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Kenton Hanson

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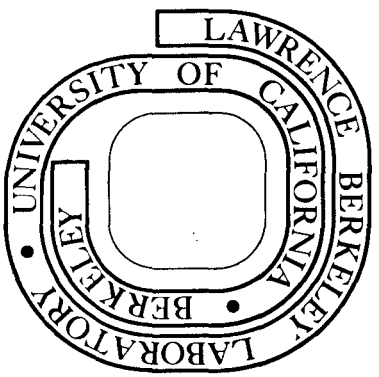

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DETERMINATION OF GRAIN DENSITY IN SPACE FILLING GEOMETRIES FROM MEASURABLE TWO-DIMENSIONAL PARAMETERS

by

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Abstract - A procedure for determining grain density, N_V, in space filling geometries from measurable two-dimensional parameters is presented. A variety of microstructure morphologies are considered and all tend to obey $N_V=(2.4150\sqrt{\bar{a}}-1.4552\bar{l})^{-3}$ where $\bar{a}$ = average grain area on a random two-dimensional section and $\bar{l}$ = the average intercept length of a random test line with grain surfaces. This relationship is verified by comparison with many computer simulated microstructures and various regular space filling geometries.

Theoretical considerations are presented suggesting why this basic relationship is expected. A practical method for applying this relationship is described. An extension of this procedure to non space filling geometries is discussed.

1. INTRODUCTION

Quantitative stereologists are concerned with the science of inferring three-dimensional microstructural features from two-dimension sections. While the expected surface density, S_V, and the expected edge density, L_V, in three-dimensional space are obtainable by measurements of their respective densities on two-dimensional sections, the density of grains (cells) in a space filling microstructure, N_V, has remained elusive. This paper presents a method for determining the three-dimensional grain density from easily measurable parameters on a two-dimensional section. This method is confirmed by comparison with a variety of computer generated space filling microstructures and three regular space filling geometries. Theoretical considerations are also presented suggesting why this basic relationship is expected. Finally, a practical method for estimating N_V is described and an extension of this procedure to non space filling geometries is discussed.

2. FUNDAMENTAL RELATIONSHIPS

Two fundamental equations relating the features of a three-dimensional microstructure (edges and surfaces) and their intersections with an arbitrary two-dimensional test section or test line are

$$L_V = 2P_A \tag{1}$$

$$S_V = 2N_L = \frac{2}{\bar{l}} \tag{2}$$

where

L_V= density of lines per unit volume

P_A= density of line intersections per unit area

S_V= density of surfaces per unit volume

$N_L = 1/\bar{l}$= density of surface intersections per unit length of the test line

The derivation of these relationships is described in detail in chapter 4 of reference 1. These relationships are valid if the edges and surfaces have no preferred orientation with respect to the test plane or line. However, if these features have preferential orientation, e. g. a simple cubic structure, care must be exercised to collect test data on a sufficient number of randomly orientated test planes and lines to ensure the validity of equations 1 and 2.

These relationships will be used to determine the densities of surfaces S_V and edges L_V in three-dimensional space from two-dimensional sections. Our analysis will be concerned with space filling geometries where all surfaces are boundaries between two grains, and where edges are intersections of grain surfaces. Figure 1 shows some examples of two-dimensional sections of three-dimensional microstructures. Notice, that three-dimensional surfaces and edges appear as lines and points respectively in the two-dimensional section, i. e., their dimensionality decreases by one. If, on a two-dimensional test plane, all points are the intersections of three and only three lines (see figure 2) then the density of points on the test plane, P_A, is

