



Stereology

27-750

Texture, Microstructure & Anisotropy

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Last revised: 21st April 2014

Outline

- Objectives
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 - Quantities,
 - definitions
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 - Derivable
 - Problems that use Stereology, Topology
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 - Surface area per unit volume
- Facet areas
 - Oriented objects
 - Particle spacings
 - Mean Free Path
 - Nearest Neighbor Distance
 - Zener Pinning
 - Grain Size
 - Sections through objects
 - Size Distributions

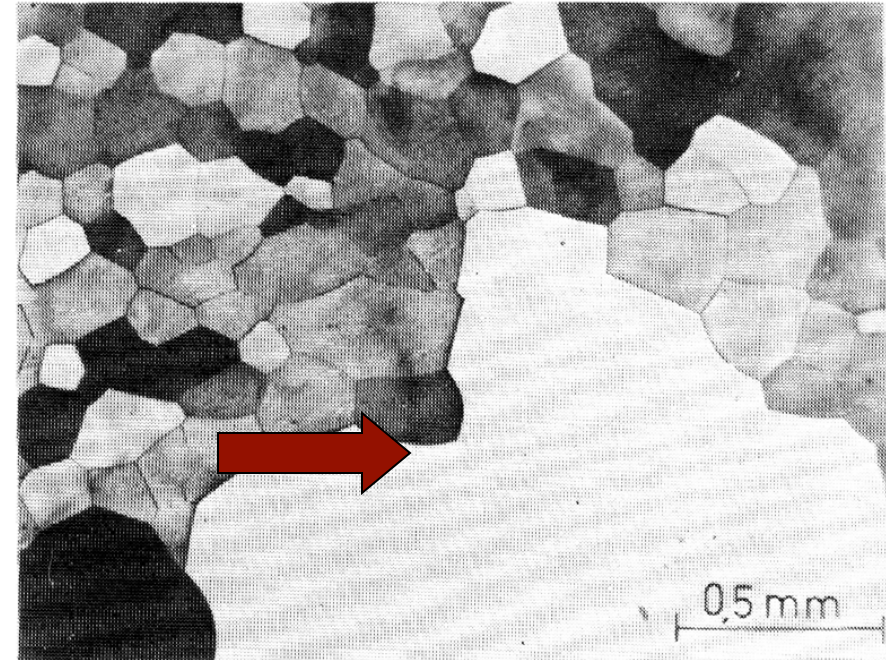
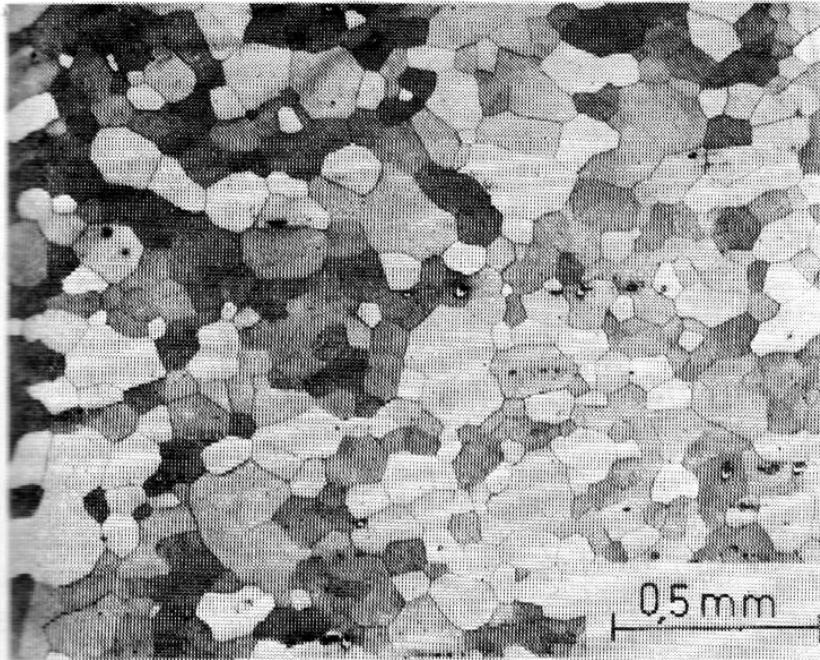
Objectives

- To instruct in methods of *measuring* characteristics of microstructure: grain size, shape, orientation; phase structure; grain boundary length, curvature etc.
- To describe methods of obtaining 3D information from 2D planar cross-sections: *stereology*.
- To illustrate the principles used in extracting grain boundary properties (e.g. energy) from geometry+crystallography of grain boundaries: *microstructural analysis*.

Objectives, contd.

- [Stereology] To show how to obtain useful microstructural quantities from plane sections through microstructures.
- [Image Analysis] To show how one can analyze images to obtain data required for stereological analysis.
- [Property Measurement] To illustrate the value of stereological methods for obtaining relative interfacial energies from measurements of relative frequency of faceted particles.
- Note that true 3D data is available from serial sectioning, tomography, and 3D microscopy (using diffraction). All these methods are time consuming and therefore it is always useful to be able to infer 3D information from standard 2D sections.

Motivation: *grain size*



- Secondary recrystallization in Fe-3Si at 1100°C
- How can we obtain the average grain size (as, say, the caliper diameter in 3D) from measurements from the micrograph?
- Grain size becomes heterogeneous, anisotropic: how to measure?

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Motivation: *precipitate sizes, frequency, shape, alignment*

- Gamma-prime precipitates in Al-4a/oAg.
- Precipitates aligned on $\{111\}$ planes, elongated: how can we characterize the *distribution* of directions, lengths?
- Given crystal directions, can we extract the habit plane?

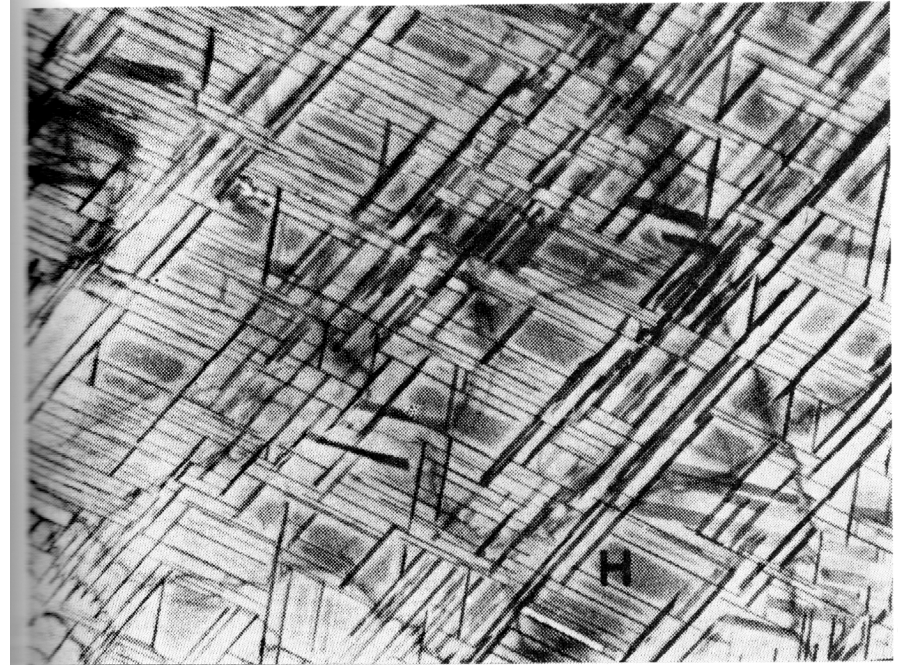


Fig. 3.42 Electron micrograph showing the Widmanstätten morphology of γ' precipitates in an Al-4 atomic % Ag alloy. GP zones can be seen between the γ' , e.g. at H ($\times 7000$). (R.B. Nicholson and J. Nutting, *Acta Metallurgica*, 9 (1961) 332.)

[Porter & Easterling]

Stereology: References

- These slides are based on: *Quantitative Stereology*, E.E. Underwood, Addison-Wesley, 1970. - equation numbers given where appropriate.
- *Practical Stereology*, John Russ, Plenum (1986, IDBN 0-306-42460-6).
- A very useful, open source software package for image analysis: *ImageJ*, <http://rsb.info.nih.gov/ij/>.
- A more comprehensive commercial image analysis software is *FoveaPro*, <http://www.reindeergraphics.com>.
- Also useful, and more rigorous: M.G. Kendall & P.A.P. Moran, *Geometrical Probability*, Griffin (1963).
- More modern textbook, more mathematical in approach: *Statistical Analysis of Microstructures in Materials Science*, J. Ohser and F. Mücklich, Wiley, (2000, ISBN 0-471-97486-2).
- *Stereometric Metallography*, S.A. Saltykov, Moscow: Metallurgizdat, 1958.
- Many practical (biological) examples of stereological measurement can be found in *Unbiased Stereology*, C.V. Howard & M.G. Reed, Springer (1998, ISBN 0-387-91516-8).
- *Random Heterogeneous Materials: Microstructure and Macroscopic Properties*, S. Torquato, Springer Verlag (2001, ISBN 0-387-95167-9).
- D. Sahagian and A. Proussevitch (1998) 3D particle size distributions from 2D observations: Stereology for natural applications, *J Volcanol Geotherm Res*, **84**(3-4), 173-196.
- A. Brahme, M.H. Alvi, D. Saylor, J. Fridy, A.D. Rollett (2006) 3D reconstruction of microstructure in a commercial purity aluminum, *Scripta mater.* **55**(1):75-80.
- A.D. Rollett, R. Campman, D. Saylor (2006), Three dimensional microstructures: Statistical analysis of second phase particles in AA7075-T651, *Materials Science Forum* **519-521**: 1-10 Part 1-2, Proceedings of the International Conference on Aluminium Alloys (ICAA-10), Vancouver, Canada.
- A.D. Rollett, S.-B. Lee, R. Campman and G.S. Rohrer, "Three-Dimensional Characterization of Microstructure by Electron Back-Scatter Diffraction," *Annual Reviews in Materials Science*, **37**: 627-658 (2007).
- M.A. Przystupa (1997) Estimation of true size distribution of partially aligned same-shape ellipsoidal particles, *Scripta Mater.*, **37**(11), 1701-1707.
- D. M. Saylor, J. Fridy, B El-Dasher, K. Jung, and A. D. Rollett (2004) Statistically Representative Three-Dimensional Microstructures Based on Orthogonal Observation Sections, *Metall. Trans. A*, **35A**, 1969-1979.

Problems

- What is Stereology useful for?
- Problem solving:
 - How to measure grain size (in 3D)?
 - How to measure volume fractions, size distributions of a second phase
 - How to measure the amount of interfacial area in a material (important for porous materials, e.g.)
 - How to measure crystal facets (e.g. in minerals)
 - How to predict strength (particle pinning of dislocations)
 - How to predict limiting grain size (boundary pinning by particles)
 - How to construct or synthesize *digital microstructures* from 2D data, i.e. how to re-construct a detailed arrangement of grains or particles based on cross-sections.

Measurable Quantities

- $N := \underline{n}$ umber (e.g. of points, intersections)
- $P := \underline{p}$ oints
- $L := \underline{l}$ ine length
- *Blue* \Rightarrow easily measured directly from images
- $A := \underline{a}$ rea
- $S := \underline{s}$ urface or interface area
- $V := \underline{v}$ olume
- *Red* \Rightarrow not easily measured directly

Definitions

Subscripts:

P := per test point

L := per unit of line

A := per unit area

V := per unit volume

T := total

overbar:= average

$\langle X \rangle$ = average of X

E.g. P_A :=

Points per unit area

TABLE 1.1

List of basic symbols and their definitions

Symbol	Dimensions*	Definition
P		Number of point elements, or test points
P_P		Point fraction. Number of points (in areal features) per test point
P_L	mm^{-1}	Number of point intersections per unit length of test line
P_A	mm^{-2}	Number of points per unit test area
P_V	mm^{-3}	Number of points per unit test volume
L	mm	Length of lineal elements, or test line length
L_L	mm/mm	Lineal fraction. Length of lineal intercepts per unit length of test line
L_A	mm/mm^2	Length of lineal elements per unit test area
L_V	mm/mm^3	Length of lineal elements per unit test volume
A	mm^2	Planar area of intercepted features, or test area
S	mm^2	Surface or interface area (not necessarily planar)
A_A	mm^2/mm^2	Area fraction. Area of intercepted features per unit test area
S_V	mm^2/mm^3	Surface area per unit test volume
V	mm^3	Volume of three-dimensional features, or test volume
V_V	mm^3/mm^3	Volume fraction. Volume of features per unit test volume
N		Number of features (as opposed to points)
N_L	mm^{-1}	Number of interceptions of features per unit length of test line
N_A	mm^{-2}	Number of interceptions of features per unit test area
N_V	mm^{-3}	Number of features per unit test volume
\bar{L}	mm	Average lineal intercept, L_L/N_L
\bar{A}	mm^2	Average areal intercept, A_A/N_A
\bar{S}	mm^2	Average surface area, S_V/N_V
\bar{V}	mm^3	Average volume, V_V/N_V

*Arbitrarily shown in millimeters.

[Underwood]

Objectives **Notation** Equations Delesse S_V - P_L L_A - P_L Topology Grain_Size Distributions

Other Quantities

- Δ := nearest neighbor spacing, center-to-center (e.g. between particles)
- λ := mean free path (uninterrupted distance between particles); this is important in calculating the critical resolved shear stress for dislocation motion, for example.
- $(N_A)_b$ is the number of particles per unit area in contact with (grain) boundaries
- N_S is the number of particles (objects) per unit area of a surface; this is an important quantity in particle pinning of grain boundaries, for example.

Quantities measurable in a section

- Or, **what data can we readily extract from a micrograph?**
- We can measure how many points fall in one phase versus another phase, P_P (points per test point) or P_A (points per unit area). Similarly, we can measure area e.g. by counting points on a regular grid, so that each point represents a constant, known area, A_A .
- We can measure lines in terms of line length per unit area (of section), L_A . Or we can measure how much of each test line falls, say, into a given phase, L_L .
- We can *use* lines to measure the presence of boundaries by counting the number of intercepts per line length, P_L .
- We can measure the *angle* between a line and a reference direction; for a grain boundary, this is an *inclination*.

Relationships between Quantities

- $V_V = A_A = L_L = P_P \quad \text{mm}^0$
- $S_V = (4/\pi)L_A = 2P_L \quad \text{mm}^{-1}$
- $L_V = 2P_A \quad \text{mm}^{-2}$
- $P_V = 0.5L_V S_V = 2P_A P_L \quad \text{mm}^{-3} \quad (2.1-4).$
- These are exact relationships, provided that measurements are made with statistical uniformity (randomly). Obviously experimental data is subject to error.

Measured vs. Derived Quantities

TABLE 2.1

Relationship of measured (○) to calculated (□) quantities

Microstructural feature	Dimensions of symbols (arbitrarily expressed in terms of millimeters)			
	mm^0	mm^{-1}	mm^{-2}	mm^{-3}
Points	P_P	P_L	P_A	P_V
Lines	L_L	L_A	L_V	—
Surfaces	A_A	S_V	—	—
Volumes	V_V	—	—	—

Diagram illustrating the relationships between measured (circles) and calculated (squares) quantities across different dimensions:

- P_P (measured) leads to L_L (measured), which leads to A_A (measured), which leads to V_V (calculated).
- P_L (measured) leads to L_A (measured), which leads to S_V (calculated).
- P_L (measured) leads to P_A (measured), which leads to L_V (calculated).
- P_A (measured) leads to P_V (calculated).
- L_A (measured) leads to L_V (calculated).
- S_V (calculated) leads to L_V (calculated).
- L_V (calculated) leads to P_V (calculated).

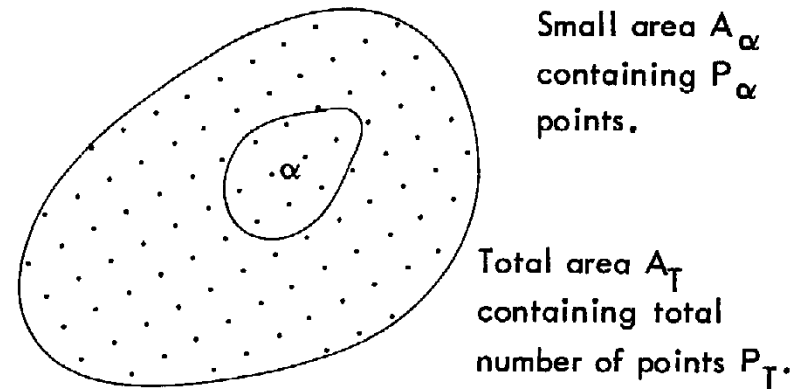
Remember that it is very difficult to obtain true 3D measurements (squares) and so we must find stereological methods to estimate the 3D quantities (squares) from 2D measurements (circles).

Objectives Notation **Equations** Delesse S_V - P_L L_A - P_L Topology Grain_Size Distributions

Volume Fraction

- Typical method of measurement is to identify phases by contrast (gray level, color) and either use pixel counting (point counting) or line intercepts.
- *Volume fractions, surface area (per unit volume), diameters and curvatures* are readily obtained.

Point Counting

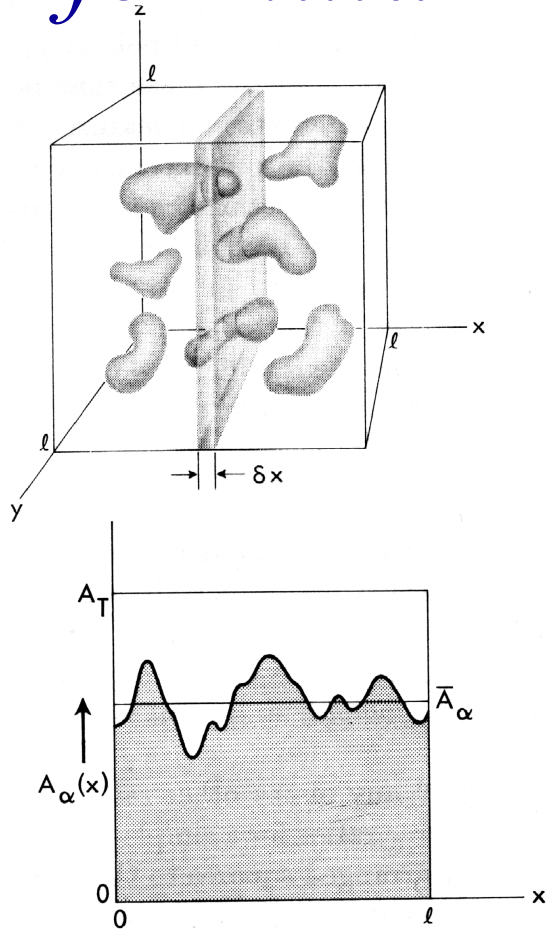


- Issues:
 - Objects that lie partially in the test area should be counted with a factor of 0.5.
 - Systematic point counts give the lowest coefficients of deviation (errors):
coefficient of deviation/variation (CV) = standard deviation (σ) divided by the mean ($\langle x \rangle$), $CV = \sigma(x) / \langle x \rangle$.

