the \(D_1\) only represents the dispersion within the clusters and \(D_2\) the dispersion associated with the clusters (both of them may be good ones). However, this problem may be solved by superimposing the normal distribution curve and the lognormal distribution curves with the same mean \(\mu\) and standard deviation \(\sigma\) of the entire data (mixture of the two peaks), as shown in Fig. 2(d). Such distribution curves reflect the scattering of the entire data, and thus one may use them to quantify the dispersion in this case. It is seen that when clusters are present, the dispersion grade is very decreased as expected.

The treatments by normal and lognormal distributions are presented in the appendix separately to deduce general dispersion formulas, which show that the dispersions \(D_{0,1}\) and \(D_{0,2}\) are only monotonous increasing functions of \(\mu/\sigma\), as shown in Fig. 3. Once the spacing data \(x_1, x_2, \ldots, x_i, \ldots, x_N\) are measured, one may use the data sample mean \(\bar{x} = \frac{\sum x_i}{N}\) and data sample standard deviation \(s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N-1}}\) to estimate the bulk material population mean \(\mu\) and population standard deviation \(\sigma\), respectively, if the number of measurements \(N\) is very large, that is, \(\hat{\mu} = \bar{x}\) and \(\hat{\sigma} = s\), where \(\hat{\mu}\) and \(\hat{\sigma}\) are the estimators of \(\mu\) and \(\sigma\), respectively. Therefore, \(D\) can be obtained accurately from (12) and (13) for the normal, and (17) and (18) for the lognormal distributions, respectively, or estimated fairly rapidly from (14) and (15) for the normal, and (19) and (20) for the lognormal distributions, respectively.

**Examples**

Two examples of the microstructure of carbon nanofibre (CNF)-reinforced polymer matrix composite (Koo et al., 2003) obtained by transmission electron microscope (TEM) are presented in Figs. 4 and 5. The samples were prepared by ion milling (for hard materials), or ultra-microtome (for soft materials) using a diamond knife at 5–6° cutting angle. The selection of the sample preparation method depends on the sample hardness. The ion mill sample preparation process were as follows: the bulk sample was first cut into slices about 1-mm thick, then these slices were mechanically grounded and polished until 50–100 \(\mu\)m and then punched into small disks with 3-mm diameter. These disks were further thinned by dimple grinding to achieve the centre thickness of 10–30 \(\mu\)m, and finally were thinned by ion beam until perforation. The image processing was performed using an IMAGEx program.

The sample prepared by ion milling has non-uniform thickness, as shown in Fig. 4(a). The sample by ultra-microtome has uniform thickness but cutting artefacts are introduced, as indicated by arrowheads in Fig. 5(a). After image
Fig. 3. Calculated dispersion $D_{0.1}$ and $D_{0.2}$ as functions of $\mu/\sigma$ for normal and lognormal distributions. The lognormal distribution shows slight higher values over the normal distribution.

Fig. 4. (a) TEM image of CNF composite (sample prepared by ion milling); (b) processed image by removing the background; (c) spacing frequency distribution with lognormal distribution fitting (smooth curve). The arrow indicates the mean position.

Fig. 5. (a) TEM image of CNF composite (sample prepared by ultra-microtome); (b) processed image by removing the background; (c) spacing frequency distribution with lognormal distribution fitting (smooth curve). The arrow indicates the mean position.
processing to show the reinforcement edges, these sample preparation artefacts by both methods are removed, as shown in Figs 4(b) and 5(b), respectively. From such images, the spacing data between these CNF are measured from $20 \times 20$ equal distance horizontal and vertical grid lines, respectively. The histograms are shown in Figs 4(c) and 5(c), respectively, and the statistical data of the measurements are given in Table 1.

From the histogram in Figs 4(c) and 5(c), it is found that the lognormal distribution gives the best fit, as shown by smooth curves that are superimposed. In Fig. 4, $\bar{x}/s = 1.2171$, and in Fig. 5, $\bar{x}/s = 1.0296$. Using $\bar{x}$ and $s$ to estimate $\mu$ and $\sigma$, respectively, therefore, $D_{0.1}$ and $D_{0.2}$ for the lognormal distribution are calculated out by the Eqs. (17) and (18) for accurate results, and by (19) and (20) for approximations, as shown in Table 2. Basically, the results by these two different methods are reasonably close to each other. Visual inspection shows that Fig. 4 has better dispersion over Fig. 5, because Fig. 5 contains some unoccupied areas in the centre. The quantified results of $D$, as listed in Table 2, reveal that the dispersion of Fig. 4 is higher than Fig. 5.