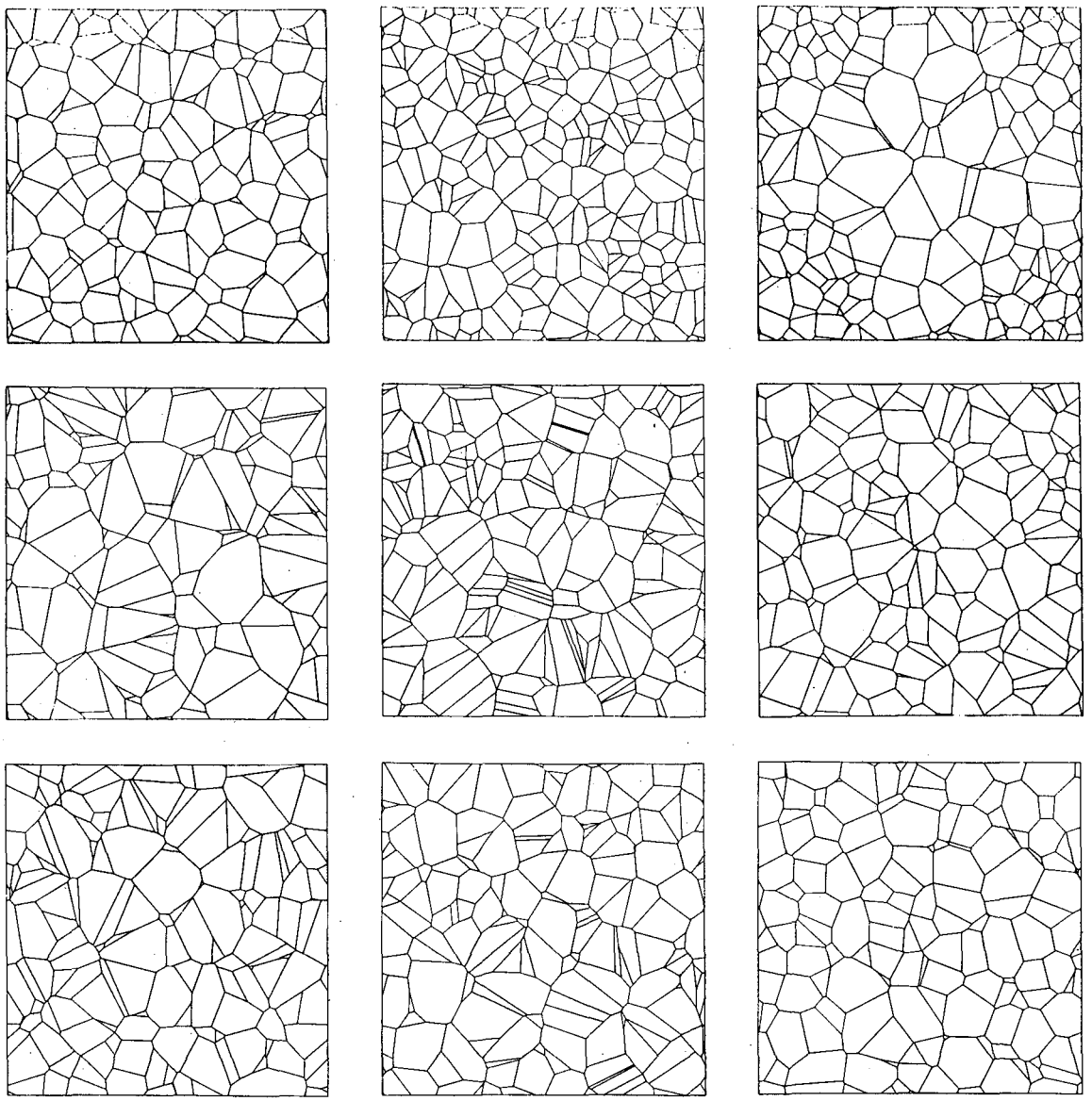

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FIG. 1

estimated by $2/\bar{a}$, where $\bar{a}$ ($= 1/N_A$) is the average area per grain in a two-dimensional section. This relationship can be inferred from the Euler relationship and combined with equation 1 yields

$$L_V=2P_A=4N_A=\frac{4}{\bar{a}} \tag{3}$$

where

$$N_A=\frac{1}{\bar{a}}= \text{Number of grains per unit area}$$

Thus, L_V can be inferred from $\bar{a}$.