Delesse's Principle: Measuring volume fractions of a second phase

- The French geologist Delesse pointed out (1848) that $A_A = V_V$ (2.11).
- Rosiwal pointed out (1898) the equivalence of point and area fractions, $P_P = A_A$ (2.25).
- Relationship for the surface area per unit volume derived from considering lines piercing a body: by averaging over all inclinations of the line

Derivation: Delesse's formula



$$V_{test} = l^3$$

$$A_{test} = l^2$$

$$\delta V_{\alpha} = l^2 \delta x (V_V)_{\alpha}$$

$$\delta V_{\alpha} = A_{\alpha}(x) \delta x$$

$$\bar{A}_{\alpha} = \int_0^l dV_{\alpha} = \int_0^l A_{\alpha}(x) dx$$

$$V_{\alpha} = l \bar{A}_{\alpha}$$

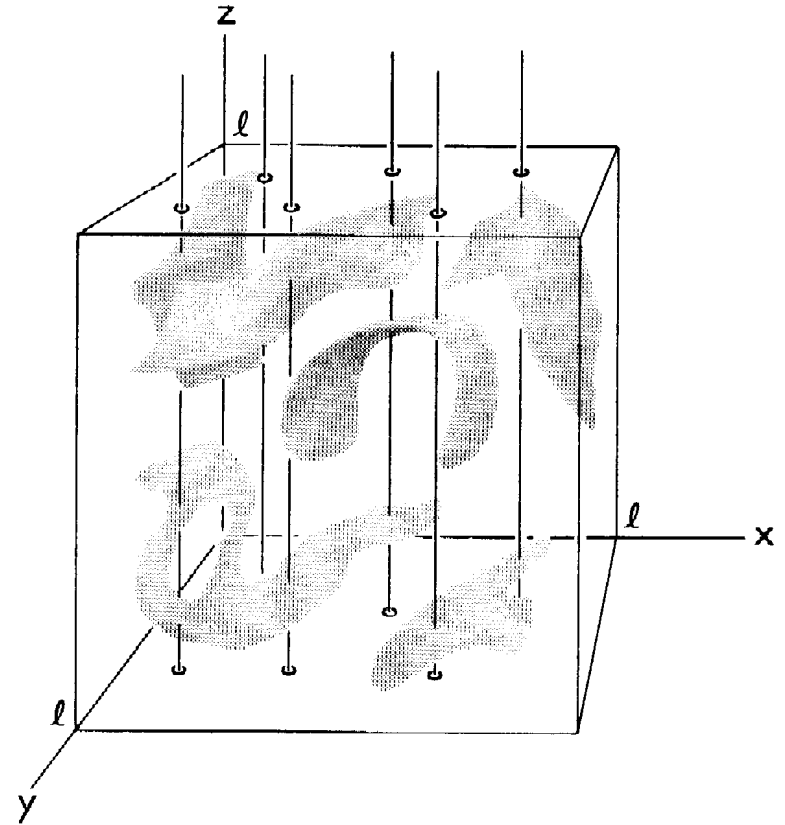
$$V_{\alpha} / V_{test} = \bar{A}_{\alpha} / A_{test}$$

$$V_V = \bar{A}_A = A_A$$

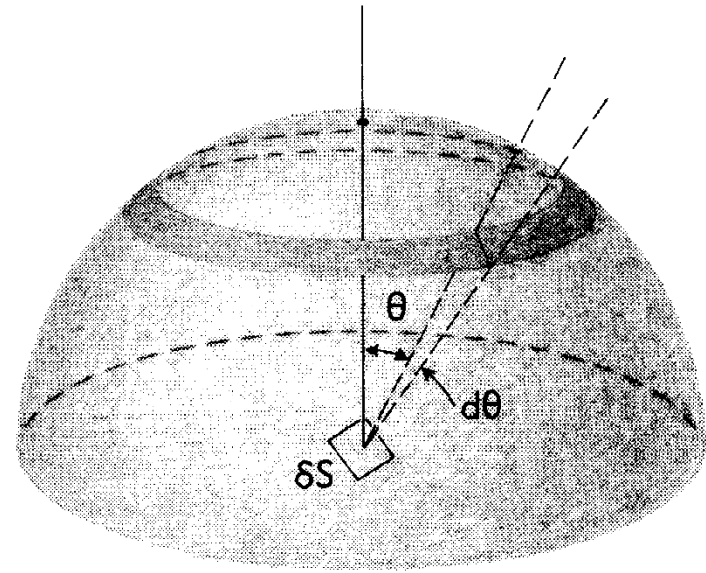
Basic idea:
Integrate area
fractions over
the volume

Surface Area (per unit volume)

- $S_V = 2P_L$ (2.2).
- Derivation based on random intersection of lines with (internal) surfaces. Probability of intersection depends on inclination angle, θ , between the test line and the normal of the surface. Averaging θ gives factor of 2.



$$S_V = 2P_L$$



- Derivation based on uniform distribution of elementary areas.
- Consider the dA to be distributed over the surface of a sphere. The sphere represents the effect of randomly (uniformly) distributed surfaces.
- Projected area = $dA \cos\theta$.
- Probability that a line will intersect with a given patch of area on the sphere is proportional to *projected* area on the plane.
- This is useful for obtaining information on the full 5 parameter *grain boundary character distribution* (a later lecture).

$$S_V = 2P_L$$

$$dA = r^2 \sin\theta d\theta d\varphi; \quad dA_{\text{projected}} = dA \cos\theta$$

$$\frac{\langle A_{\text{projected}} \rangle}{A_{\text{total}}} = \frac{\iint dA \cos\theta}{\iint dA}$$

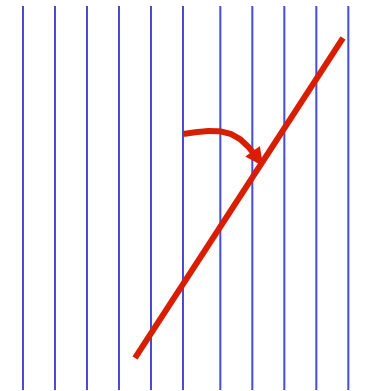
$$\frac{\langle A_{\text{projected}} \rangle}{A_{\text{total}}} = \frac{\int_0^{2\pi} \int_0^{\pi/2} r^2 \sin\theta \cos\theta d\theta d\varphi}{\int_0^{2\pi} \int_0^{\pi/2} r^2 \sin\theta d\theta d\varphi} = \frac{0.5 \int_0^{\pi/2} \sin 2\theta d\theta}{\int_0^{\pi/2} \sin\theta d\theta}$$

$$\frac{\langle A_{\text{projected}} \rangle}{A_{\text{total}}} = \frac{1/4 [-\cos 2\theta]_0^{\pi/2}}{[\cos\theta]_0^{\pi/2}} = \frac{1/4 [1 - (-1)]}{1} = \frac{2}{4}$$

$$\frac{\langle A_{\text{projected}} \rangle}{A_{\text{total}}} = \frac{1}{2} = \frac{P_L}{S_V}$$

Length of Line per Unit Area, L_A versus Intersection Points Density, P_L

- Set up the problem with a set of **test lines** (vertical, arbitrarily) and a **line to be sampled**. The sample line can lie at any angle: what will we measure?



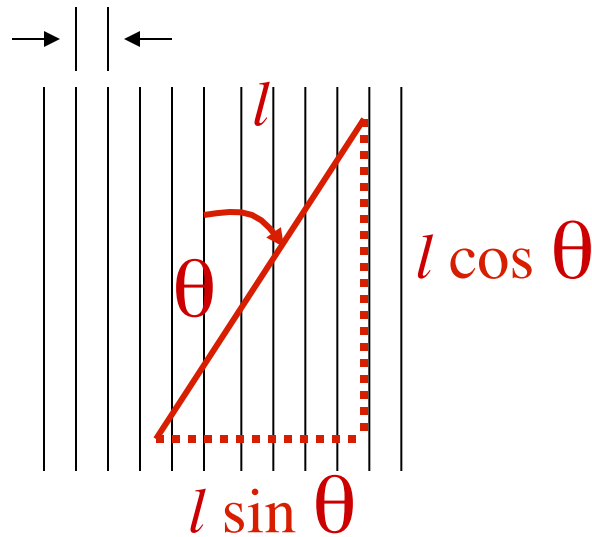
ref: p38/39 in Underwood

This was first considered by Buffon, *Essai d'arithmétique morale*, Supplément à l'Histoire Naturelle, **4**, (1777) and the method has been used to estimate the value of π . Consequently, this procedure is also known as Buffon's Needle.

Objectives Notation Equations Delesse S_V - P_L L_A - P_L Topology Grain_Size Distributions

$$L_A = \pi/2 P_L, \text{ contd.}$$

Δx , or d



The number of points of intersection with the test grid depends on the angle between the sample line and the grid. Larger θ value means more intersections. The projected length = $l \sin \theta = l P_L \Delta x$.

$$P_L = \frac{\sin \theta}{\Delta x}$$

- Line length in area, L_A ; consider an arbitrary area of x by x :

$$L_A = x \frac{x}{\Delta x} \div \frac{1}{x^2} = \frac{1}{\Delta x}$$

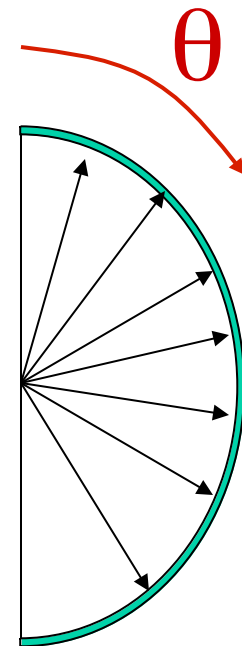
Therefore to find the relationship between P_L and L_A for the general case where we do not know Δx , we must average over all values of the angle θ .

$$L_A = \pi/2 P_L, \text{ contd.}$$

- Probability of intersection with test line given by average over all values of θ :

$$p = \frac{\int_0^\pi l \sin \theta d\theta}{\int_0^\pi l d\theta} = \frac{l [-\cos \theta]_0^\pi}{l [\theta]_0^\pi} = \frac{2}{\pi}$$

- Density of intersection points, P_L , to Line Density per unit area, L_A , is given by this probability. Note that a simple experiment estimates π (but beware of errors!).



Buffon's Needle Experiment

- In fact, to perform an actual experiment by dropping a needle onto paper requires care. One must always perform a very large number of trials in order to obtain an accurate value. The best approach is to use ruled paper with parallel lines at a spacing, d , and a needle of length, l , less than (or equal to) the line spacing, $l \leq d$. Then one may use the following formula. (A more complicated formula is needed for long needles.) The total number of dropped needles is N and the number that cross (intersect with) a line is n .

$$\pi = \frac{2(l/d)N}{n}$$

See: <http://www.ms.uky.edu/~mai/java/stat/buff.html>

Also <http://mathworld.wolfram.com/BuffonsNeedleProblem.html>

$$S_V = (4/\pi)L_A$$

- If we can measure the line length per unit area directly, then there is an equivalent relationship to the surface area per unit volume.
- This relationship is immediately obtained from the previous equations:

$$S_V/2 = P_L \quad \text{and} \quad P_L = (2/\pi)L_A.$$

- In the OIM software, for example, grain boundaries can be automatically recognized and their lengths counted to give an estimate of L_A . From this, the *grain boundary area per unit volume* can be estimated (as S_V).

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Line length per unit volume, L_V vs. Points per unit area, P_A

- Equation 2.3 states that $L_V = 2P_A$.
- Practical application: estimating dislocation density from intersections with a plane.
- Derivation based on similar argument to that for surface:volume ratio. Probability of intersection of a line with a section plane depends on the inclination of the line with respect to (*w.r.t.*) the plane: therefore we average a term in $\cos(\theta)$.

Oriented structures: 2D

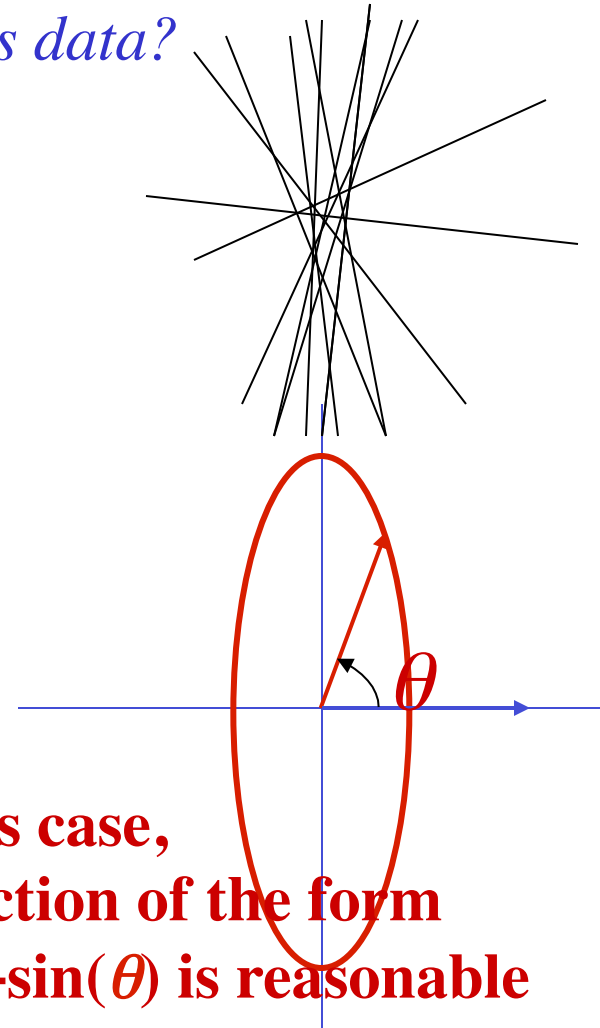
- For highly oriented structures, it is sensible to define specific directions (axes) aligned with the preferred directions (e.g. twinned structures) and measure L_A w.r.t. the axes.
- For less highly oriented structures, *orientation distributions* should be used (just as for *pole figures!*):

$$L_A^{total} = \frac{1}{\pi} \int_0^{\pi} L_A(\theta) d\theta$$

Distribution of Lines on Plane

- The diagram in the top left shows a set of lines, obviously not uniformly distributed.
- The lower right diagram shows the corresponding distribution.
- Clearly the distribution has smoothed the exptl. data.

What function can we fit to this data?



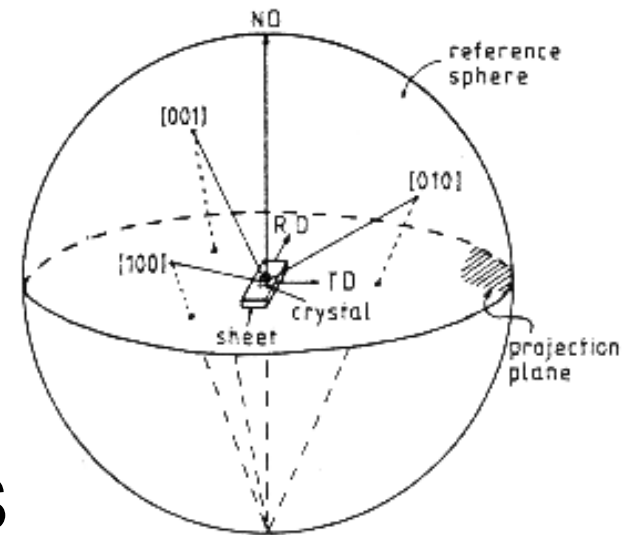
**In this case,
a function of the form
 $r = a + \sin(\theta)$ is reasonable**

Generalizations

- Now that we have seen what a circular distribution looks like, we can make connections to more complicated distributions.
- 1-parameter distributions: the distribution of line directions in a plane is exactly equivalent to the density of points along the circumference of a (unit radius) circle.
- So how can we generalize this to *two* parameters?
Answer: consider the distribution or density of points on a (unit radius) sphere. Here we want to characterize/measure the *density of points per unit area*.
- How does this connect with what we have learned about texture?
Answer: since the direction in which a specified crystal plane normal points (relative to specimen axes) can be described as the intersection point with a unit sphere, the distribution of points on a sphere is exactly a pole figure!

Oriented structures: 3D

Again, for less highly oriented structures, *orientation distributions* should be used (just as for *pole figures*): note the incorporation of the normalization factor on the RHS of (Eq. 3.32).



$$L_V^{total} = \frac{1}{2\pi} \int_0^{2\pi} \int_0^{\pi/2} L_V(\phi, \theta) \sin \phi d\phi d\theta$$

See also Ch. 12 of Bunge's book; in this case, surface spherical harmonics are useful (trigonometric functions of ϕ and θ). See, e.g.

http://imaging.indyrad.iupui.edu/projects/SPHARM/SPHARM-docs/C01_Introduction.html

for a Matlab package.

Orientation distributions

- Given that we now understand how to describe a 2-parameter distribution on a sphere, how can we connect this to orientation distributions and crystals?
- The question is, how can we generalize this to *three* parameters?

Answer: consider the distribution or density of points on a (unit radius) sphere with another direction associated with the first one. Again, we want to characterize/measure the density of points per unit area but now there is a third parameter involved. The analogy that can be made is that of determining the position *and* the heading of a boat on the globe. One needs latitude, longitude and a heading angle in order to do it. As we shall see, the functions required to describe such distributions are correspondingly more complicated (*generalized spherical harmonics*).

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Second Phase Particles

- Now we consider second phase particles
- Although the derivations are general, we mostly deal with small volume fractions of convex, (nearly) spherical particles
- Quantities of interest:
 - intercept length, P_L or N_L
 - particle spacing, Δ
 - mean free path, λ (or uninterrupted distance between particles)

S_V and 2nd phase particles

- Convex particles:= *any* two points on particle surface can be connected by a wholly internal line.
- Sometimes it is easier to count the number of particles intercepted along a line, N_L ; then the number of surface points is double the particle number. Also applies to non-convex particles if interceptions counted.

$$S_V = 4N_L \quad (2.32)$$

S:V and Mean Intercept Length

- Mean intercept length in 3 dimensions, $\langle L_3 \rangle$, from intercepts of particles of a (dispersed) alpha phase:

$$\langle L_3 \rangle = 1/N \sum_i (L_3)_i \quad (2.33)$$

- Can also be obtained as:

$$\langle L_3 \rangle = L_L / N_L \quad (2.34)$$

- Substituting:

$$\langle L_3 \rangle = 4V_V / S_V, \quad (2.35)$$

where fractions refer to the (dispersed) alpha phase only.

S:V example: sphere

- For a sphere, the volume:surface ratio ($=V_V/S_V$) is $D[\text{iameter}]/6$.
- Thus $\langle L_3 \rangle_{\text{sphere}} = 2D/3$.
- In general we can invert the relationship to obtain the surface:volume ratio, if we know (measure) the mean intercept:

$$\langle S/V \rangle_{\text{alpha}} = 4/\langle L_3 \rangle \quad (2.38)$$

Table 2.2

$\langle L_3 \rangle :=$ mean intercept length, 3D objects

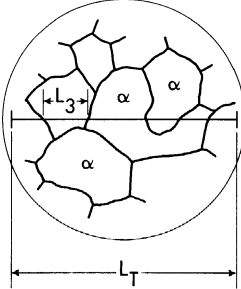
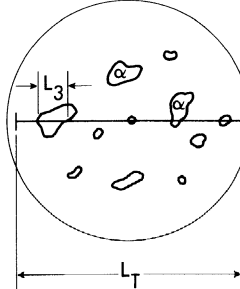
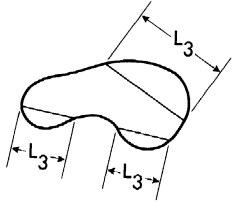
$\langle V \rangle :=$ mean volume

$l :=$ length (constant) of test lines superimposed on structure

$p :=$ number of (end) points of l -lines in phase of interest

$L_T :=$ test line length

TABLE 2.2 Three-dimensional cells and particles

<p>(a) Space-filling contiguous cells of one phase ($(V_V)_\alpha = 1$)</p>  <p>Two-dimensional internal surfaces* with sharing between contiguous cells.</p> <p>$N_L = \frac{1}{2} + 4 + \frac{1}{2} = 5$ $P_L = 5$ $P_L = N_L = 1/\bar{L}_3$</p>	<p>(b) Dispersed particles of α-phase [$(V_V)_\alpha < 1$] in a matrix phase</p>  <p>Two-dimensional interfaces† not shared with other α-particles.</p> <p>$N_L = 4$ $P_L = 8$ $P_L = 2N_L = 2(V_V)_\alpha/\bar{L}_3$</p>	<p>(c) Isolated single particle (volume = V) not associated with a matrix</p>  <p>Two-dimensional external particle surface S.§</p> <p>$N_L = 1, 2, \dots$ $P_L = 2, 4, \dots$ $P_L = 2N_L$</p>
<p>Saltykov [5,27] $S_V = 2P_L = 2N_L$</p>	<p>$(S_V)_\alpha = 2P_L = 4N_L$</p>	<p>$\frac{S}{V} = \frac{2P_L}{P}$</p>
<p>Tomkeieff [14] $\bar{L}_3 = 2 \frac{\bar{V}}{\bar{S}} = \frac{2}{\bar{S}_V}$</p>	<p>$\bar{L}_3 = \frac{4\bar{V}_\alpha}{\bar{S}_\alpha} = \frac{4(V_V)_\alpha}{(S_V)_\alpha}$</p>	<p>$\bar{L}_3 = \frac{4V}{S}$</p>
<p>Chalkley [18] $\bar{L}_3 = lp/2P$</p>	<p>$\bar{L}_3 = \frac{lp}{P} = \frac{4\bar{V}_\alpha}{\bar{S}_\alpha}$</p>	<p>$\bar{L}_3 = \frac{lp}{P} = \frac{4V}{S}$</p>
<p>*Internal surface area referred to test volume (S_V) or to mean cell volume (\bar{S}/\bar{V}).</p>	<p>†Interface area of α-particles referred to test volume (S_V)$_\alpha$ or to mean particle volume ($\bar{S}_\alpha/\bar{V}_\alpha$).</p>	<p>§External surface area referred to particle volume (S/V).</p>

[Underwood]

Grain size measurement: intercepts

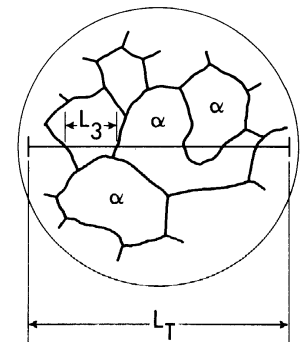
- From Table 2.2 [Underwood], column (a), illustrates how to make a measurement of the mean intercept length, based on the number of grains per unit length of test line.

$$\langle L_3 \rangle = 1/N_L$$

- Important: use many test lines that are randomly oriented w.r.t. the structure.
- Assuming spherical[†] grains, $\langle L_3 \rangle = 4r/3$, [Underwood, Table 4.1], there are 5 intersections and if we take the total test line length, $L_T = 25\mu\text{m}$, then $L_T N_L = 5$, so $N_L = 1/5 \mu\text{m}^{-1}$

$$\therefore d = 2r = 6\langle L_3 \rangle / 4 = 6 / N_L \cdot 4 = 6 * 5 / 4 = 7.5\mu\text{m}.$$

[†] Ask yourself what a better assumption about grain shape might be!