### Concluding remarks

1. An universal method to quantify the dispersion $D$ of mixture microstructures was developed. Two quantities, $D_{0.1}$ and $D_{0.2}$, are proposed, which are the probabilities of the particle spacing falling into the range of $\mu \pm 0.1 \mu$ and $\mu \pm 0.2 \mu$, respectively, according to the spacing data frequency distribution.

2. Two cases of normal and lognormal distributions of the spacing data were considered, and the dispersion quantities were formularized in Eqs. (12)–(15) and (17)–(20), respectively. In both cases, it was demonstrated that the dispersion $D$ is only a monotonous increasing function of $\mu/\sigma$.

3. The challenge to quantify the dispersion $D$ is the accurate measurement of the free path distance between the inclusions in the matrix. Once the spacing data are measured and the mean $\bar{x}$ and standard deviation $s$ are obtained, depending on data distribution, the dispersion quantity $D$ can be simply calculated from these equations by using $\bar{x}$ and $s$ to estimate the material population mean $\mu$ and population standard deviation $\sigma$, respectively, if the number of measurements $N$ is sufficiently large. The selection of normal or lognormal distribution depends on which one fits the spacing data distribution, but each of them gives consistent quantifications. Usually the measurements obey the lognormal distribution, as demonstrated in the examples. Even in the case of the presence of clusters, as depicted in Fig. 2(c), one may use the lognormal (or normal) distribution to compare the dispersions quantitatively.

4. The method proposed in this publication can be applied for either irregular inclusion particles, as shown in Fig. 1(a) and (b), or regular shape particles, as shown in Figs 2(a) and (c). In the later case, the particle spacing can be calculated from their centre coordinates using Eq. (5), and thus the dispersion can be obtained from their spacing data scattering.

### Acknowledgements

The authors are grateful to a reviewer for critical comments, and to Dr. Stan Vitha, Microscopy and Imaging Center, Texas A&M University, for some valuable discussions. This work was supported by the Air Force Office of Scientific Research (AFOSR), Arlington, Virginia.

### References


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**Table 1.** Statistics of figures 4 and 5.

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**Table 2.** Quantified dispersions of Figs 4 and 5.

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Appendix

Dispersion for the normal distribution case

Let us start with the simple case of a normal distribution, with a mean of \( \mu \) and standard deviation of \( \sigma \). This normal distribution can be converted to a standard normal distribution \( (\mu = 0, \sigma = 1) \) using a substitution of

\[
 z = \frac{x - \mu}{\sigma},
\]

and thus we have \( dx = \sigma dz \) (Dougherty, 1990). Hence, the probability (dispersion) between \( a-b \) is calculated as

\[
 D = \int_a^b \frac{1}{\sqrt{2\pi}} e^{-\frac{1}{2} \left( \frac{z - \mu}{\sigma} \right)^2} dz
\]

as

\[
 D = \int_a^b \frac{1}{\sqrt{2\pi}} e^{-\frac{1}{2} \left( \frac{x - \mu}{\sigma} \right)^2} dx
\]

Here \( \Phi(z) \) is the probability from \( -\infty \) to \( z \), \( \Phi(z) = \int_{-\infty}^{z} e^{-\frac{1}{2} t^2} dt \), whose value can be found in statistical references or a number of online calculators. Therefore, for \( D \), \( a = 0.9 \mu \), \( b = 1.1 \mu \), hence

\[
 D_{0.1norm} = \Phi(0.1\mu/\sigma) - \Phi(-0.1\mu/\sigma).
\]

Similarly we have

\[
 D_{0.2norm} = \Phi(0.2\mu/\sigma) - \Phi(-0.2\mu/\sigma).
\]

From the above (Eqs (8) and (9)), it is clear that \( D_{0.1} \) and \( D_{0.2} \) are only functions of \( \mu/\sigma \). Therefore, \( D_{0.1norm} \) and \( D_{0.2norm} \) are plotted as functions of \( \mu/\sigma \) from 0 to 10, as shown in Fig. 3. It can be seen that the dispersion \( D \) is a monotonous increasing function of \( \mu/\sigma \), that is, \( D \) increases with increasing \( \mu \), which is reasonable because for larger \( \mu \) the sample looks more uniform. This case is similar to the study by Tschesche et al. (2005) who showed that smaller values of \( k_{0.5} \) indicate smaller objects and thus higher dispersion. \( D \) also increases with decreasing \( \sigma \), because smaller \( \sigma \) indicates less spacing data scattering.