If the average grain size, b (where b^3 = average volume), is known and the average edge length per grain, $e\ b$ (e is a numerical factor), is known, then

$$L_V=\frac{1}{3}N_V e\ b \tag{4}$$

where

$$N_V=\frac{1}{b^3}= \text{the number of grains per unit volume}$$

The $\frac{1}{3}$ factor is necessary since each edge is shared by three grains. Solving for b in equation 4 yields

$$b_{L_V}=\sqrt{\frac{e}{3L_V}}=\frac{k_{L_V}}{\sqrt{N_A}}=k_{L_V}\sqrt{\bar{a}} \tag{5}$$

where

$$k_{L_V}=\sqrt{\frac{e}{12}}$$

Similarly, if the average surface area per grain, $s\ b^2$ (s is a numerical factor), is known, then

$$S_V=\frac{1}{2}N_V s\ b^2=\frac{1}{2}\frac{1}{b^3}s\ b^2$$

and thus,

$$b_{S_V}=\frac{s}{2S_V}=\frac{k_{S_V}}{N_L}=k_{S_V}\bar{l} \tag{6}$$

where

$$k_{S_V}=\frac{s}{4}$$

The subscripts L_V and S_V denote the source of the calculation. e and s are strongly shape dependent and are difficult to determine for real microstructures.

In a classic paper J. L. Meijering [2] calculates e and s exactly for microstructures created by random nucleation and growth. In his "cell model" nucleation sites are random in space and each volume element belongs to that grain whose nucleus is nearest. The resulting microstructure consists of space filling irregular polyhedrons whose surfaces are points equidistant from two nucleation sites but no closer to any other nucleus. In the "cell model"

$$e=\frac{(4\pi)^{\frac{5}{3}}3^{\frac{1}{3}}}{5}\Gamma(\frac{4}{3})\approx 17.4956$$

$$s=(\frac{256\pi}{3})^{\frac{1}{3}}\Gamma(\frac{5}{3})\approx 5.82087$$

The corresponding values of k_{S_V} and k_{L_V} are 1.4552 and 1.2075 respectively. Therefore, given a microstructure defined by Meijering's "cell model" we can calculate the average grain density by either measuring $\bar{a}$ or $\bar{l}$ on a two-dimensional test section. Although other microstructures will have different values for k_{S_V} and k_{L_V}, we shall use Meijering's values extensively since they are exactly derivable for a non-regular microstructure. An inherent difficulty in the experimental analysis of real microstructures is the determination of b. To overcome this difficulty a variety of non-regular microstructures are computer modeled where b is precisely known. The computer modeling technique and results follow.