Particles and Grains

- “Where the rubber meets the road”, in stereology, that is! By which we mean that particles and pores are very important in materials processing therefore we need to know how to work with them.
- Mean free distance, $\lambda :=$ uninterrupted interparticle distance through the matrix averaged over all pairs of particles (in contrast to interparticle distance for nearest neighbors only).

$$\lambda = \frac{1 - V_V^{(\alpha)}}{N_L} \quad (4.7)$$

Number of interceptions with particles is same as number of interceptions with the matrix. Thus lineal fraction of occupied by matrix is λN_L , equal to the volume fraction, $1 - V_V^{(\alpha)}$.

Mean Random Spacing

- The number of interceptions with particles per unit test length = $N_L = P_L/2$. The reciprocal of this quantity is the *mean random spacing*, σ , which is the mean uninterrupted center-to-center length between *all possible pairs* of particles (also known as the *mean free path*). Thus, the particle mean intercept length, $\langle L_3 \rangle$:

$$\langle L_3 \rangle = \sigma - \lambda \quad [\text{mm}] \quad (4.8)$$

Particle Relationships

- Application: *particle coarsening* in a 2-phase material; *strengthening* of solid against dislocation flow.
- Eqs. 4.9-4.11, with $L_A = \pi P_L / 2 = \pi N_L = \pi S_V / 4$
- dimension: length units (e.g.): mm

$$\langle L_3 \rangle = 4 \frac{V_V^{(\alpha)}}{S_V^{(\alpha)}}$$

$$\lambda = 4 \frac{1 - V_V^{(\alpha)}}{S_V^{(\alpha)}}$$

$$\lambda = \langle L_3 \rangle \frac{1 - V_V^{(\alpha)}}{V_V^{(\alpha)}}$$

$$\lambda = \pi \frac{1 - V_V^{(\alpha)}}{L_A^{(\alpha)}}$$

Mean free path, λ , versus Nearest neighbor spacing, Δ

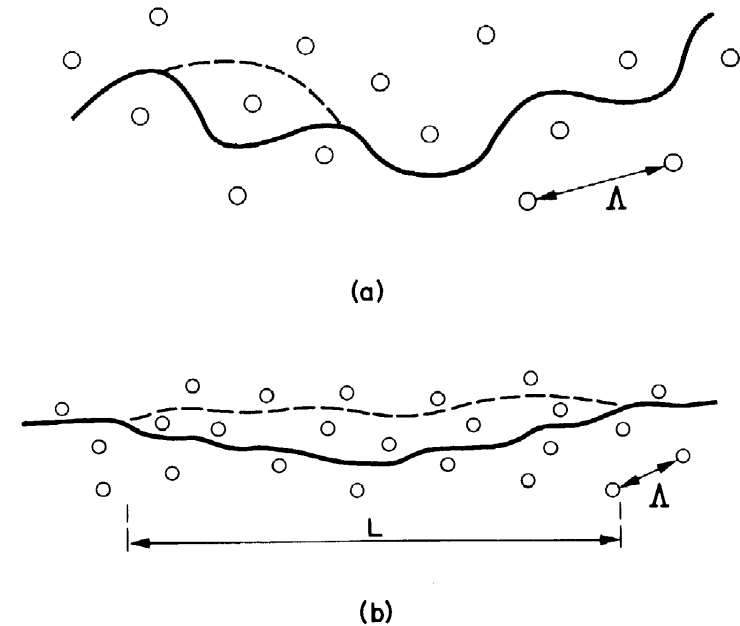
- It is useful (and therefore important) to keep the difference between *mean free path* and *nearest neighbor spacing* separate and distinct.
- *Mean free path* is how far, on average, you travel from one particle until you encounter another one.
- *Nearest neighbor spacing* is how far apart, on average, two nearest neighbors are from each other.
- They appear at first glance to be the same thing but they are not!
- They are related to one another, as we shall see in the next few slides.

Nearest-Neighbor Distances, Δ

- Also useful are distances between nearest neighbors: S. Chandrasekhar, “Stochastic problems in physics and astronomy”, *Rev. Mod. Physics*, **15**, 83 (1943).
- Note how the nearest-neighbor distances, Δ , grow more slowly than the mean free path, λ .
- $r :=$ particle radius
- 2D: $\Delta_2 = 0.5 / \sqrt{P_A}$ (4.18a)
- 3D: $\Delta_3 = 0.554 (P_V)^{-1/3}$ (4.18)
- Based on $\lambda \sim 1/N_L$, $\Delta_3 \approx 0.554 (\pi r^2 \lambda)^{1/3}$
for small V_V , $\Delta_2 \approx 0.500 (\pi/2 r \lambda)^{1/2}$

Application of Δ_2 to Dislocation Motion

- Percolation of dislocation lines through arrays of 2D point obstacles.
- Caution! “Spacing” has many interpretations: select the correct one!
- In general, if the obstacles are weak (lower figure) and the dislocations are nearly straight then the relevant spacing is the *mean free path*, λ .
Conversely, if the obstacles are strong (upper figure) and the dislocations bend then the relevant spacing is the (smaller) *nearest neighbor spacing*, Δ_2 .



Hull & Bacon;
fig. 10.17

Particle Pinning - Summary

- **Strong obstacles** + flexible entities: *nearest neighbor spacing*, Δ , applies.
- **Weak obstacles** + inflexible entities: *mean free path*, λ , applies.
- This applies to dislocations *or* grain boundaries *or* domain walls.
- Note the same dependence on particle size, r , but very different dependence on volume fraction, f !

$$\Delta_3 \approx 0.811 \frac{r}{f^{1/3}}$$

$$\lambda \approx \frac{r}{f}$$

$$f \equiv V_V^{(\alpha)}$$

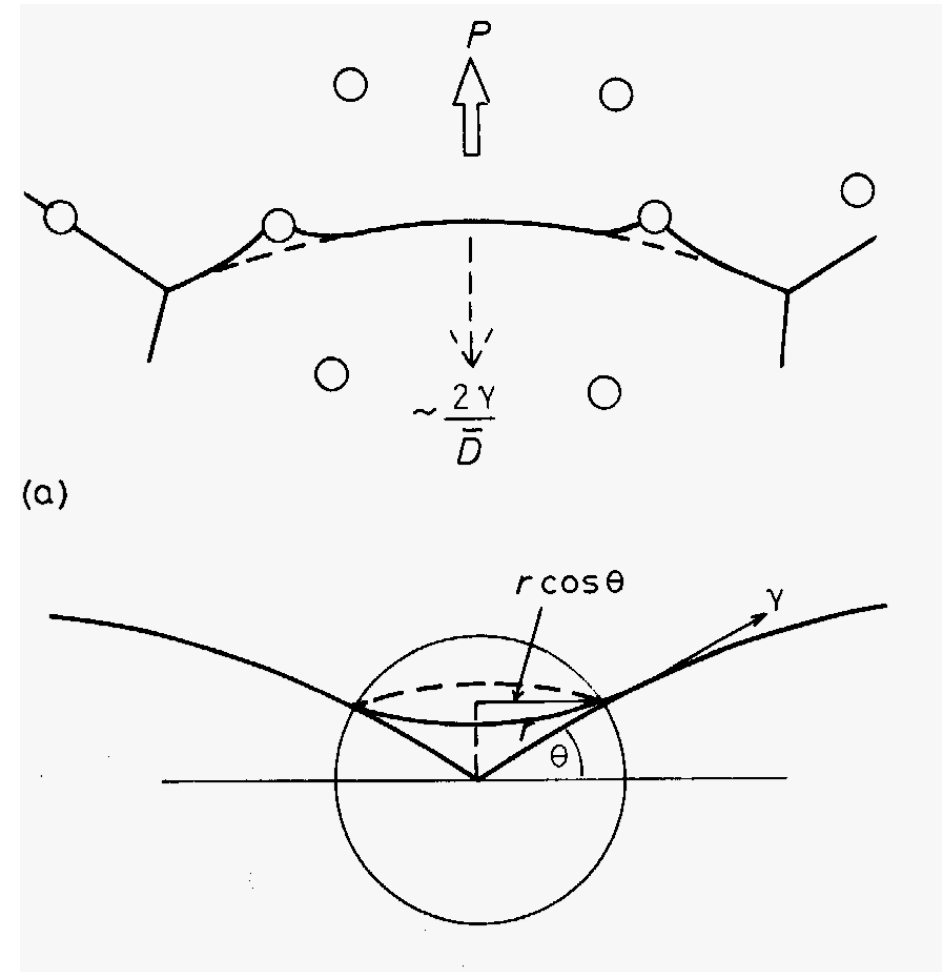
Smith-Zener Pinning of Boundaries

Limiting Grain Size:

$$R_{\max} = K \frac{r}{f^m}$$

Limiting Assumptions:

<i>Zener Model Assumptions (1948)</i>
Rigid Grain Boundary
Spherical Grains
Isotropic Interfacial Energy
Uniform Particle Size
Spherical Particle Shape
Random Distribution
Uniform Grain Size Distribution
Maximum Pinning (Drag) Pressure
Incoherent Particles
Inert Particles



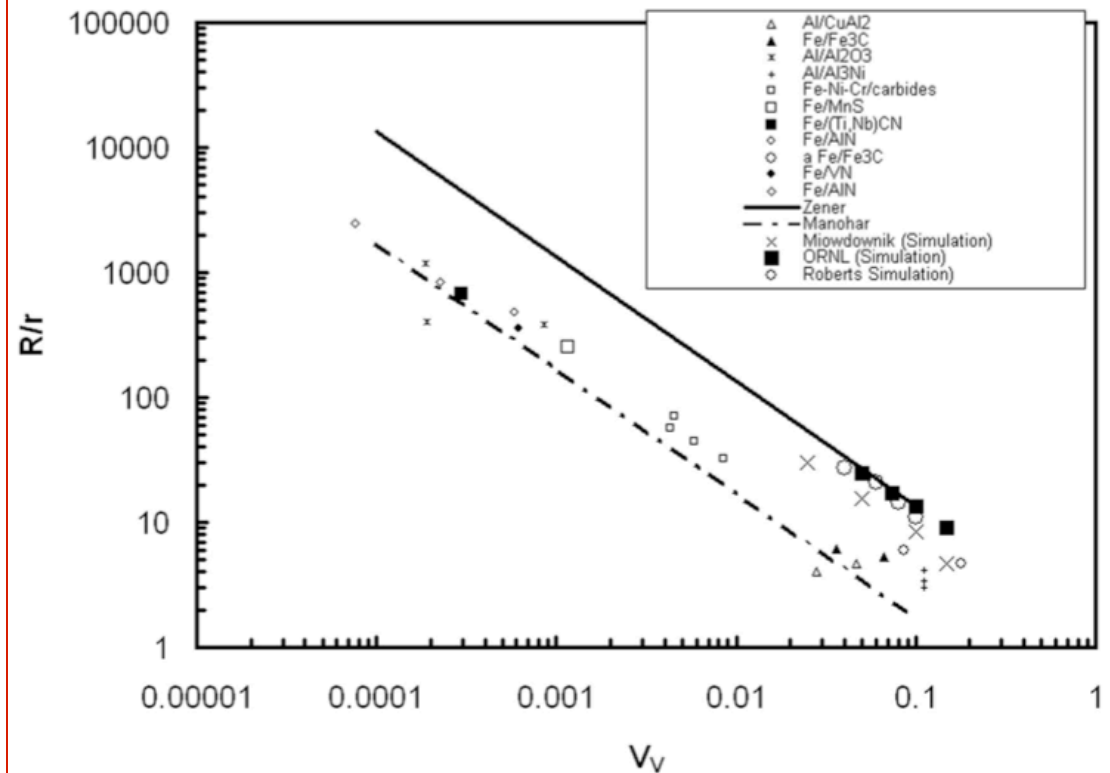
Zener, C. (1948). communication to C.S. Smith. *Trans. AIME*. **175**: 15.

Srolovitz, D. J., M. P. Anderson, et al. (1984), *Acta metall.* **32**: 1429-1438.

E. Nes, N. Ryum and O. Hunderi, *Acta Metall.*, **33** (1985), 11

Smith-Zener Pinning

The literature indicates that the theoretical limiting grain size (solid line) is significantly higher than both the experimental trend line (dot-dash line) and recent simulation results. **The volume fraction dependence, however, corresponds to an interaction of boundaries with particles based on mean free path, λ , $m=1$, not nearest neighbor distances, Δ , $m=0.33$ (in 3D).**



C.G. Roberts, Ph.D. thesis, Carnegie Mellon University, 2007.

B. Radhakrishnan, Supercomputing 2003.

Miodownik, M., E. Holm, et al. (2000), *Scripta Materialia* **42**: 1173-1177.

P.A. Manohar, M.Ferry and T. Chandra, *ISIJ Intl.*, **38** (1998), 913.

Particles on boundaries, in cross-section

An interesting question is to compare the number of particles on boundaries, as a fraction of the total particles in view in a cross-section. We can use the analysis provided by Underwood to arrive at an estimate. If, for example, boundaries have pinned out during grain growth, one might expect the *measured* fraction on boundaries to be higher than this estimate based on random intersection.

- $(N_A)_b$ is the number of particles per unit area in contact with boundaries..
- L_A is the line length per unit area of (grain) boundary.
- The other quantities have their usual meanings.

Underwood 4.36: $N_V = \frac{N_A}{2r}$, uniform spherical particles

Underwood 4.48: $N_S = \frac{(N_A)_b}{2rS_V}$

Underwood 4.49: $N_S = 2rN_V$

Combine 4.36 & 4.49: $N_S = \frac{N_A}{2r} 2r = N_A$

$$\therefore 2rS_V N_S = (N_A)_b$$

$$2r \frac{4}{\pi} L_A N_A = (N_A)_b$$

$$(N_A)_b = \frac{8r}{\pi} L_A N_A$$

As a fraction:

$$\frac{(N_A)_b}{N_A} = \frac{8r}{\pi} L_A$$

See: "Particle-Associated Misorientation Distribution in a Nickel-Base Superalloy".
Roberts C.G., Semiatin S.L., Rollett A.D., *Scripta materialia* **56** 899-902 (2007).

Outline

- Objectives
 - Motivation
 - Quantities,
 - definitions
 - measurable
 - Derivable
 - Problems that use Stereology, Topology
 - Volume fractions
 - Surface area per unit volume
- Facet areas
 - Oriented objects
 - Particle spacings
 - Mean Free Path
 - Nearest Neighbor Distance
 - Zener Pinning
 - **Grain Size**
 - Sections through objects
 - Size Distributions

Grain Size Measurement

- Measurement of grain size is a classic problem in stereology. There are two different approaches (for 2D images), which rarely yield the same answer.
- *Method A*: measure **areas of grains**; calculate grain size based on an assumed shape (that determines the size:projected_area ratio.)
- *Method B*: measure **linear intercepts of grains**; calculate grain size based on an assumed shape (that, in this case, determines the ratio of size to projected length).
- Underwood recommends the latter approach because the mean intercept length, $\langle L_3 \rangle$ is closely related to the surface area per volume, $\langle L_3 \rangle = 2/S_V$.
- Grain size number based on the E112 ASTM standard.
- The problem of plane sections (stereology).
- The problem of grain shape.
- See: <http://www.metallography.com/grain.htm>
- Useful references: *Quantitative Stereology*, E.E. Underwood, Addison-Wesley, 1970; *Practical Stereology* by John C. Russ.

Method A: typical section

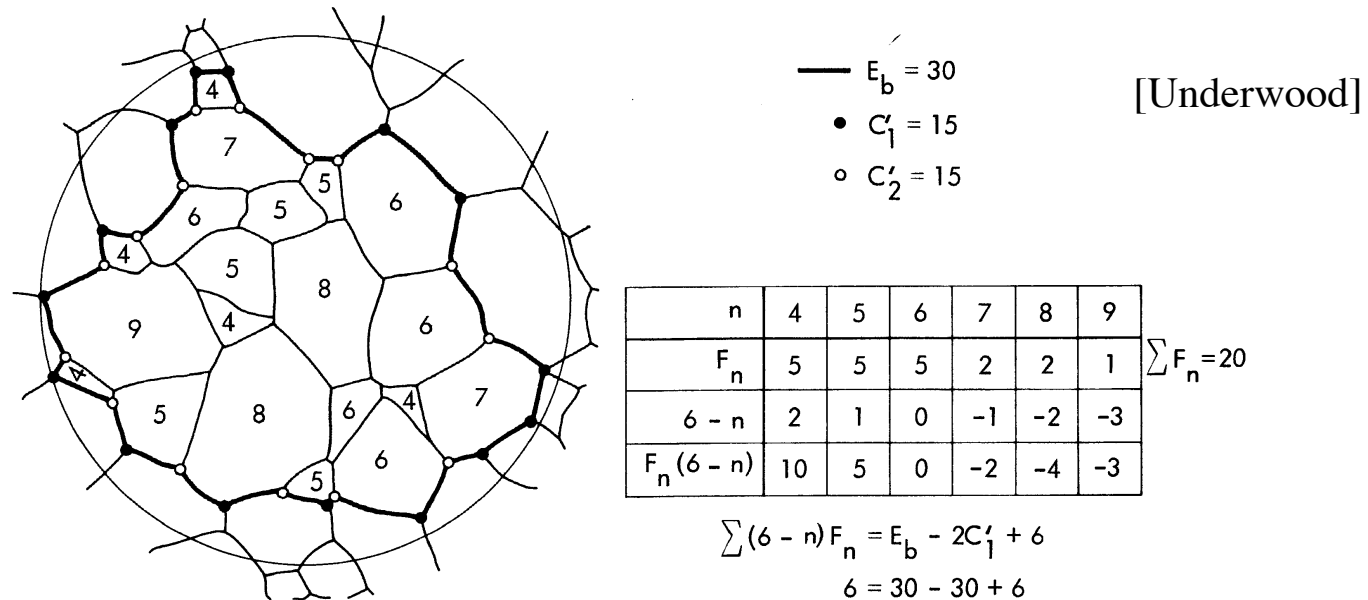


Fig. 7.12. Topological analysis of typical section [8] through metal grains.

- Correction terms (E_b , C_1' , C_2') allow finite sections to be interpreted.
 C_1' := number of incomplete corners against 1 polygon;
 C_2' := same for 2 polygons

Method A: area based

- Grain count method:

$$\langle A \rangle = 1/N_A$$

- Number of whole grains = 20

Number of edge grains = 21

$$\begin{aligned} \text{Effective total} &= N_{\text{whole}} + N_{\text{edge}}/2 \\ &= 30.5 \end{aligned}$$

Total area = 0.5 mm²

Thus, $N_A = 61 \text{ mm}^{-2}$; $\langle A \rangle = 16,400 \text{ } \mu\text{m}^2$

- Assume spherical* grains, $\langle A \rangle$ mean intercept area = $2/3\pi r^2$

$$\therefore d = 2\sqrt{(3\langle A \rangle/2\pi)} = 177 \text{ } \mu\text{m}.$$

*Do you think this is a reasonable assumption?!

[Underwood]

[Underwood]
Fig. 7.12

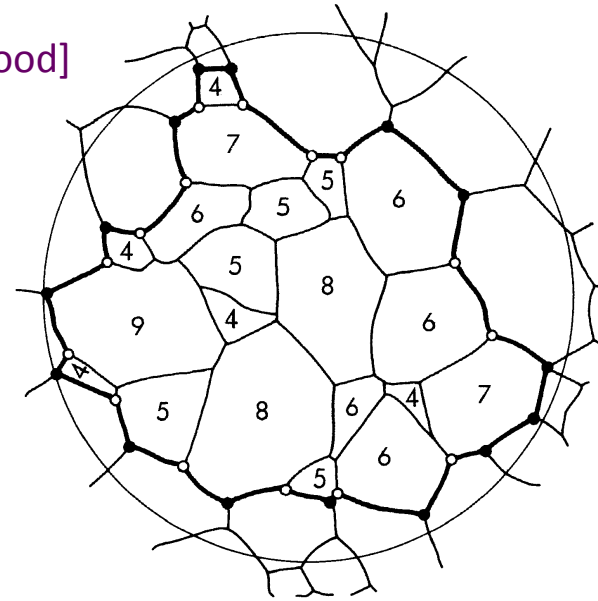


Fig. 7.12. Topological analysis of typical section

Method B: linear intercept

- From Table 2.2 [Underwood], column (a), illustrates how to make a measurement of the mean intercept length, based on the number of grains per unit length of test line.

$$\langle L_3 \rangle = 1/N_L$$

- Important: use many test lines that are randomly oriented w.r.t. the structure.
- Assuming spherical† grains, $\langle L_3 \rangle = 4r/3$, [Underwood, Table 4.1], if we take the total line length (diameter of test area), $L_T = 798 \mu\text{m}$, and draw a line that intersects 7 boundaries, then $N_L = 1/114 \mu\text{m}^{-1}$
 $\therefore d = 6\langle L_3 \rangle/4 = 6/N_L/4 = 6 \cdot 114/4 = 171 \mu\text{m}$.
- Clearly the two measures of grain size are similar but not necessarily the same.