For convenience, we give here the regression results of \( D_{0.1norm} \) and \( D_{0.2norm} \) by curve fitting as follows:

\[
 D_{0.1norm} = 6.8843 \times 10^{-5} + 7.964 \times 10^{-2}(\mu/\sigma)
\]

\[
 + 1.043 \times 10^{-4}(\mu/\sigma)^2 - 1.6286 \times 10^{-4}(\mu/\sigma)^3
\]

\[
 + 3.8639 \times 10^{-6}(\mu/\sigma)^4
\]

(14)

and

\[
 D_{0.2norm} = -4.0117 \times 10^{-4} + 0.16056(\mu/\sigma)
\]

\[
 - 2.8118 \times 10^{-4}(\mu/\sigma)^2 - 1.1826 \times 10^{-3}(\mu/\sigma)^3
\]

\[
 + 5.6084 \times 10^{-5}(\mu/\sigma)^4
\]

(15)

Dispersion for the lognormal distribution case

Lognormal distribution should also be considered, because most of the measurements are found to obey the lognormal distribution instead of the normal distribution (Carpenter et al., 1998; Veleycky & Popisil, 2000), as will be demonstrated in the following examples. In the case of lognormal distribution, as defined in (7), let \( z = \ln x \), then we have \( dz = dx/x \). Hence, the probability (dispersion) between \( a-b \) is calculated as

\[
 D = \int_a^b \frac{1}{x\sqrt{2\pi}} e^{-\frac{1}{2} \left( \ln(x/a) - \mu \right)^2} dx
\]

\[
 D = \int_a^b \frac{1}{x\sqrt{2\pi}} e^{-\frac{1}{2} \left( \ln(x/a) - \mu \right)^2} dx
\]
\[
\int_{\ln a}^{\ln b} \frac{1}{n\sqrt{2\pi}} e^{-\frac{1}{2} \left( \frac{\ln z - \ln a}{\sqrt{\ln^2 + \sigma^2}} \right)^2} dz = \Phi \left( \frac{\ln b - m}{n} \right) - \Phi \left( \frac{\ln a - m}{n} \right). \quad (16)
\]

Therefore for \(D_{0.1}\), taking \(a = 0.9\ \mu\) and \(b = 1.1\ \mu\) and substituting \(m\) and \(n\) using (8) and (9) yield

\[
D_{0.1\log} = \Phi \left( \frac{\ln 1.1 - \ln \frac{\mu^2}{\sqrt{\mu^2 + \sigma^2}}}{\sqrt{\ln^2 + \sigma^2}} \right)
- \Phi \left( \frac{\ln 0.9 - \ln \frac{\mu^2}{\sqrt{\mu^2 + \sigma^2}}}{\sqrt{\ln^2 + \sigma^2}} \right)
= \Phi \left[ \frac{\ln 1.1}{\sqrt{1 + (\mu/\sigma)^2}} \right]
- \Phi \left[ \frac{\ln 0.9}{\sqrt{1 + (\mu/\sigma)^2}} \right]. \quad (17)
\]

Similarly,

\[
D_{0.2\log} = \Phi \left( \frac{\ln 1.2 - \ln \frac{\mu^2}{\sqrt{\mu^2 + \sigma^2}}}{\sqrt{\ln^2 + \sigma^2}} \right)
- \Phi \left( \frac{\ln 1.0 - \ln \frac{\mu^2}{\sqrt{\mu^2 + \sigma^2}}}{\sqrt{\ln^2 + \sigma^2}} \right)
= \Phi \left[ \frac{\ln 1.2}{\sqrt{1 + (\mu/\sigma)^2}} \right]
- \Phi \left[ \frac{\ln 1.0}{\sqrt{1 + (\mu/\sigma)^2}} \right]. \quad (18)
\]

As for the case of normal distribution, \(D_{0.1\log}\) and \(D_{0.2\log}\) are only functions of \(\mu/\sigma\). For a given value of \(\mu/\sigma\), \(D_{0.1\log}\) and \(D_{0.2\log}\) can be calculated according to Eqs (17) and (18). These relations are also plotted in Fig. 3. It is seen that \(D_{0.1\log}\) and \(D_{0.2\log}\) are slightly higher than \(D_{0.1\norm}\) and \(D_{0.2\norm}\), respectively, in the range computed. \(D_{0.1\log}\) and \(D_{0.2\log}\) are regressed as

\[
D_{0.1\log} = 1.1539 \times 10^{-2} + 7.5933 \times 10^{-2} (\mu/\sigma)
+ 6.6838 \times 10^{-4} (\mu/\sigma)^2 - 1.9169 \times 10^{-4} (\mu/\sigma)^3
+ 3.9201 \times 10^{-6} (\mu/\sigma)^4 \quad (19)
\]

and

\[
D_{0.2\log} = 2.266 \times 10^{-2} + 0.15629 (\mu/\sigma)
+ 4.442 \times 10^{-4} (\mu/\sigma)^2 - 1.2738 \times 10^{-4} (\mu/\sigma)^3
+ 5.9978 \times 10^{-5} (\mu/\sigma)^4. \quad (20)
\]