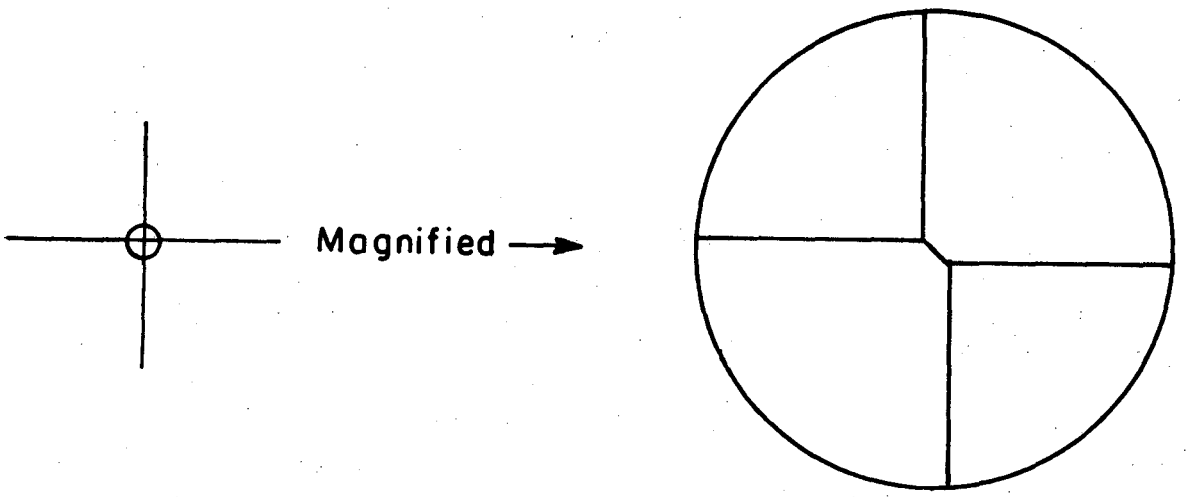

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FIG. 2

3. COMPUTER MODELING PROCEDURE

A Meijering "cell model" is computer simulated by first generating random nucleation sites in a three-dimensional unit cube with periodic boundary conditions. Then sophisticated computer techniques are used to construct a connected graph where nodes are corners of the resulting irregular polyhedrons and paths are polyhedron edges that connect corners. Nodes are locations in space equidistant from four nucleation sites but no closer to any other nucleus. Edges are lines equidistant from three nucleation sites but no closer to any other nucleus. Once the connected graph is constructed all three-dimensional information including volume, surface and edge distributions are calculated precisely. Two-dimensional test planes are chosen (see figure 1) and the corresponding two-dimensional features are extracted. Lines are scribed onto the two-dimensional section to determine N_L. The simulation code samples 100 evenly spaced planes and 100 scribed lines on each plane. The computer model is verified by testing all of the previously described fundamental relationships and Meijering's constants for the cell model. The first two cases in table 1 show the two and three-dimensional agreement for a simulated Meijering "cell model." The first half of table 1 is calculated from two-dimensional data while the second half is calculated from three-dimensional data. The agreement between columns 3 and 6 simply verifies equation 1 and similarly the agreement between columns 4 and 7 verifies equation 2. All results in table 1 are expressed in units of b.

The microstructure modeling technique described does not require random nucleation sites. The simulation can be used to study a variety of microstructures created by preferred nucleation sites and isotropic growth until impingement. Transformed microstructures are generated by chosing a density of preferred sites (corners, edges or surfaces) from a previous microstructure. Figure 1 shows examples of a "cell model" and various transformed microstructures. Any criteria can be used to select nucleation sites, enabling examination of the effects of preferred nucleation sites on microstructure morphologies.

The computer simulation technique described has been used for a variety of

Case	N_t	Two-dimensional data			Three-dimensional data		
		b_{L_V}	b_{S_V}	$2b_{L_V}-b_{S_V}$	b_{L_V}	b_{S_V}	$2b_{L_V}-b_{S_V}$
1	1011				.9998	.9996	1.0001
2	1000	.9999	1.0012	.9986	.9997	.9992	1.0002
3	671	.9608	.9322	.9894	.9614	.9342	.9887
4	1000	.9505	.8951	1.0059	.9533	.9065	1.0002
5	1000	.9819	.9641	.9997	.9860	.9732	.9987
6	1000	.9699	.9471	.9927	.9659	.9372	.9946
7	1000	.9835	.9491	1.0178	.9775	.9543	1.0006
8	1000	.9902	.9800	1.0004	.9945	.9885	1.0006
9	510				.9621	.9369	.9873
10	1011				.9577	.9244	.9910
11	2732				.9591	.9287	.9894
12	2698				.9628	.9356	.9900
13	2423				.9867	.9808	.9926
14	2839				.9849	.9806	.9893
15	256				1.0330	1.0786	.9874
16	500				1.0314	1.0736	.9891
17	500				1.0302	1.0706	.9898
18	500				1.0301	1.0698	.9905
19	500				1.0316	1.0739	.9892
20	500				1.0327	1.0766	.9887
21	1000	.8538	.8051	.9025	.9304	.8793	.9815
22	1000	1.0565	1.1216	.9914	1.0550	1.1197	.9902
23					1.0906	1.1318	1.0494
24					.9859	.9701	1.0017
25					1.0445	1.0952	.9939
26					1.0299	1.0889	.9708