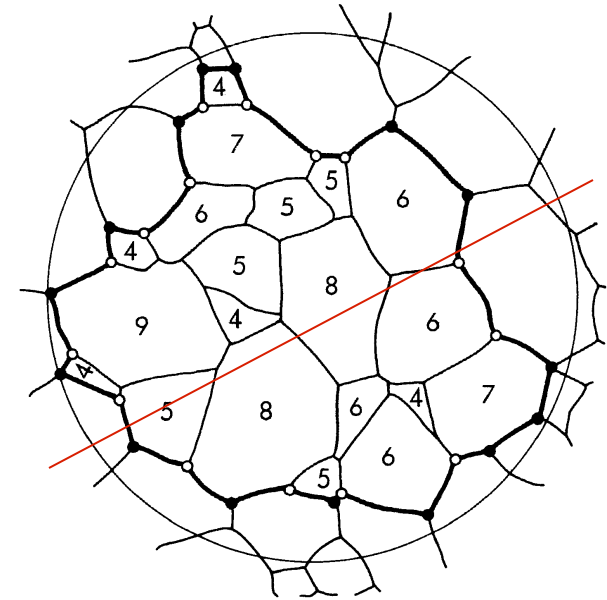
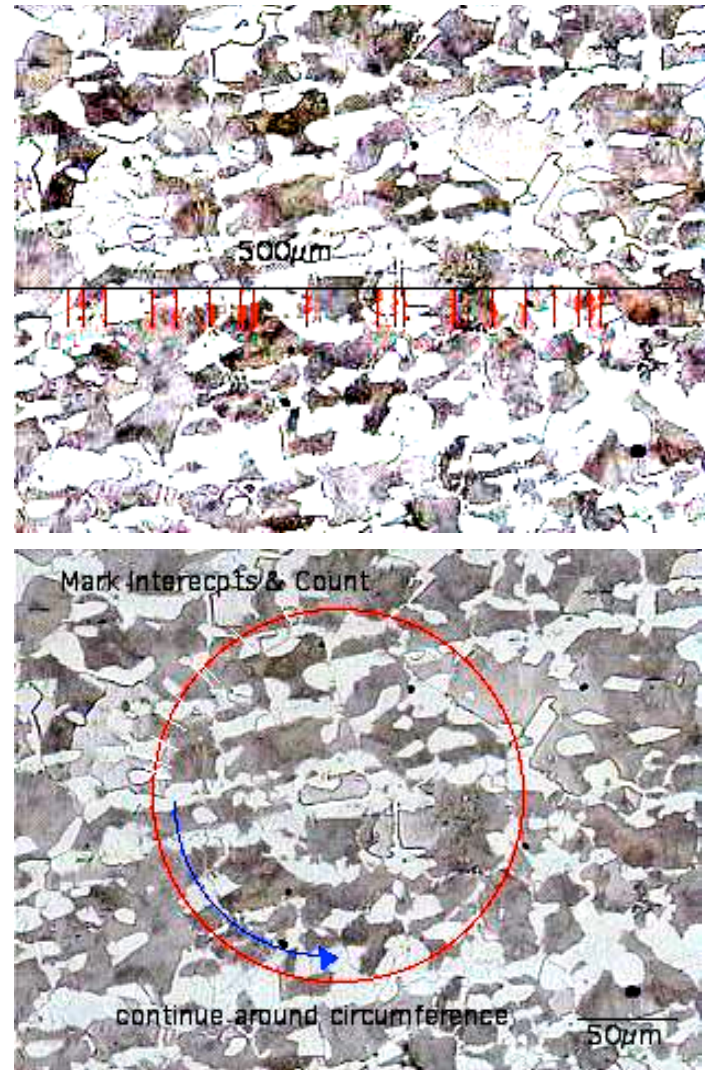


Fig. 7.12. Topological analysis of typical section

† Ask yourself what a better assumption about grain shape might be!

More about the Line Intercept Technique

- One can either count the number of intercepts per unit length along a **straight** line (which is sensitive to the orientation of the line)
- Or, one can count intercepts around a **circle** (eliminates any anisotropy in the microstructure) and divide by the perimeter length of the circle to obtain P_L .
- $Grain\ size = \langle L_3 \rangle = P_L^{-1}$
- Note some elementary image analysis: increasing the *contrast* on the original images made it much easier to perceive the two separate phases.



Alternative Representation: ASTM Grain Size Number

- ASTM has defined a standard, E112, for grain size measurement.
- ASTM has a grain size parameter, G , which can be calculated based on *either* area *or* linear measurements.

Equation	Units
$G = (3.321928 \log_{10} \bar{N}_A) - 2.954$	\bar{N}_A in mm^{-2}
$G = (6.643856 \log_{10} N_L) - 3.288$	N_L in mm^{-1}
$G = (6.643856 \log_{10} P_L) - 3.288$	P_L in mm^{-1}
$G = (-6.643856 \log_{10} \ell) - 3.288$	ℓ in mm

- This ASTM grain size number, G , is commonly employed within industry and earlier research efforts (before computer technologies were available).
- *Higher* grain size number means *smaller* grain size.

E 112 – 96^{€2}

TABLE 4 Grain Size Relationships Computed for Uniform, Randomly Oriented, Equiaxed Grains

Grain Size No. <i>G</i>	N_A Grains/Unit Area		\bar{A} Average Grain Area		\bar{d} Average Diameter		\bar{r} Mean Intercept		N_L No./mm
	No./in. ² at 100X	No./mm ² at 1X	mm ²	μm ²	mm	μm	mm	μm	
00	0.25	3.88	0.2581	258064	0.5080	508.0	0.4525	452.5	2.21
0	0.50	7.75	0.1290	129032	0.3592	359.2	0.3200	320.0	3.12
0.5	0.71	10.96	0.0912	91239	0.3021	302.1	0.2691	269.1	3.72
1.0	1.00	15.50	0.0645	64516	0.2540	254.0	0.2263	226.3	4.42
1.5	1.41	21.92	0.0456	45620	0.2136	213.6	0.1903	190.3	5.26
2.0	2.00	31.00	0.0323	32258	0.1796	179.6	0.1600	160.0	6.25
2.5	2.83	43.84	0.0228	22810	0.1510	151.0	0.1345	134.5	7.43
3.0	4.00	62.00	0.0161	16129	0.1270	127.0	0.1131	113.1	8.84
3.5	5.66	87.68	0.0114	11405	0.1068	106.8	0.0951	95.1	10.51
4.0	8.00	124.00	0.00806	8065	0.0898	89.8	0.0800	80.0	12.50
4.5	11.31	175.36	0.00570	5703	0.0755	75.5	0.0673	67.3	14.87
5.0	16.00	248.00	0.00403	4032	0.0635	63.5	0.0566	56.6	17.68
5.5	22.63	350.73	0.00285	2851	0.0534	53.4	0.0476	47.6	21.02
6.0	32.00	496.00	0.00202	2016	0.0449	44.9	0.0400	40.0	25.00
6.5	45.25	701.45	0.00143	1426	0.0378	37.8	0.0336	33.6	29.73
7.0	64.00	992.00	0.00101	1008	0.0318	31.8	0.0283	28.3	35.36
7.5	90.51	1402.9	0.00071	713	0.0267	26.7	0.0238	23.8	42.04
8.0	128.00	1984.0	0.00050	504	0.0225	22.5	0.0200	20.0	50.00
8.5	181.02	2805.8	0.00036	356	0.0189	18.9	0.0168	16.8	59.46
9.0	256.00	3968.0	0.00025	252	0.0159	15.9	0.0141	14.1	70.71
9.5	362.04	5611.6	0.00018	178	0.0133	13.3	0.0119	11.9	84.09
10.0	512.00	7936.0	0.00013	126	0.0112	11.2	0.0100	10.0	100.0
10.5	724.08	11223.2	0.000089	89.1	0.0094	9.4	0.0084	8.4	118.9
11.0	1024.00	15872.0	0.000063	63.0	0.0079	7.9	0.0071	7.1	141.4
11.5	1448.15	22446.4	0.000045	44.6	0.0067	6.7	0.0060	5.9	168.2
12.0	2048.00	31744.1	0.000032	31.5	0.0056	5.6	0.0050	5.0	200.0
12.5	2896.31	44892.9	0.000022	22.3	0.0047	4.7	0.0042	4.2	237.8
13.0	4096.00	63488.1	0.000016	15.8	0.0040	4.0	0.0035	3.5	282.8
13.5	5792.62	89785.8	0.000011	11.1	0.0033	3.3	0.0030	3.0	336.4
14.0	8192.00	126976.3	0.000008	7.9	0.0028	2.8	0.0025	2.5	400.0

Outline

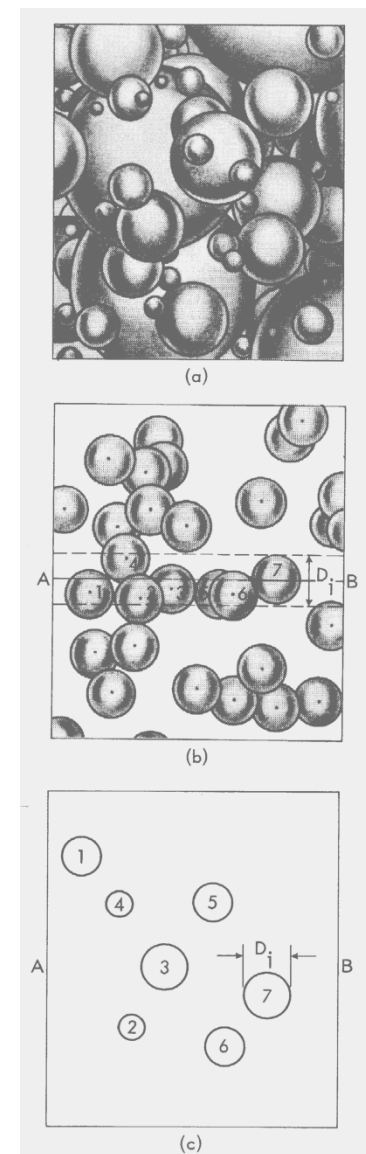
- Objectives
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 - Grain Size
 - Sections through objects
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3D Size Derived from 2D Sections

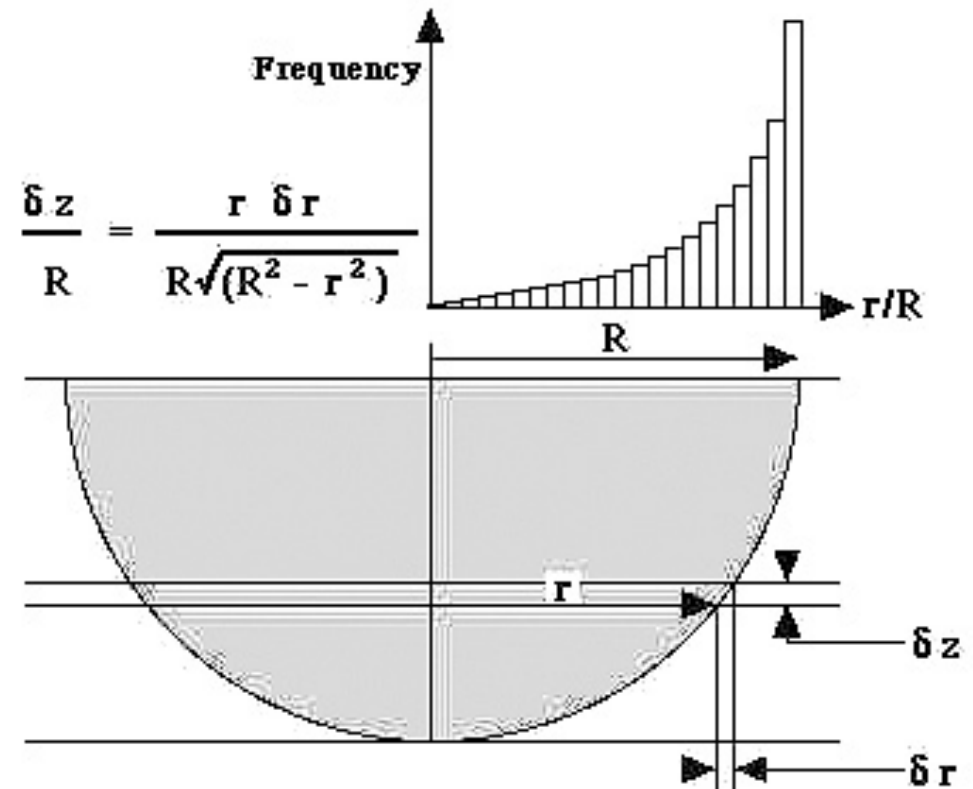
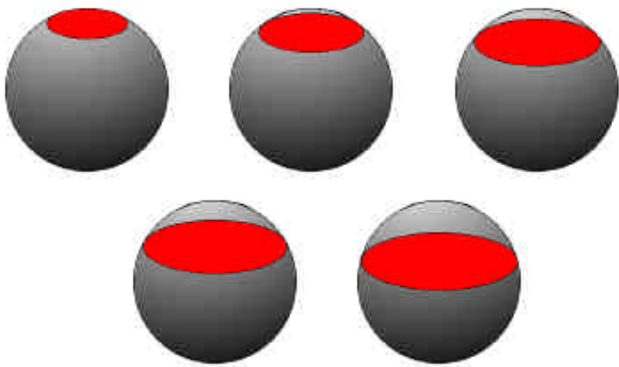
- Purpose: how can we relate measurements in plane sections to what we know of the geometry of regularly shaped objects with a distribution of sizes?
- In general, the mean intercept length is *not* equal to the grain diameter, for example! Also, the proportionality factors depend on the (assumed) shape.
- Example: for **monodisperse spherical particles** (all the same size) distributed (randomly) in space, sectioning through them and measuring the size distribution will show a **spread in apparent size**.

Sections through dispersions of spherical objects

- Even mono-disperse spheres exhibit a variety of diameters in cross section.
- *Only* if you know that the second phase is monodisperse may you measure diameter from maximum cross-section!



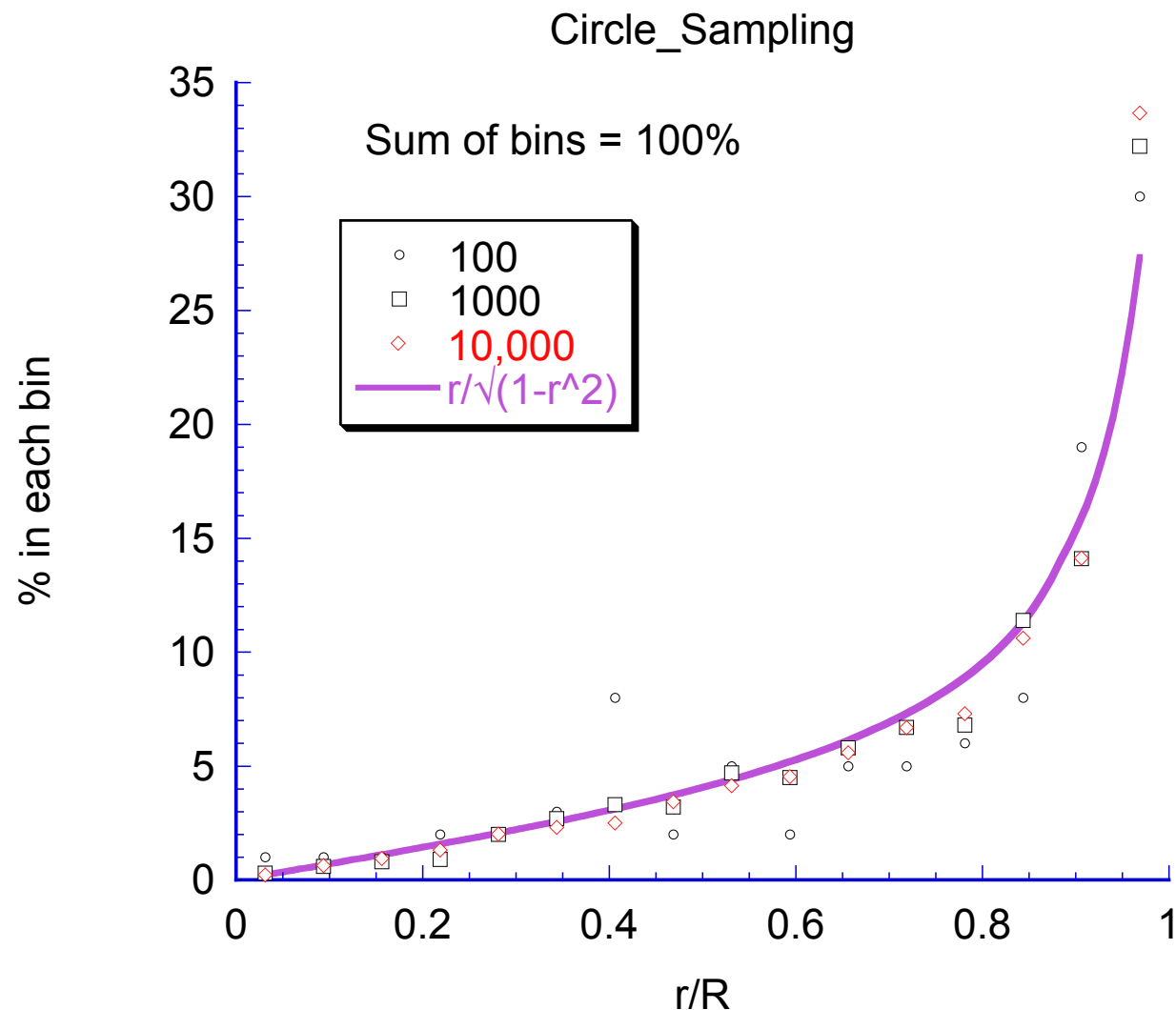
Sectioning Spheres



- The radius, r , of a circle sectioned at a distance h from the center is $r = \sqrt{(R^2 - h^2)}$.
- Since the sectioning planes intersect a sphere at a random location relative to its size, R , we can assume that the probability of observing a circle between a given intercept radius, r , and $r + dr$, is equal to the relative thickness, dz/R , of the corresponding slice.
- The result is a distribution of intercept sizes that varies between zero and the actual sphere size.

Circle Sampling: example

- Numbers for each plot indicate the number of samples taken
- A random number was generated in the range 0..1
- Value of radius of “sampled circle” taken to be $\text{RAN}()/\sqrt{1-\text{RAN}^2}$
- Values binned in 16 bins - note how noisy random sampling often is, which means that a large number of samples must be taken to obtain an accurate distribution



Distributions of Sizes

- Measurement of an average quantity is reasonably straightforward in stereology.
- Deduction of a 3D *size distribution* from the projection of that distribution on a section plane is much less straightforward (and still controversial in certain respects).
- Example: it is useful to be able to measure particle size and grain size distributions from plane sections (without resorting to serial sectioning).
- Assumptions about particle shape must be made.

True dimension(s) from measurements: examples

- Measure the number of objects per unit area, N_A . Also measure the mean number of intercepts per unit length, N_L .
- Assume that the objects are spheres: then their radius, $r = 8N_L/3\pi N_A$.
- Alternatively, assume that the objects are truncated octahedra, or tetrakaidcahedra: then their edge length, $a, = L_3/1.69 = 0.945 N_L/N_A$.

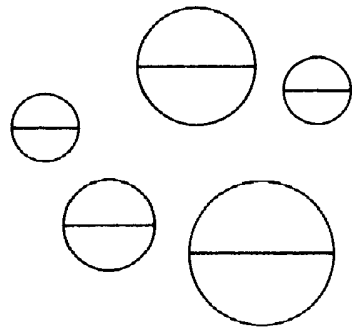
Volume of truncated octahedron

$$= 11.314a^3 = 9.548 (N_L/N_A)^3.$$

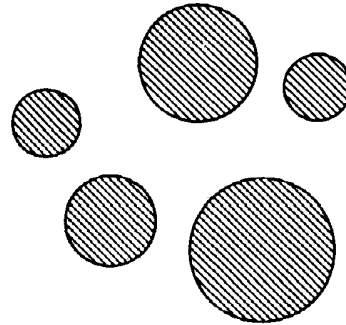
Equivalent spherical radius, based on $V_{sphere} = 4\pi/3 r^3$ and equating volumes:

$$r_{sphere} = 1.316 N_L/N_A.$$

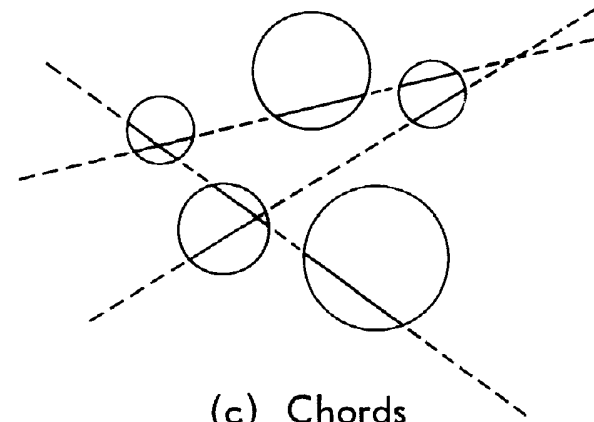
Measurements on Sections



(a) Diameters



(b) Areas



(c) Chords

- Areas are convenient if automated pixel counting available
- Either areas or diameters are a type of *planar sampling* involving measurement of circles (or some other basic shape)
- Chords are convenient for use of random test lines, which is a type of *linear sampling*: $n_L :=$ number of chords per unit length

Extraction of Size Distribution

- Whenever you section a distribution of particles of a finite size, the section plane is unlikely to cut at the maximum diameter (of, say, spherical particles).
- Therefore the *observed sizes* are always an *underestimate* of the *actual sizes*.
- Any method for estimating size distributions in effect starts with the largest size class and, based on some assumption about the shape and distribution of the particles, reduces the volume fraction of the next smallest size class by an amount that is proportional to the fraction of the current size class.

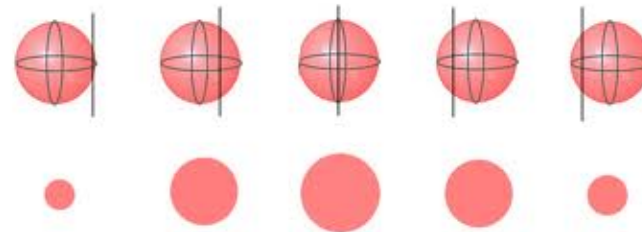
Size distributions from measurement

- Distribution of cross sections very different from 3D size distribution, as illustrated with monosize spheres.
- Measurement of chord lengths is most reliable, i.e. experimental frequency of $n_L(l)$ versus l .
- See articles by Lord & Willis; Cahn & Fullman; book by Saltykov
- $\langle D \rangle$:= mean diameter;
 $\sigma(D)$:= standard deviation
 N_V := number of particles (grains) per unit volume.

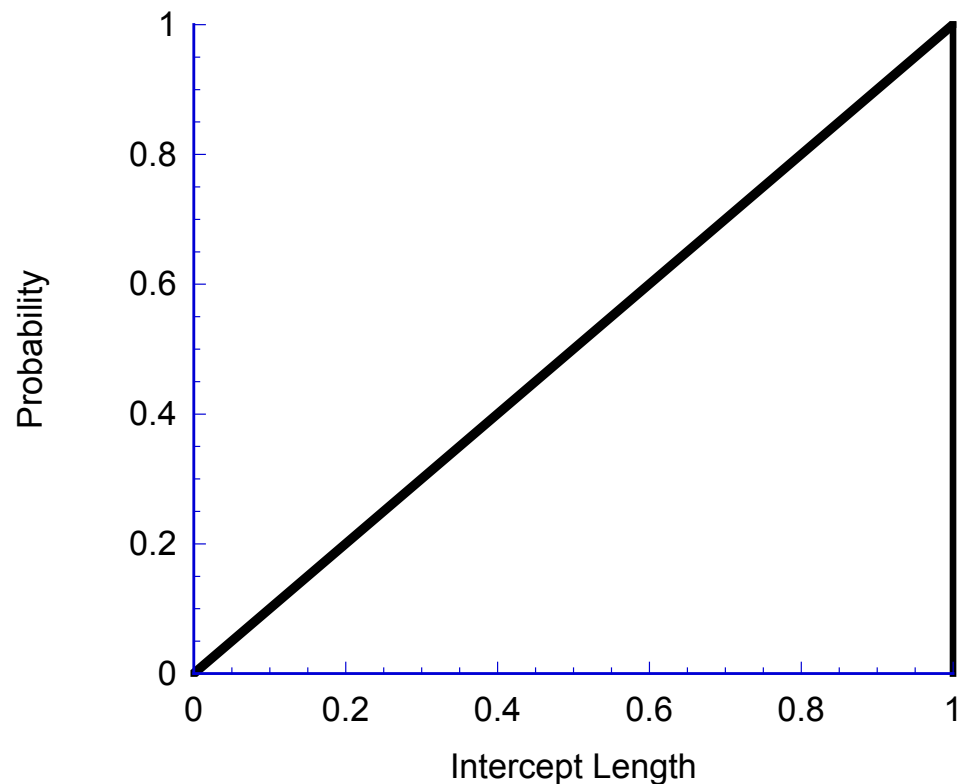
Chord lengths

<http://131.111.17.74/issue51/features/buckley/index.html>

- It happens that making random intersections of a test line (L_L) with a sphere leads to a rather simple probability distribution (in contrast to planar intercepts). In the graph, the value of the intercept length is normalized by the sphere diameter (effectively the largest observed length).



Intersection with Sphere



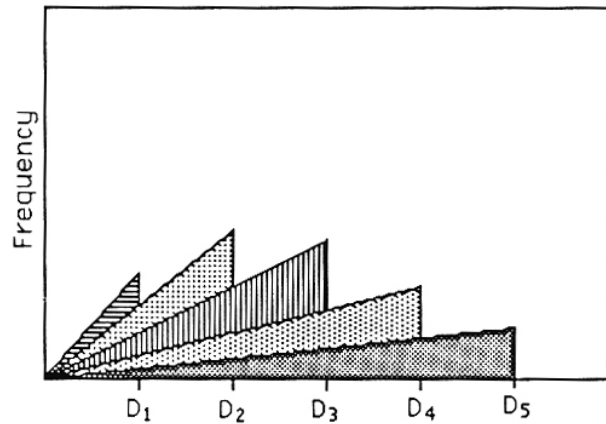


Figure 5. Superposition of intercept length curves for several sphere sizes.

- A consequence of the linear probability distribution is a particularly simple superposition for different sphere sizes, fig. 5 above.
- This also means that the sphere size distribution can be obtained purely graphically, fig. 6: one starts with the vertical intercept (RH axis) for the smallest size and subtracts off the intercept for the next largest size. Each intercept on the right-hand axis represents the value of the 3D sphere diameter density.
- Examples shown from Russ's *Practical Stereology* and is explained in more detail in Underwood's book. Note that in order to obtain the number of spheres, N_V , the vertical line on the RHS of the graph must be drawn at an intercept length = $2/\pi$ in the same units as the length measurement.

Multiple sphere sizes

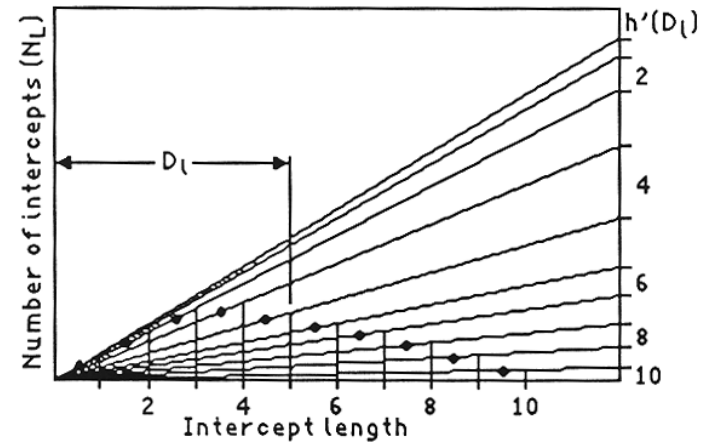


Figure 6. Extraction of the relative abundance of different sphere sizes from the measured histogram of intercept lengths.

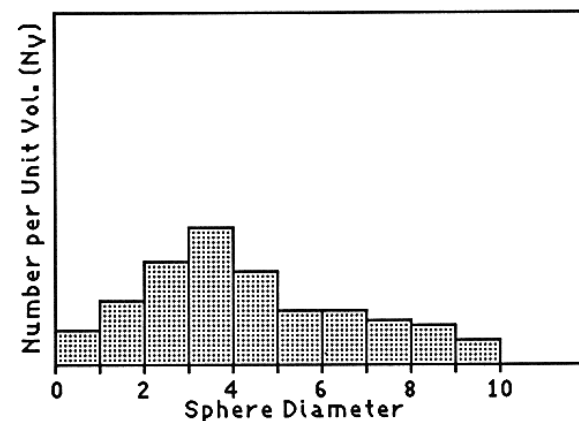


Figure 7. Plot of number of spheres versus diameter derived from the graph in Figure 6.

Number per unit volume

$$N_{V_j} = \frac{2}{\pi} \left\{ \frac{n_L(l)_j / \Delta l}{a_j} - \frac{n_L(l)_{j+1} / \Delta l}{a_{j+1}} \right\}$$

Current size class → (points to the first term in the numerator)

Next largest size class → (points to the second term in the numerator)

- Lord & Willis also described a numerical procedure, based on measurement of number of chords of a given length, which accomplishes the same procedure as the graphical procedure. One simply starts with the smallest size value and proceeds to progressively larger sizes. For the last bin (largest size), no subtraction is performed.
- Δl := size interval
 a_j := median of class intervals (can use average of the size, l , in the j^{th} interval)
- ASTM Bulletin **177** (1951) 56.

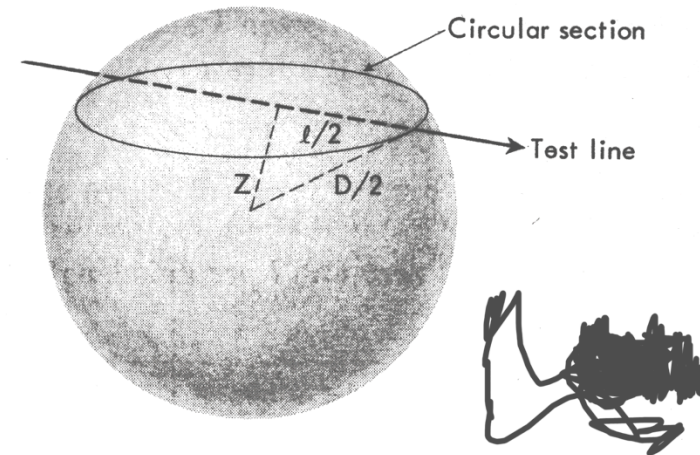
Number per unit volume: Cahn & Fullman

- Cahn & Fullman:
Trans AIME **206** (1956) 610.
 $D :=$ diameter = l
numerical differentiation of $n_L(l)$ required.
- Can be applied to systems other than spheres.

$$N_V(D) = \frac{2}{\pi l} \left\{ \frac{n_L(l)}{l} - \frac{dn_L}{dl} \right\}$$

Projections of Lines: Spektor

Spektor developed a method of extracting a distribution of sizes of spheres from chord length data (very similar result to Lord & Willis).



- $Z = \sqrt{[D/2]^2 - [l/2]^2}$
- Consider a cylindrical volume of length L , and radius Z centered on the test line. Volume is $\pi Z^2 L$ and the intercepted chord lengths vary between l and D .

Projections of Lines, contd.

- Number of chords per unit length of line:

$$n_L = \pi Z^2 N_V = \pi/4 (D^2 - l^2) N_V.$$
 where N_V is the no. of spheres per unit vol.
- For a dispersion of spheres, sum up:

$$\begin{aligned} (n_L)_l^{D_{\max}} &= \sum_{D=l}^{D=D_{\max}} \frac{\pi}{4} (D_j^2 - l^2) N_{V_j} \\ &= \frac{1}{4} \sum_{D=l}^{D=D_{\max}} \pi D_j^2 N_{V_j} - \frac{\pi l^2}{4} \sum_{D=l}^{D=D_{\max}} N_{V_j} \end{aligned}$$

Projections of Lines, contd.

- The terms on the RHS can be related to the total surface area, S_V , and the total no of particles per unit volume, N_V , respectively:

$$(n_L)_l^{D_{\max}} = \frac{1}{4}(S_V)_l^{D_{\max}} - \frac{\pi l^2}{4}(N_V)_l^{D_{\max}}$$

Differentiating this expression gives:

$$d(n_L)_l^{D_{\max}} = \frac{1}{4}d(S_V)_l^{D_{\max}} - \frac{\pi l^2}{4}d(N_V)_l^{D_{\max}} - \frac{\pi l}{2}(N_V)_l^{D_{\max}} dl$$

Projections of Lines, contd.

$$d(n_L)_l^{D_{\max}} = \frac{1}{4} d(S_V)_l^{D_{\max}} - \frac{\pi l^2}{4} d(N_V)_l^{D_{\max}} - \frac{\pi l}{2} (N_V)_l^{D_{\max}} dl$$

- The first two terms cancel out; also we note that $d(n_L)_l^{D_{\max}} = -d(n_L)_0^l$, so that we obtain:

$$d(n_L)_0^l = \frac{\pi}{2} (N_V)_l^{D_{\max}} l dl$$

$$(N_V)_l^{D_{\max}} = \frac{2}{\pi} \frac{1}{l} \frac{d(n_L)_0^l}{dl}$$

Projections of Lines, contd.

- In order to relate a distribution of the number of spheres per unit volume to the distribution of chord lengths, we can take differences: n_L is a number of chords over an interval of lengths, Δl is the length interval (essentially the Lord & Willis result).

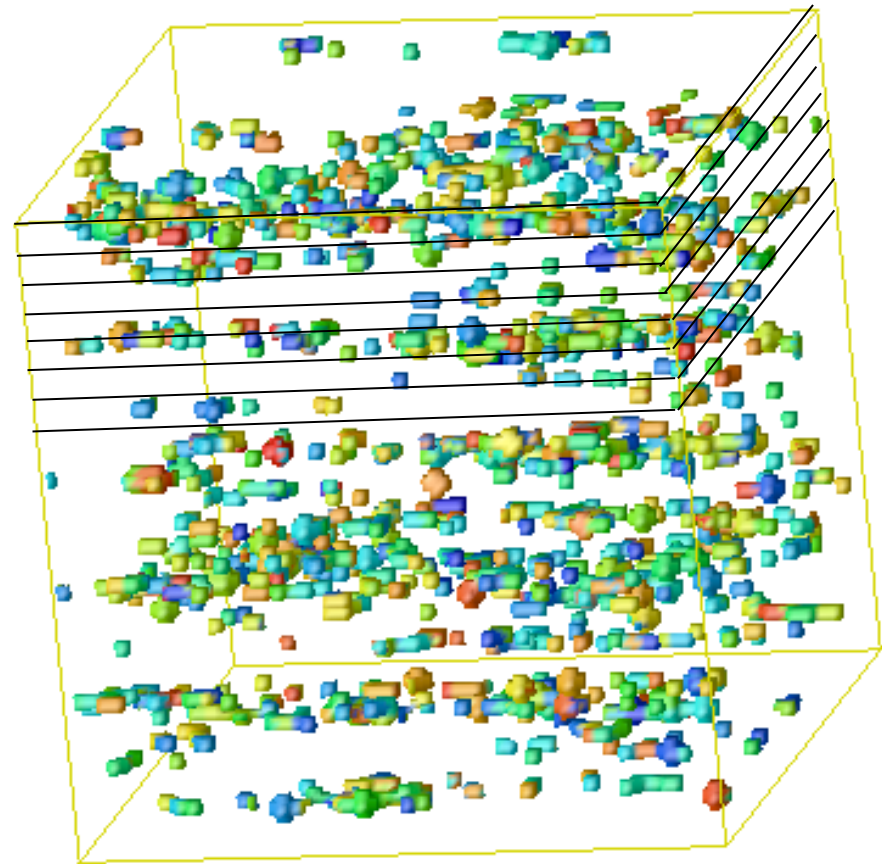
$$(N_V)_{l_1}^{l_2} = \frac{2}{\pi \Delta l} \left\{ \frac{(n_L)_{l_1 + \Delta l / 2}^{l_1 - \Delta l / 2}}{l_1} - \frac{(n_L)_{l_2 + \Delta l / 2}^{l_2 - \Delta l / 2}}{l_2} \right\}$$

Artificial Digital Particle Placement

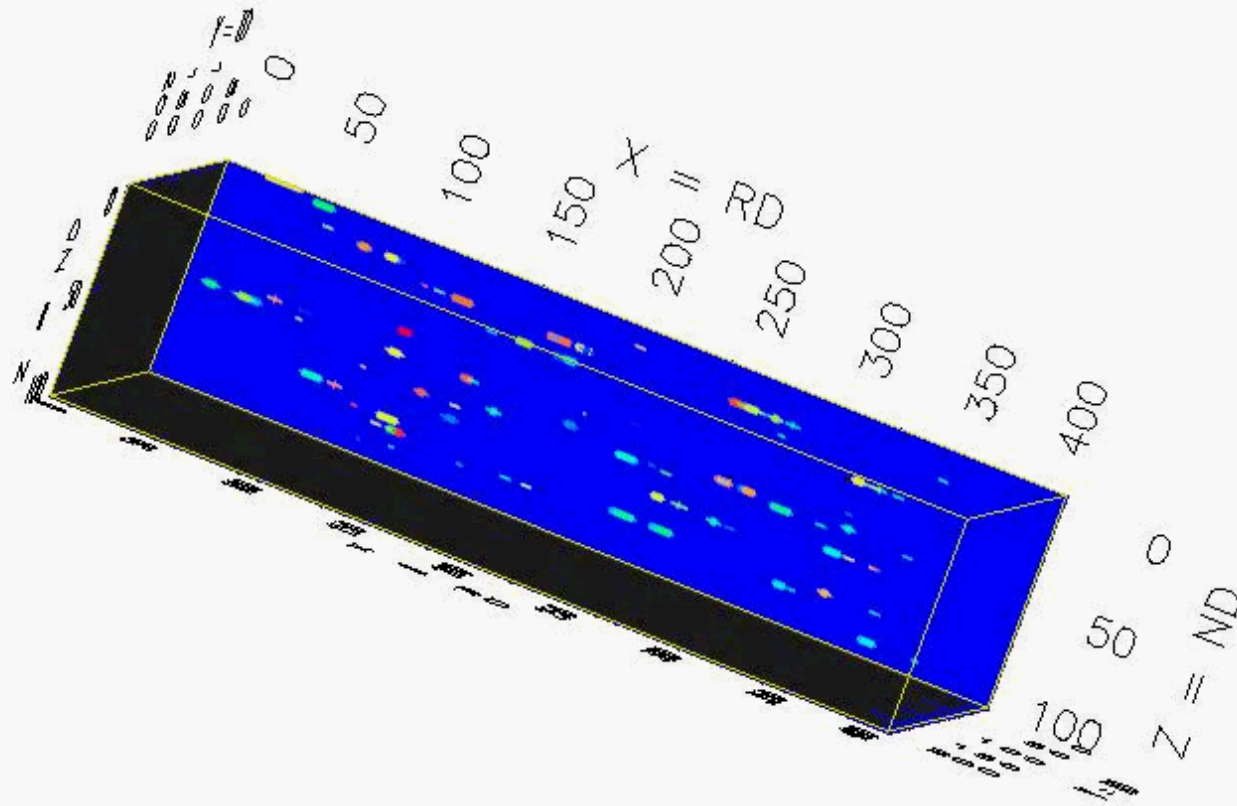
- To test the system of particle analysis and generation of a 3D digital microstructure of particles, an artificial 3D microstructure was generated using a Cellular Automaton on a 400x200x100 regular grid (equi-axed voxels or pixels). Particles were injected along lines to mimic the stringered distributions observed in 7075. The ellipsoid axes were constrained to be aligned with the domain axes (no rotations).
- This microstructure was then sectioned, as if it were a real material, the sections were analyzed, and a 3D particle set reconstructed.
- The main analytical tool employed in this technique is the (anisotropic) *pair correlation function* = *pcf* (to be explained in a later lecture).
- The length units for this calculation are pixels or voxels.
- See: “Three-Dimensional Characterization of Microstructure by Electron Back-Scatter Diffraction”, A.D. Rollett, S.-B. Lee, R. Campman, G.S. Rohrer, *Annual Review of Materials Research*, 37: 627-658 (2007).