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scientific applications. In the interest of completeness all results are reported, including those obtained before the two-dimensional subroutine was written. N_V is the number of cells per unit volume of the final microstructure. $b=N_V^{-1/3}$, was used to normalize table 1. Cases 1 and 2 were created by chosing random nucleation sites. Cases 3 through 14 were created by first generating microstructures from random nucleation sites and then creating a new microstructure by randomly nucleating on either surfaces, edges, or corners. Cases 13 and 14 were created by a double transformation involving nucleation on all corners of the initially randomly nucleated "cellular" microstructure and then nucleating on all corners of the resultant microstructure. Cases 3, 9, 10, 11 and 12 were created by nucleating on all corners of an initially random microstructure. Case 6 was created by nucleating on 1000 random corners of an initially randomly nucleated microstructure containing 200 cells (not all corners were nucleated.) Cases 4 and 5 were created by nucleation of 1000 random sites on edges and surfaces respectively of an initial microstructure containing 100 randomly nucleated cells. Cases 7 and 8 are identical to 4 and 5 respectively, except that the original microstructure contained 200 cells. In cases 3 through 14 each transformation consumes the previous microstructure.

Cases 15 through 20 were created by considering high densities of hard spheres in various semi-ordered and random configurations. The centers of these spheres were used as nucleation sites for the resultant microstructures. Case 21 was created by nucleating 1000 random sites on five equally space planes. The two-dimensional planes represent a random nucleation in two-dimensions. These planes were used in table 1 and therefore do not represent random sections. However, the corresponding three-dimensional results are surprisingly good and would be expected if randomly oriented sections had been examined.

Case 22 was generated by chosing x, y and z nucleation coordinates independently with a density proportional to their distance from the center of their respective axes, i. e., cell density decreases near the center. Case 23 is for a "Johnson Mehl" microstructure where nucleation occurs randomly in time and space [2]. Cases 24, 25 and 26 are regular space filling geometries created by nucleation at simple cubic, body center cubic and face center cubic lattice

sites respectively. The cell shape for case 25 is a truncated octahedron ($e=2^{2/3}6\sqrt{3}$ and $s=2^{5/6}3$). The cell shape for case 26 is a rhombic dodecahedron ($e=2^{5/6}9$ and $s=2^{-1/3}(9/2+3\sqrt{3})$).

4. RESULTS

Table 1 containes the estimated values (equations 5 and 6) for b_{L_V} and b_{S_V} using Meijering's "cell model" constants (e = 17.4956 and s = 5.82087) for a variety of microstructures. The error in these estimated values reflect the microstructural dependence of k_{L_V} and k_{S_V}. However, the error in b_{S_V} is approximately twice that of b_{L_V} suggesting the following relationship,

$$b=2b_{L_V}-b_{S_V} \tag{7}$$

$$=2k_{L_V}\sqrt{\bar{a}}-k_{S_V}\bar{l}$$

$$=2\frac{2k_{L_V}}{\sqrt{L_V}}-\frac{k_{S_V}}{S_V}$$

Columns 5 and 8 in table 1 were calculated from equation 7. Although the grain size predicted by either equation 5 or 6 independently vary significantly, equation 7 yields a value usually within 1% of the actual value. Therefore, the linear extrapolation suggested by equation 7 is relatively insensitive to microstructure morphologies.