Simulation Domain with Particles

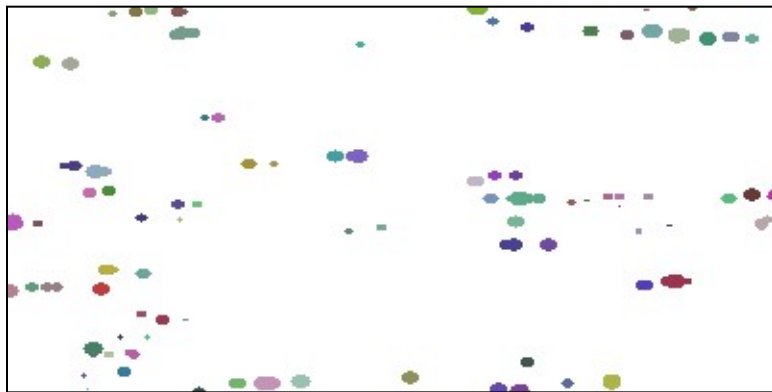
- Particles distributed randomly along lines to reproduce the effect of stringers.
- Series of slices through the domain used to calculate *pcfs*, just as for the experimental data.
- Averaged *pcfs* used with simulated annealing to match the measured *pair correlation functions*.



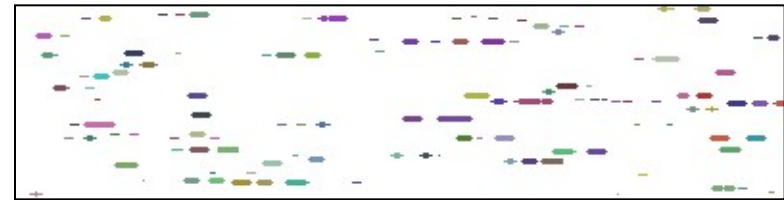
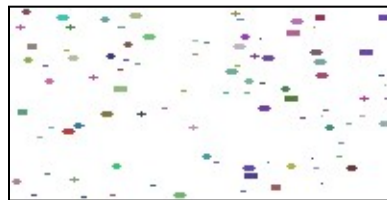
Sections through 3D Image



Generated Particle Structure: Sections



Ellipsoids were inserted into the domain with a constant aspect ratio of $a:b:c = 3:2:1$. The target correlation length was $0.07 \times 400 = 28$, with 10 particles per colony

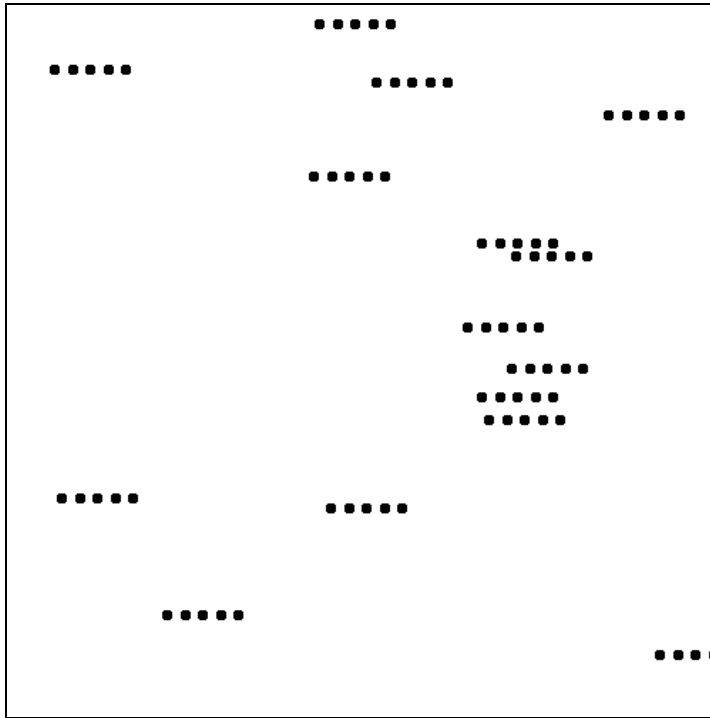


Rolling plane (Z) - Transverse (X) - Longitudinal (Y)

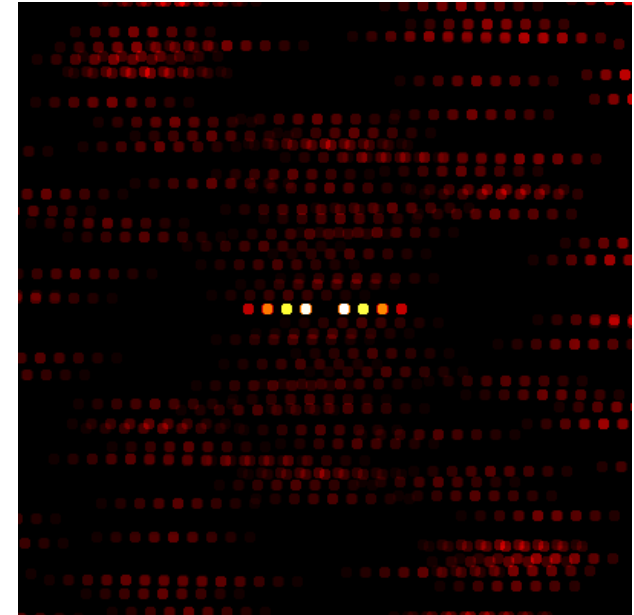
Pair Correlation Function: example

The PCF is the probability of finding neighboring particle at a certain distance & direction relative to the any particle.

$$PCF(x,y) = \sum_{i=1}^n P_i(x,y) / N_i(x,y)$$



Input (500X500)
Center of 1 dot to end of 5th dot is
53 pixels

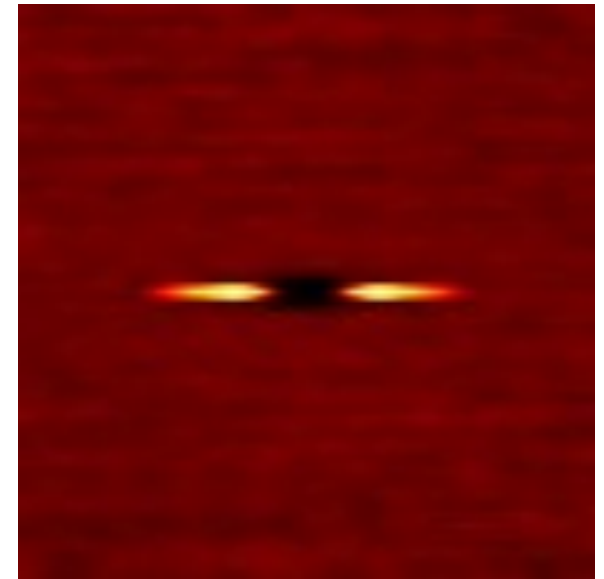
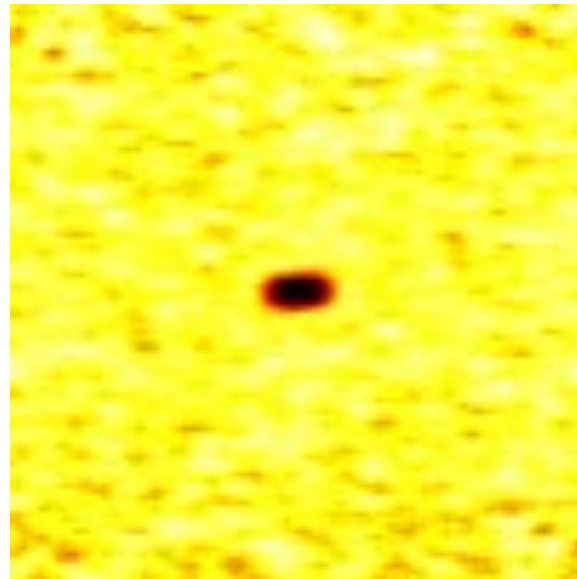
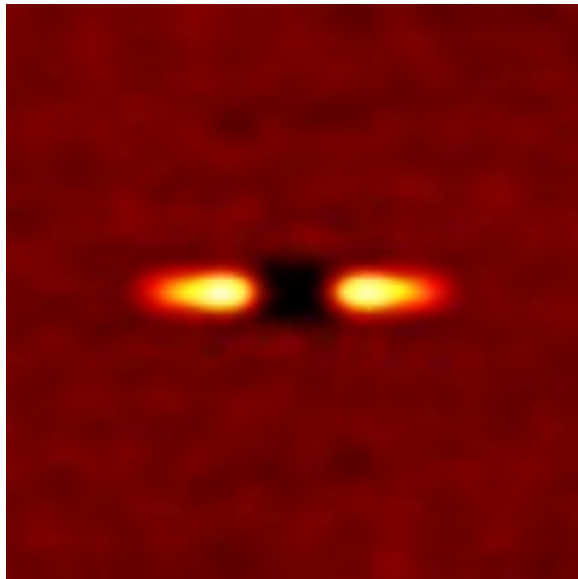


Output (401X401)
Center of image to end of red dot is
53 pixels

See also: Tewari, A.M Gokhale, J.E Spowart, D.B Miracle, Quantitative characterization of spatial clustering in three-dimensional microstructures using two-point correlation functions, *Acta Materialia*, Volume 52, Issue 2, 19 January 2004, Pages 307-319; also chemwiki.ucdavis.edu/Physical_Chemistry/Statistical_Mechanics/Advanced_Statistical_Mechanics/Distribution_Function_Theory/The_pair_correlation_function

Generated Particle Structure: PCFs

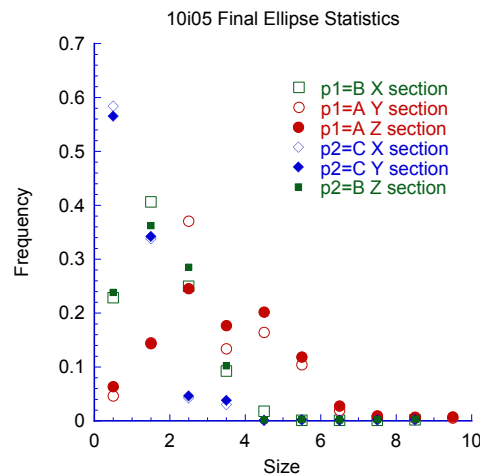
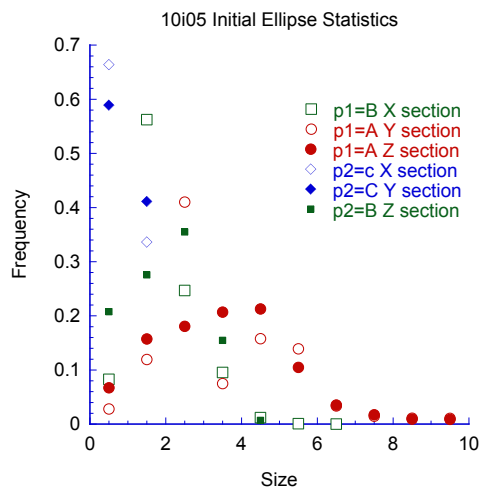
- Pair Correlation Functions were calculated on a 50x50 grid. The x-direction correlation length was ~ 29 pixels (half-length of the streak), in good agreement with the input.



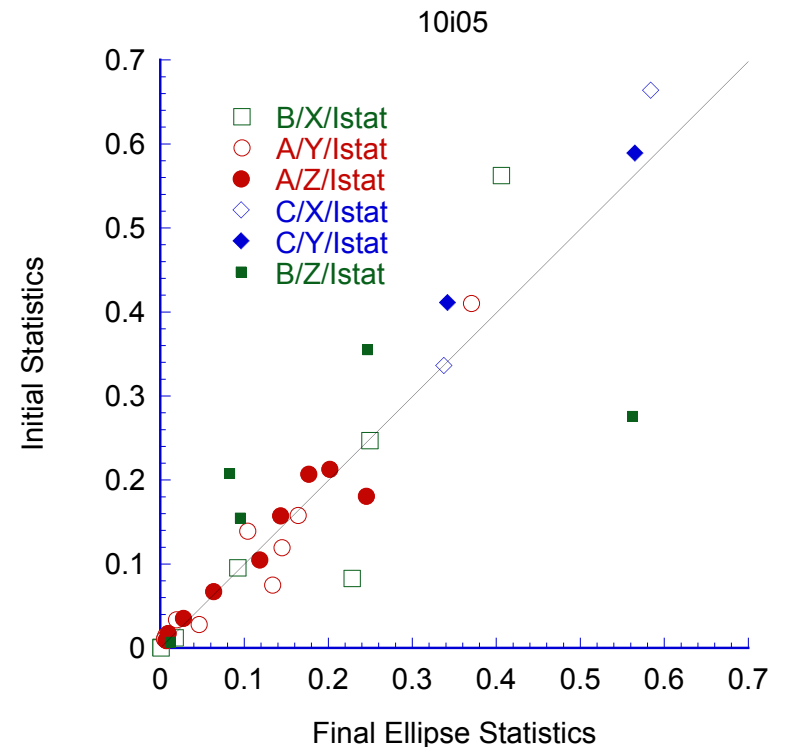
Rolling plane (Z) - Transverse (X) - Longitudinal (Y)

2D section size distributions

- A comparison of the shapes of ellipses shows reasonable agreement between the fitted set of ellipsoids and initial cross-section statistics (size distributions)



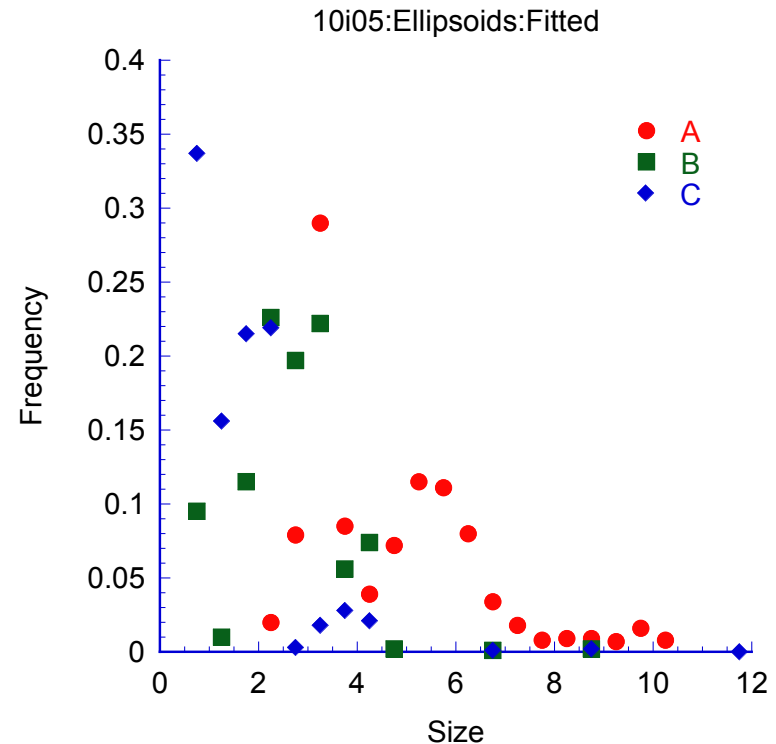
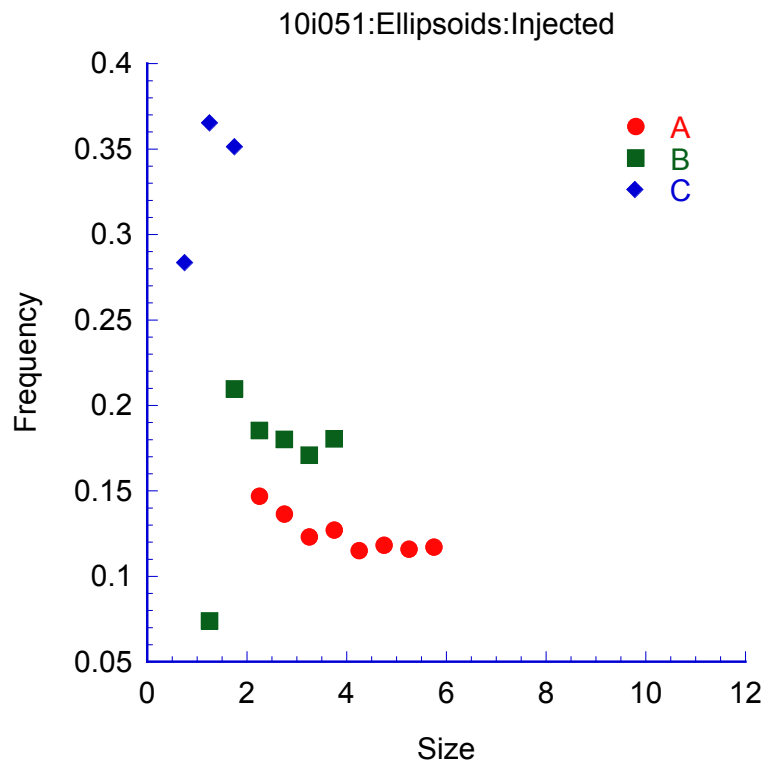
Initial vs. Final section distributions



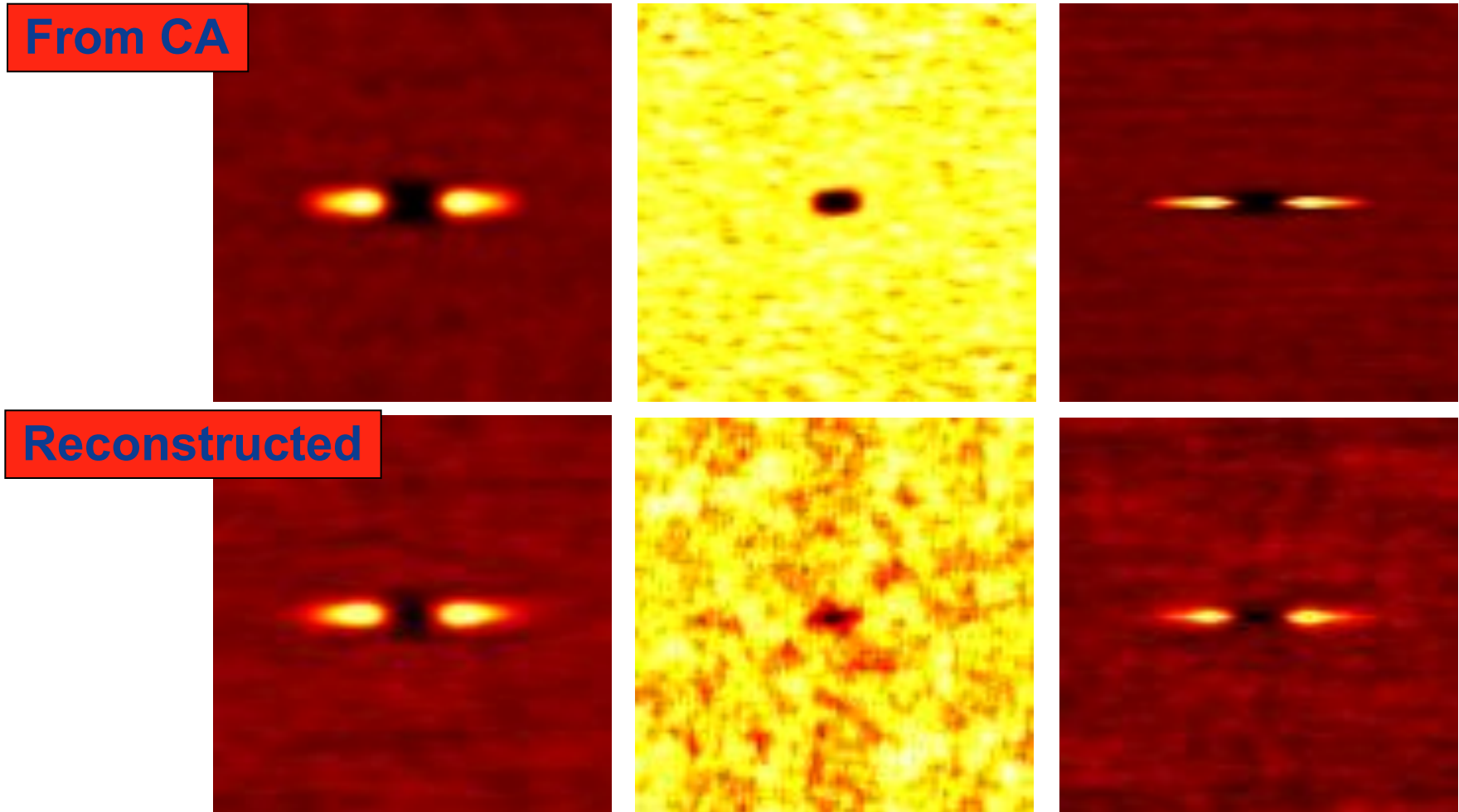
Cross-plot

Comparison of 3D Particle Shape, Size

- Comparison of the semi-axis size distributions between the set of 5765 ellipsoids in the generated structure and the 1,000 ellipsoids generated from the 2D section statistics shows reasonable agreement, with some “leakage” to larger sizes.
- Much larger data sets clearly needed to test the reconstruction of ellipsoidal particles

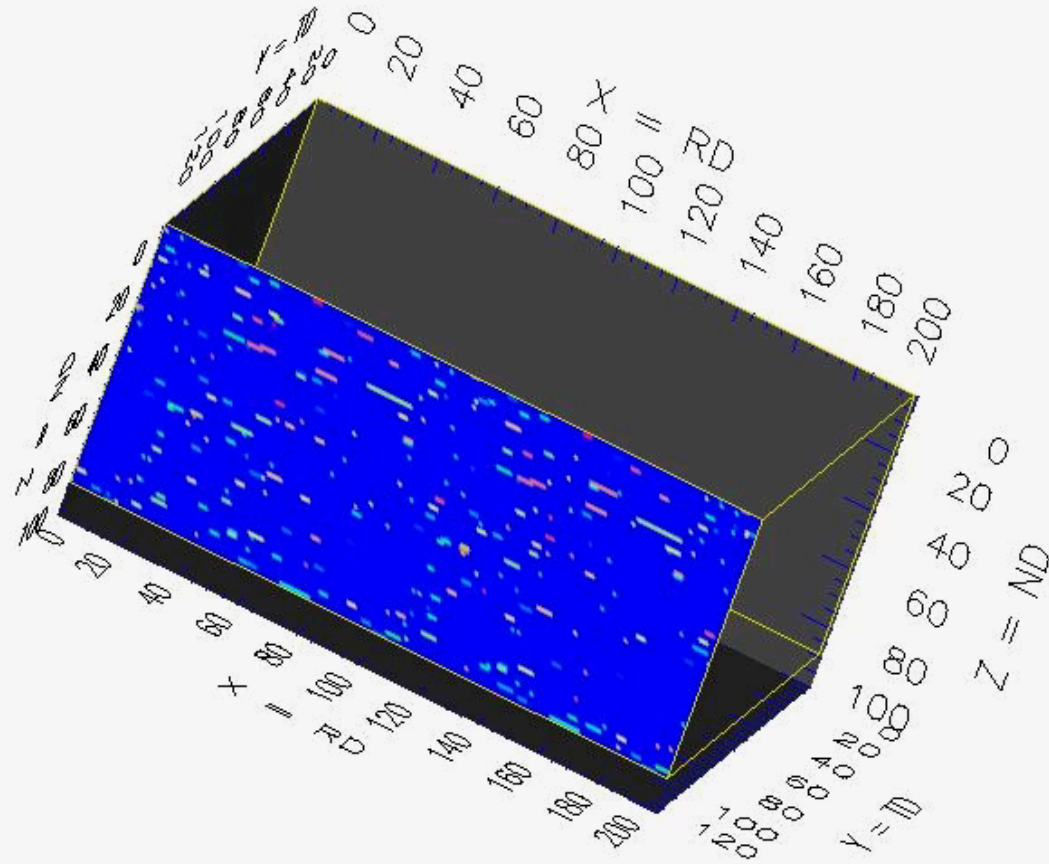


Comparison of PCFs for Original and Reconstructed Particle Distribution



Rolling plane (Z) - Transverse (X) - Longitudinal (Y)

Reconstructed 3D particle distribution



Geometric Relationships

- For each regular shape, whether sphere or tetrakaidecahedron, there is a set of analytical expressions that relate the dimensions of the object in 3D to its geometry in cross section.
- The following tables reproduced from Underwood summarize the available formulae.
- Note the difference between *projected* quantities and *mean intercept* quantities. Example: for spheres, the *projected area* is the equatorial area, πr^2 , whereas the *mean intercept area* is only $2/3 \pi r^2$.
- First slide is for bodies of revolution; second slide is for polyhedral shapes.

TABLE 4.1
Properties of particles with surfaces of revolution

	1.	2.	3.	4.
Particle shape	Symbol Volume	Surface	\bar{A}' Mean projected area	\bar{H}' Mean projected height
Sphere	$\frac{4}{3}\pi r^3$	$4\pi r^2$	πr^2	$2r$
Disk	$\pi r^2 t$	$2\pi r^2$	$\pi r^2/2$	$\pi r/2$
Cylinder	$\pi r^2 h$	$2\pi r(r + h)$	$\frac{\pi}{2}(r^2 + rh)$	$\frac{1}{2}(\pi r + h)$
Rod	$\pi r^2 l$	$2\pi r l$	$\frac{\pi}{2} r l$	$l/2$
Hemisphere	$\frac{2}{3}\pi r^3$	$3\pi r^2$	$\frac{3}{4}\pi r^2$	$(1 + \frac{\pi}{4})r$
Prolate spheroid (worked out for $b/a = \frac{1}{2}$)	$\frac{4}{3}\pi a b^2$ $= 8.38b^3$	$2\pi(b^2 + ab \frac{\sin^{-1}e}{e})$ $= 23.04b^{2*}$	$5.76b^2$	$2.76b$
Oblate spheroid (worked out for $b/a = \frac{1}{2}$)	$\frac{4}{3}\pi a^2 b$ $= 2.10a^3$	$2\pi(a^2 + \frac{b^2}{2e} \ln \frac{1+e}{1-e})$ $= 6.58a^{2*}$	$1.645a^2$	$1.70a$

* $e = \sqrt{1 - (b/a)^2}$; for $b/a = \frac{1}{2}$, $e = 0.866$.

5.	6.	7.	8.	Ref.	
\bar{A} Mean intercept area	\bar{L}_3 Mean intercept length	N_V Number per unit volume	r, t, h, l, a, b True dimensions of particles		
$\frac{2}{3}\pi r^2$	$4r/3 = 8N_L/3\pi N_A$	$\pi N_A^2/4N_L$	$r = \frac{2N_L}{\pi N_A} = \frac{3V_V}{4N_L}$	7, 26	Radius, r
$2rt$	$2t$	$2N_A^2/\pi N_L$	$r = N_L/N_A$ $t = V_V/2N_L$	7	Radius, r $r \gg t$ Thickness, t
$\frac{2\pi r^2 h}{\pi r + h}$	$\frac{2rh}{r + h}$	(if $r = h$) $0.732 \frac{N_A^2}{N_L}$ $= \frac{2N_A}{\pi r + h}$	$h = V_V/\pi N_V r^2$ $r = N_L/2N_A \pm \dagger$ $(\frac{N_L^2}{4N_L^2} - \frac{(\pi - 1)V_V}{2\pi N_A})^{1/2}$	7	$r \approx h$ Radius, r Height, h
$2\pi r^2$	$2r = 2N_L/\pi N_A$	$2N_A/l$	$r = N_L/\pi N_A$ $l = 2N_A/N_V$	7	Radius, r Length, l $l \gg r$
$\frac{8\pi r^2}{3(4 + \pi)}$	$8r/9$	$0.739 \frac{N_A^2}{N_L}$	$r = 1.522 \frac{N_L}{N_A}$	40	Radius, r
$3.025b^2$	$1.46b$	$0.756 \frac{N_A^2}{N_L}$	$b = V_V/1.46N_L$ $= 0.479N_L/N_A$	26, 28	$2b$ $2a$
$1.235a^2$	$1.278a$	$0.569 \frac{N_A^2}{N_L}$	$a = V_V/1.278N_L$ $= 1.034N_L/N_A$	26, 28	$2b$ $2a$

†For $r > h$, use + sign. For $r < h$ use - sign.

TABLE 4.2 Properties of regular polyhedrons

	1.	2.	3.	4.	5.
Symbol Particle shape	V Volume	S Surface	\bar{A}' Mean projected area	\bar{H}' Mean projected height	\bar{A} Mean intercept area
Cube	a^3	$6a^2$	$3a^2/2$	$3a/2$	$2a^2/3$
Rectangular parallelepiped	abc	$2(ab + bc + ca)$	$\frac{ab + bc + ca}{2}$	$\frac{a + b + c}{2}$	$\frac{2abc}{a + b + c}$
Square plate	a^2t	$2a^2 + 4at$	$a^2/2$	a	at
Square rod	a^2c	$2a^2 + 4ac$	ac	$c/2$	$2a^2$
Hexagonal prism	$\frac{3\sqrt{3}}{2}a^2c$	$3\sqrt{3}a^2 + 6ac$	$\frac{3\sqrt{3}a^2}{4} + \frac{3ac}{2}$	$\frac{3a + c}{2}$	$\frac{3\sqrt{3}a^2c}{3a + c}$
Tetrahedron	$0.1179a^3$	$1.732a^2$	$0.433a^2$	$0.9123a$	$0.129a^2$
Octahedron	$0.4714a^3$	$3.464a^2$	$0.866a^2$	$1.175a$	$0.4a^2$
Truncated octahedron	$11.314a^3$	$26.785a^2$	$6.696a^2$	$3.0a$	$3.77a^2$
Rhombic dodecahedron	$2a^3$	$6\sqrt{2}a^2$	$\frac{3\sqrt{2}}{2}a^2$	$2.0a$	a^2
Pentagonal dodecahedron	$7.663a^3$	$20.646a^2$	$5.161a^2$	$2.57a$	$2.97a^2$

	6.	7.	8.	
	L_3 Mean intercept length	N_V Number per unit volume	a, b, c, t True dimensions of particles	Ref.
	$2a/3$	$2N_A^2/3N_L$	$a = N_L/N_A$	24, 25 26, 30
	$\frac{2abc}{ab + bc + ca}$	(If $c = 3a, b = 2a$) $11N_A^2/18N_L$	(If $c = 3a, b = 2a$) $a = 6N_L/11N_A$	26, 30
	$2t$	$N_A^2/2N_L$	$a = 2N_L/N_A$	26, 30
	a	$4aN_A^2/cN_L$	$a = N_L/2N_A$	26, 30
	$\frac{2\sqrt{3}ac}{\sqrt{3}a + 2c}$	(If $c = a$) $0.7N_A^2/N_L$	(If $c = a$) $a = 0.715N_L/N_A$	24, 26 30
	$0.2725a$	$0.131N_A^2/N_L$	$a = 0.421N_L/N_A$	26, 30
	$0.545a$	$0.625N_A^2/N_L$	$a = 0.136N_L/N_A$	26, 30
	$1.69a$	$0.744N_A^2/N_L$	$a = 0.45N_L/N_A$	22, 24 25, 26 27, 30
	$\frac{4}{3\sqrt{2}}a$	$\frac{3\sqrt{2}}{8}N_A^2/N_L$	$a = 0.945N_L/N_A$	24, 26 30
	$1.485a$	$0.784N_A^2/N_L$	$a = 0.497N_L/N_A$	25, 26 31

Questions

1. Which set of quantities are equal to each other? **The point/line/area/volume fractions.**
2. How does Buffon's needle relate to the measurement of π ? **The intersection of a test line with a grid of parallel lines is related via $2L_A = \pi P_L$.**
3. Under what circumstances do we need to consider projected quantities rather than intercepts? **Projected areas, e.g., are appropriate when viewing a sample in transmission (e.g. TEM) and the feature is, say, blocking the illumination, as opposed to being viewed in cross-section.**
4. In general, do size distributions measured in 2D show larger or smaller means than their true 3D means? **Since 2D sections cut objects in all possible locations, the observed mean sizes are invariably smaller than the true sizes.**
5. Why are intercepts of grain boundaries with a circle sometimes used for measuring grain size? **Using a circle ensures that any bias in the grain morphology does not affect the results (of grain size measurement).**
6. Why are nearest neighbor distances smaller than the mean free path for a given volume fraction and size of particle? **In qualitative terms, a nearest neighbor distance is based on finding the nearest neighbor object (particle) regardless of direction, whereas a mean free path is measured in a straight line and so is unlikely to pass through the nearest neighbor (but rather a next-nearest neighbor). See also the Eqs.**

Summary

- Provided that certain assumptions about the way in which a section plane samples the 3D microstructure are valid, statistically based relationships exist between experimental measures of points, lines and areas and various corresponding 3D quantities.

Supplemental Slides

- Following slides contain useful information of various kinds.
 1. Definitions of statistical terms
 2. Measurement of area and circumference of spheres that are instantiated on a regular grid (voxelized).
 3. Verification of Stereological Relationships for (voxelized) objects on regular grids

1. Statistics: definitions

- *Population*: a well defined set of individual elements or measurements (e.g. areas of grains in a micrograph).
- *Parameter*: a numerical quantity that is defined for the population (e.g. mean grain area).
- *Sampling Units*: non-overlapping sets of elements. The union of all sampling units is equal to the population.
- *Sample*: a collection of sampling units taken from the population.
- *Estimate*: a numerical approximation of a population *parameter* calculated from a particular *sample* (e.g. mean grain area calculated from a subset of the areas).
- *Estimator*: a well-defined numerical method that describes how to calculate an *estimate* from a *sample*.
- *Uniform random sample*: a *sample* taken so that all *sampling units* within the population possess the same probability of falling within the *sample*.

Statistics: quantitative definitions

- Population mean of a quantity R :

$$\mu = E[R] = \frac{R_1 + R_2 + \dots + R_N}{N} = \frac{1}{N} \sum_{i=1}^N R_i$$

- Population variance, or mean square deviation:

$$\sigma^2 = Var(R) = E[(R - \mu)^2] = \frac{1}{N} \sum_{i=1}^N (R_i - \mu)^2$$

- Population standard deviation:

$$\sigma = SD(R) = \sqrt{Var(R)}$$

- Coefficient of variation:

$$CV(R) = \frac{\sigma}{\mu}$$

- Estimates:
sample mean:

$$\bar{R}_n = \frac{R_1 + R_2 + \dots + R_n}{n} = \frac{1}{n} \sum_{i=1}^n R_i$$

- Variance of sampling distribution:

$$Var(\bar{R}_n) = \frac{N - n}{N - 1} \frac{\sigma^2}{n} \approx \frac{\sigma^2}{n}, \quad \text{for large } n$$

Quantities in turquoise apply to the entire population;
Estimates from samples are in red.

Quantitative definitions, contd.

- *Standard Error of the sampling distribution (SE) and the Coefficient of Error (CE):*

$$SE(R) = \sqrt{\text{Var}(\bar{R}_n)}; \quad CE(\bar{R}_n) = \frac{SE(R)}{\mu}$$

- *Sample Variance, s, the square root of which is the sample standard deviation:*

$$s^2 = \frac{1}{n-1} \sum_{i=1}^n (R_i - \bar{R}_n)^2$$

- *Estimates of the coefficient of variation and the standard error:*

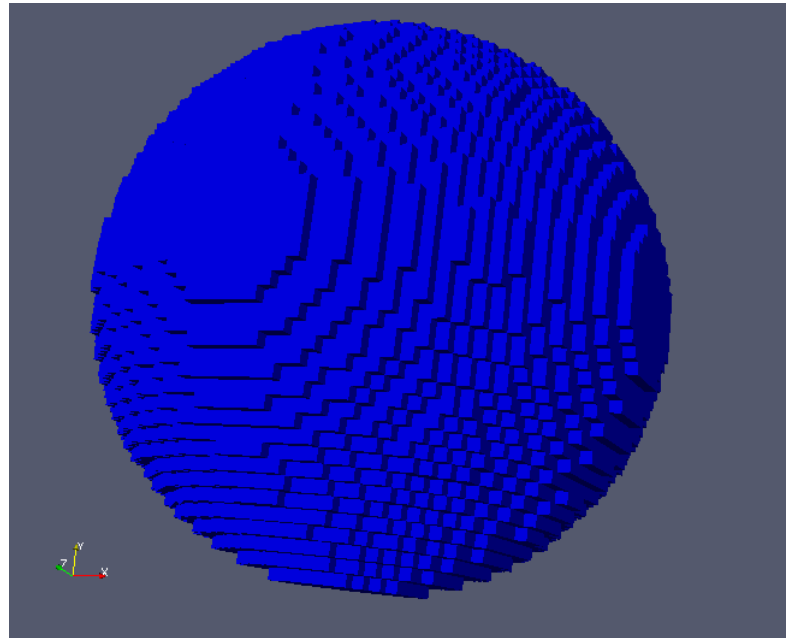
$${}_{est} SE(\bar{R}_n) = \frac{s}{\sqrt{n}}$$

$${}_{est} CE(\bar{R}_n) = \frac{{}_{est} SE(\bar{R}_n)}{\bar{R}_n} = \frac{s}{\sqrt{n} \bar{R}_n}$$

Note the sample size dependence of these estimates of the population quantities.

2. Sampling of Voxelized Sphere

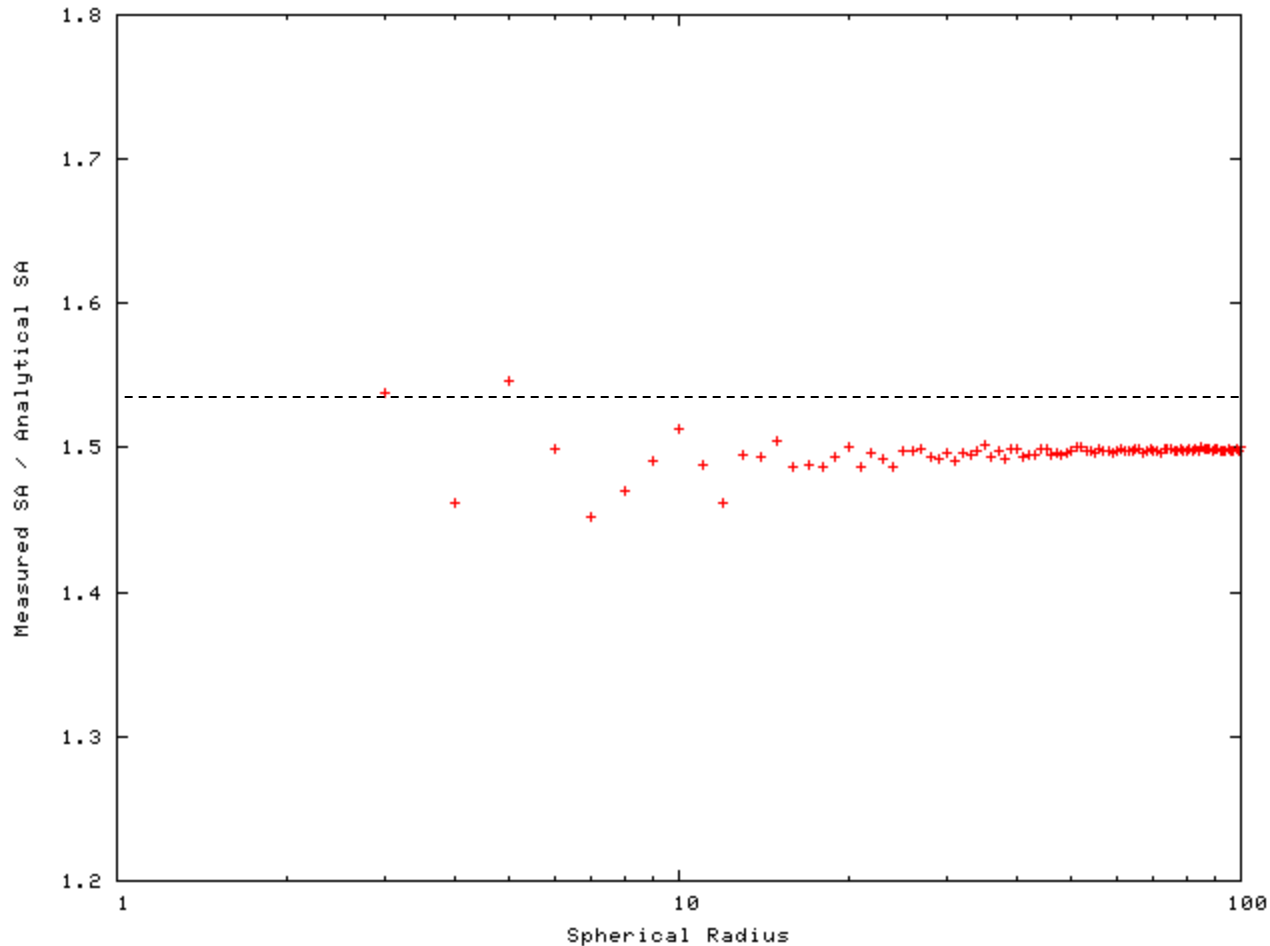
This exercise attempts to measure how accurately the surface area and circumference of a sphere can be measured on a rectilinear grid (i.e. the sphere has been voxelized) using a simple ledge counting method.



From the PhD
thesis work by
C.G. Roberts

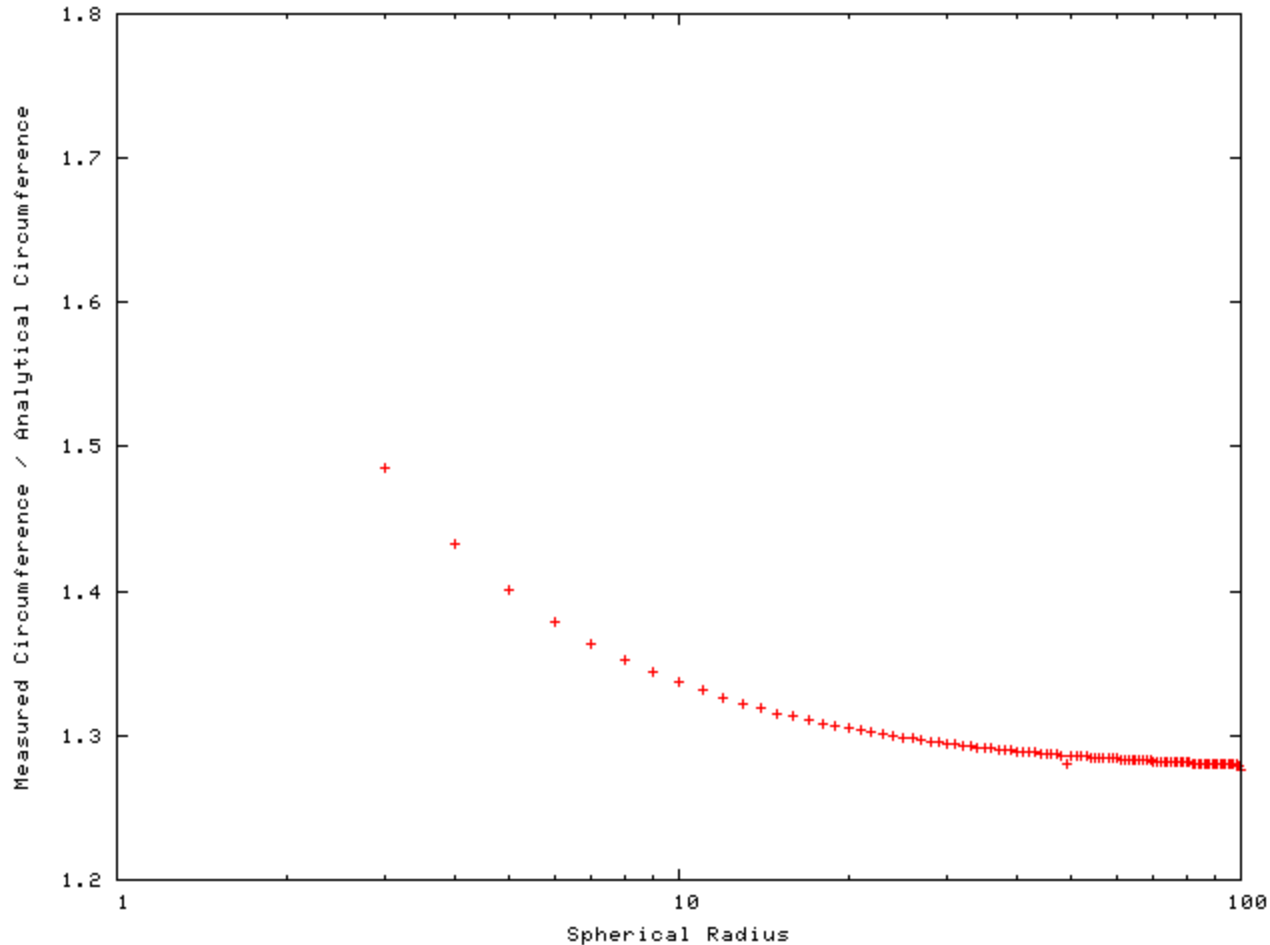
The figure above reveals the steps on the surface of a sphere with a radius equal to 50 pixels.

Surface Area of Voxelized Sphere



The surface area was measured and normalized by the analytical value ($4\pi r^2$). A constant ratio of 1.5 is obtained for radii greater than or equal to 3.

Circumference of Voxelized Sphere



A two-dimensional cross section was removed from the equatorial plane of the sphere and the circumference was measured and normalized by the analytical value ($2\pi r$).

Contrary to the surface area results, the ratio begins at a larger value for small radii and reaches an asymptotic value of 1.27 for radii greater than 30 pixels.

3. Verification of Stereological Relationships

Definition:

Stereology is the interpretation of three-dimensional structures based on two-dimensional observations. The relationships between lower and higher dimensionality are primarily mathematical in nature.

Practicality:

A majority of experimental investigations involve destructive evaluation of the specimen wherein the researcher measures the parameter of interest on a cross-sectional area; therefore, stereology provides the link between the planar and volumetric quantities.

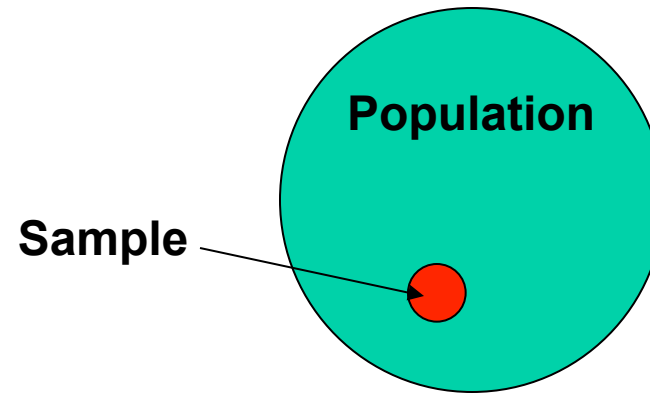
Quick Statistics Review

Population Mean = μ

Population Standard Deviation = σ

Sample Mean = \bar{x}

Sample Standard Deviation = s



Usually the population mean and error are unknown, but we would like to be able to estimate it using our sample subset.

$$\bar{x} = \sum_{i=1}^N \frac{x_i}{N}$$

$$s = \sqrt{\frac{\sum_{i=1}^N x_i^2 - \frac{\left(\sum_{i=1}^N x_i\right)^2}{N}}{N-1}}$$

The sample mean and standard deviation are the best estimates for the population mean and standard deviation. $\bar{x} \approx \mu$ $s \approx \sigma$

How good is the fit between the sample and population mean? In this case, we need to find the difference between \bar{x} and μ . This is known as the “standard error” and is given as:

$$s_e = \frac{\sigma}{\sqrt{N}} \approx \frac{s}{\sqrt{N}}$$

L_A Algorithm Verification

Using 1st nearest neighbors only (up, down, left, right)

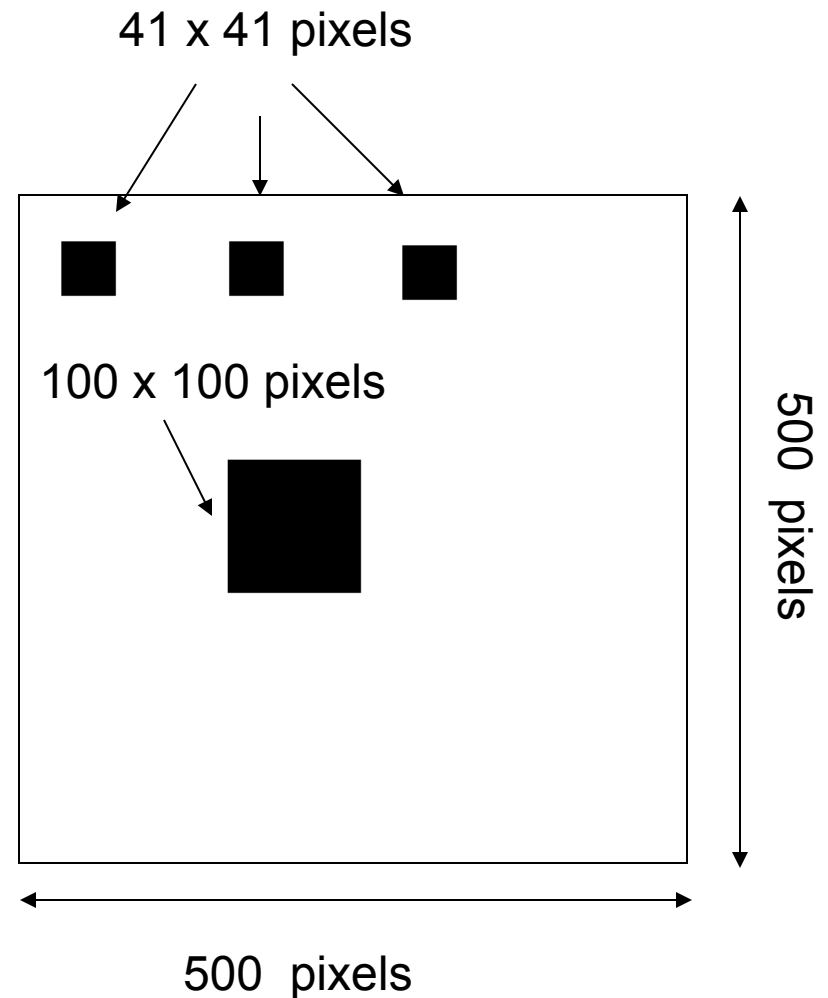
Particle-Matrix Trace = 3 boxes * (4 x 41) + 1 box * (4 * 100) = 892

Cross-sectional Area = 500 x 500

$$S_V = \frac{4}{\pi} L_A = \frac{4}{\pi} (0.3568) = 0.045429$$

Comparing this to the program output....

```
gs_methods.r90  misorient.r90  symmetry.r90
gs_methods.f90~ ntables.f90   symmetry.hp
croberts@mrsec017: ~/Fortran/Modules\ $ ./ssubv
image dimension 500 500
What is scaling factor? (pixels/um)
1
LsubA= 0.003568
SsubV= 0.00454291860308497
croberts@mrsec017: ~/Fortran/Modules\ $
```



Algorithm produces correct result

S_V Algorithm Verification

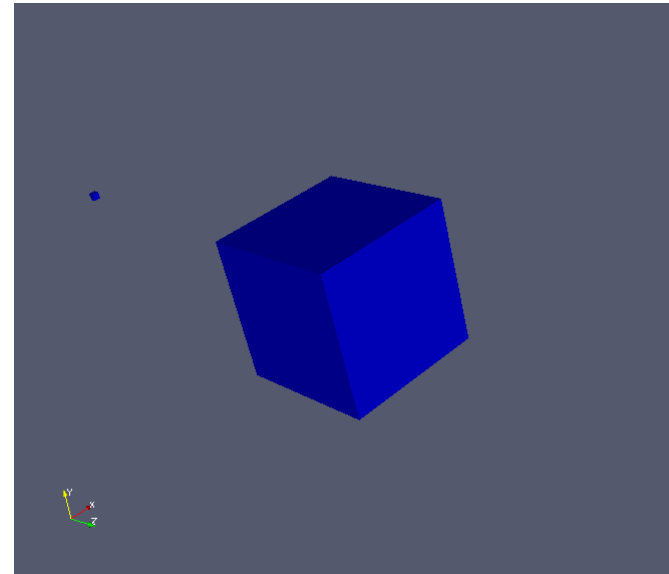
Two cubes inserted into a 100 x 100 x 100 box.

- a) Small Cube: $a=3$
SA = 6 faces * 9 pixels = 54 pixels
- b) Large Cube: $a=50$
SA = 6 faces * 2500 pixels = 15000

$$S_V = \frac{(15000 + 54)}{100^3} = 0.15054$$

Output from Fortran...

```
croberts@mrsec017: ~/Fortran/Modules\ $ ./a.out
seed array size= 4
new seed values= 1161789056 60210 3111 23280
Total SA= 15054
SsubV= 0.015054
Writing data to structure.modified
croberts@mrsec017: ~/Fortran/Modules\ $
```



Algorithm produces correct result

Particle Fractions

Estimation of volume fraction from cross-sectional areas is typically accomplished by using the following equation:

$$P_P^\alpha = L_L^\alpha = A_A^\alpha = V_V^\alpha$$

Since our images are a square grid, the point counting method is the easiest to implement for each dimensionality.

INPUT V_V	V_V	A_A	L_L
0.001	0.001026	$0.00097 \pm 4 \times 10^{-5}$	$0.00091 \pm 4 \times 10^{-5}$
0.01	0.010017	$0.00991 \pm 1.3 \times 10^{-4}$	$0.00851 \pm 1.4 \times 10^{-4}$
0.1	0.100008	$0.10030 \pm 4.4 \times 10^{-4}$	$0.06422 \pm 4.1 \times 10^{-4}$

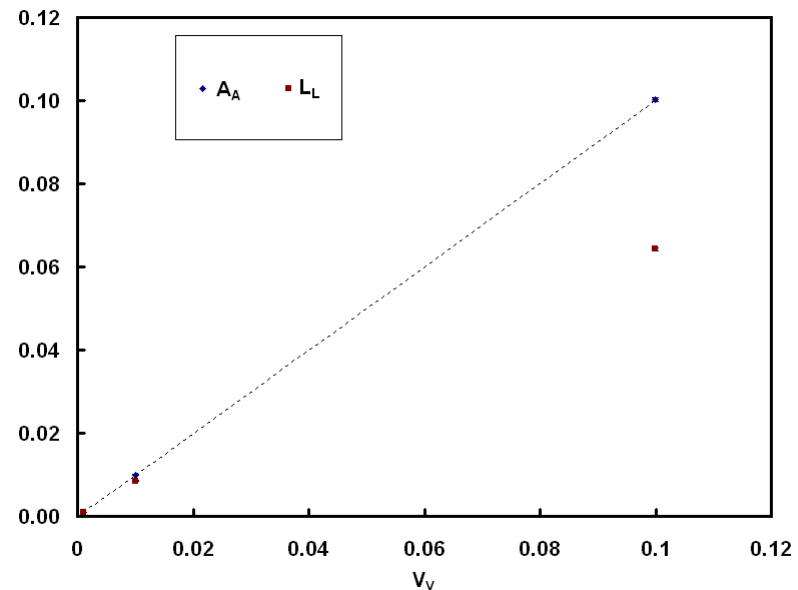
Particle Fractions, contd.

20 microstructures were generated and monosized ($a=3$) particles were randomly inserted into each 100^3 domain.

For any linear or area-based measurements: 10 sections were randomly selected from the x, y, and z planes (total of 30) and the area and linear fractions were measured.

600
measurements

At low volume fractions, the agreement among all three parameters is very close; however, the L_L parameter deviates significantly from the A_A and V_V values are larger particle fractions.

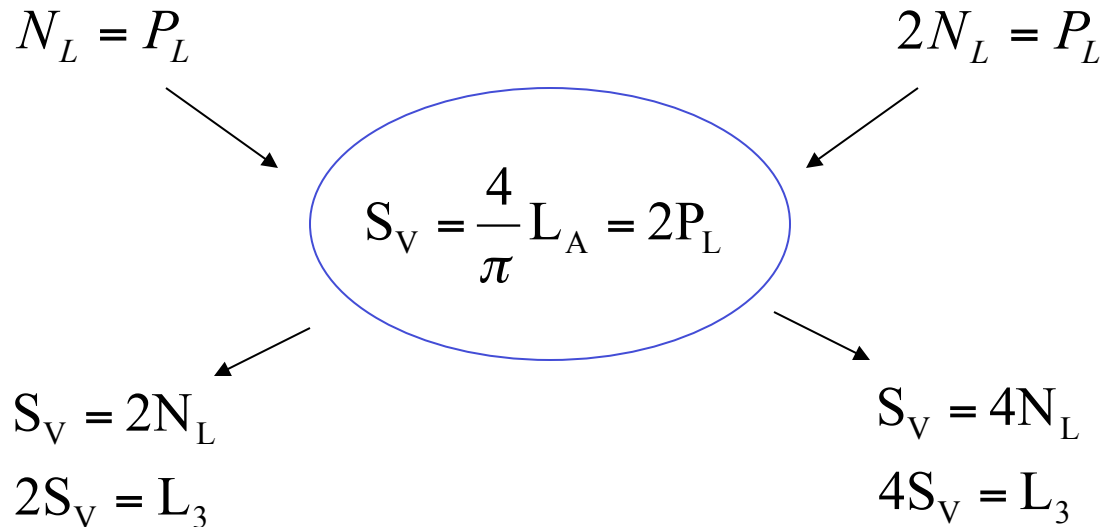


Recommendation: Use the area fraction (A_A) as a replacement for any equation or expression containing the linear fraction term.

Stereology: Grains vs. Particles

Space-filling structures

Dispersed Phase



When we analyze the grain characteristics in typical metal alloys, we will use the left-hand relationships; for particle statistics ($V_V \ll 1$), the right-hand equation is valid.

It is apparent that a factor of **2** is the difference between the two approaches, which can be attributed to the sharing of grain boundary area between **2** grains.

Stereology: L_A and S_V

Since most experimental studies involve two-dimensional statistical analyses, one inevitably will need to apply stereology to obtain a 3D parameter. Quantities highlighted with circles are easily measured on 2D planes.

TABLE 2.1
Relationship of measured (○) to calculated (□) quantities

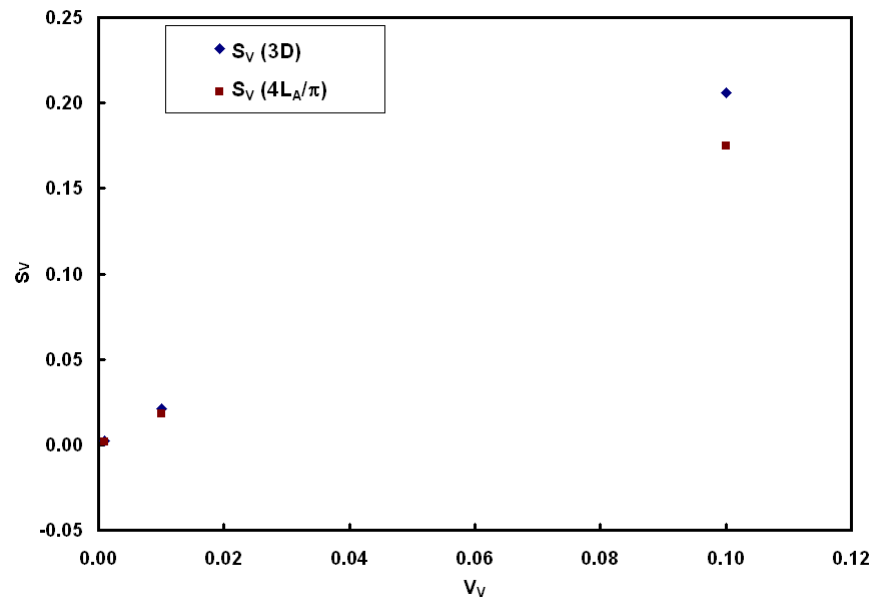
Microstructural feature	Dimensions of symbols (arbitrarily expressed in terms of millimeters)			
	mm ⁰	mm ⁻¹	mm ⁻²	mm ⁻³
Points	○ P_F	○ P_L → ○ P_A → □ P_V		
Lines	○ L_L	○ L_A → □ L_V → □ P_V		—
Surfaces	○ A_A	□ S_V → □ L_V → □ P_V		—
Volumes	□ V_V	—	—	—

We are interested in finding out how accurate the highlighted relationship is using computer generated three-dimensional structures.

$$S_V = \frac{4}{\pi} L_A$$

Stereology: L_A and S_V

Using the same particles microstructures, the two quantities S_V and L_A were measured.



At larger volume fractions, the stereological prediction appears to under-estimate the true surface area per unit volume.

Particle Shape Effect??

INPUT V_V	V_V	S_V (3D)	$S_V = (4/\pi)L_A$
0.001	0.00101	0.00216	$0.00176 \pm 7 \times 10^{-5}$
0.01	0.01001	0.02129	$0.01789 \pm 2.4 \times 10^{-4}$
0.1	0.10002	0.205722	$0.175084 \pm 7.6 \times 10^{-4}$

$$\left. \begin{array}{c} \text{ } \\ \text{ } \\ \text{ } \end{array} \right\} \frac{S_V}{V_V}$$

Is approximately constant

Mean Intercept Length

Another quantity of interest is the mean intercept length since it is an integral part of the relationship:

$$\frac{S_V}{4} = \frac{f}{\lambda} \quad \text{For particles ONLY}$$

Measured Intercept -- Based on our previous results on particle fractions, the mean intercept length can be obtained using:

$$\lambda_{\text{measured}} = \frac{V_V}{N_L} = \frac{A_A}{N_L} = \frac{L_L}{N_L}$$

Predicted Intercept – Knowledge of the 3D quantity, S_V , enables us to predict the mean intercept and compare it to the measured quantity. But be very careful about how λ is defined.

For dispersed particles.... $\lambda = \frac{V_V^\alpha}{N_L} = \frac{f}{N_L}$

$$\lambda_{\text{estimated}} = \frac{4V_V}{S_V} \quad \text{OR} \quad \lambda_{\text{estimated}} = \frac{4}{S_V}$$

Mean Intercept Length, contd.

How well does the 3D and 2D mean intercept measurements compare?

The constant ratio of S_V/V_V creates a situation where the relationship would imply that the mean intercept length must be a constant also.

The artificial condition of monosized particles may be responsible for this behavior.

$$\lambda_{\text{measured}} = \frac{L_L}{N_L} \qquad \lambda_{\text{predicted}} = \frac{4V_V}{S_V}$$

V_V	Measured	Predicted
0.001	3.25 ± 1.96	2
0.01	2.75 ± 0.83	2
0.1	2.74 ± 0.20	2.1

Conclusions

- The area fraction measurements provide an accurate estimate of the three-dimensional volume fraction for $V_V \leq 0.1$ while the line fraction significantly underestimates the true 3D quantity.
- Line trace per unit area under-estimates the surface area per unit volume for volume fractions above 1 percent.
- The predicted mean intercept length cannot be used as a substitute for the measurement of the mean intercept length.