Furthermore, some well known space filling geometries are examined with respect to equation 7. A simple cubic structure has a surface area per grain of $6b^2$ and an average edge length of $18b$ if each edge is shared by only three cubes. This can be visualized by perturbing each edge slightly such that it is really two edges (see figure 2). This is especially convenient since a measure of L_V for a cubic structure using $\bar{a}$ (equation 3) on random test planes would predict $18b$. Similarly, for a cube-octahedron the surface area is $5.315b^2$ and the edge density is

16.04*b*. Table 1 shows the estimated values of b for these structures using equation 7. In both cases the predicted value of b is surprisingly good considering the constants for equation 7 are derived for a non-regular microstructure. The errors, although small, indicate that equation 7 is not an exact relationship. However, equation 7 provides a very accurate estimate of grain density, N_V, for all morphologies considered.

5. THEORY

Many space filling microstructures have been examined and all tend to obey the relationship suggested by equation 7. To analyze this relationship one must consider how S_V and L_V will vary when the geometry of a microstructure is altered. Given a microstructure, it can be transformed to another microstructure by moving and distorting grain boundaries. A completely general accounting of grain boundary distortion appears mathematically intractable. However, we consider two mathematically calculable distortions, compression and elongation.

Consider a randomly oriented microstructure of known grain density ($b=b_{S_V}=b_{L_V}$). If this system is compressed at constant volume (e.g. the rolling of metals), S_V and L_V will be altered changing the b_{S_V} and b_{L_V} predicted by equations 5 and 6. For our deformation parameter we chose ϵ, where

$$\epsilon=\sqrt{1-z^2}$$

where z is the ratio of compressed axis to the elongated axes.

Then the change in b_{S_V} and b_{L_V} as a function ϵ (see appendix) can be calculated.

$$\Delta b_{S_V}(\epsilon)=b-b_{S_V}(\epsilon) \tag{8a}$$

$$=\frac{k_{S_V}}{S_V(0)}\left\{1-2\left[(1-\epsilon^2)^{-\frac{1}{3}}+(1-\epsilon^2)^{\frac{2}{3}}\frac{\ln((1+\epsilon)/(1-\epsilon))}{2\epsilon}\right]^{-1}\right\} \tag{8b}$$

$$\Delta b_{L_V}(\epsilon)=b-b_{L_V}(\epsilon) \tag{9a}$$

$$=\frac{k_{L_V}}{\sqrt{L_V(0)}}\left\{1-\left[\frac{(1-\epsilon^2)^{\frac{1}{3}}+(1-\epsilon^2)^{-\frac{1}{6}}\frac{\sin^{-1}(\epsilon)}{\epsilon}}{2}\right]^{-\frac{1}{2}}\right\} \tag{9b}$$

where

$S_V(0)=S_V$ at zero deformation

$L_V(0)=L_V$ at zero deformation

The ratio of equation 8 to equation 9 is plotted in figure 3. This ratio goes to 2 as $\epsilon\rightarrow 0$. Consider the following limits for equations 8 and 9,

$$\lim_{\epsilon\rightarrow 0}\Delta b_{S_V}(\epsilon)=\frac{k_{S_V}}{S_V(0)}\frac{2}{45}\epsilon^3=b\frac{2}{45}\epsilon^3 \tag{10}$$

$$\lim_{\epsilon\rightarrow 0}\Delta b_{L_V}(\epsilon)=\frac{k_{L_V}}{\sqrt{L_V(0)}}\frac{1}{45}\epsilon^3=b\frac{1}{45}\epsilon^3 \tag{11}$$

Solving for b in terms of $b_{S_V}(\epsilon)$ and $b_{L_V}(\epsilon)$ using equations 8a, 9a, 10 and 11 yields

$$b=\lim_{\epsilon\rightarrow 0}(2b_{L_V}(\epsilon)-b_{S_V}(\epsilon)) \tag{12}$$

which in the limit of $\epsilon\rightarrow 0$, is identical to equation 7. A similar analysis for elongation (e.g. wire drawing procedures) yields identical results for equations 10, 11 and thus 12.

The derivation makes no assumptions about initial values of k_{S_V} or k_{L_V}. Also equation 12 is additive, i. e., different parts of the microstructure can be compressed and elongated to different degrees and the cumulative results will still yield equation 12. This result suggests why equation 7 is expected.

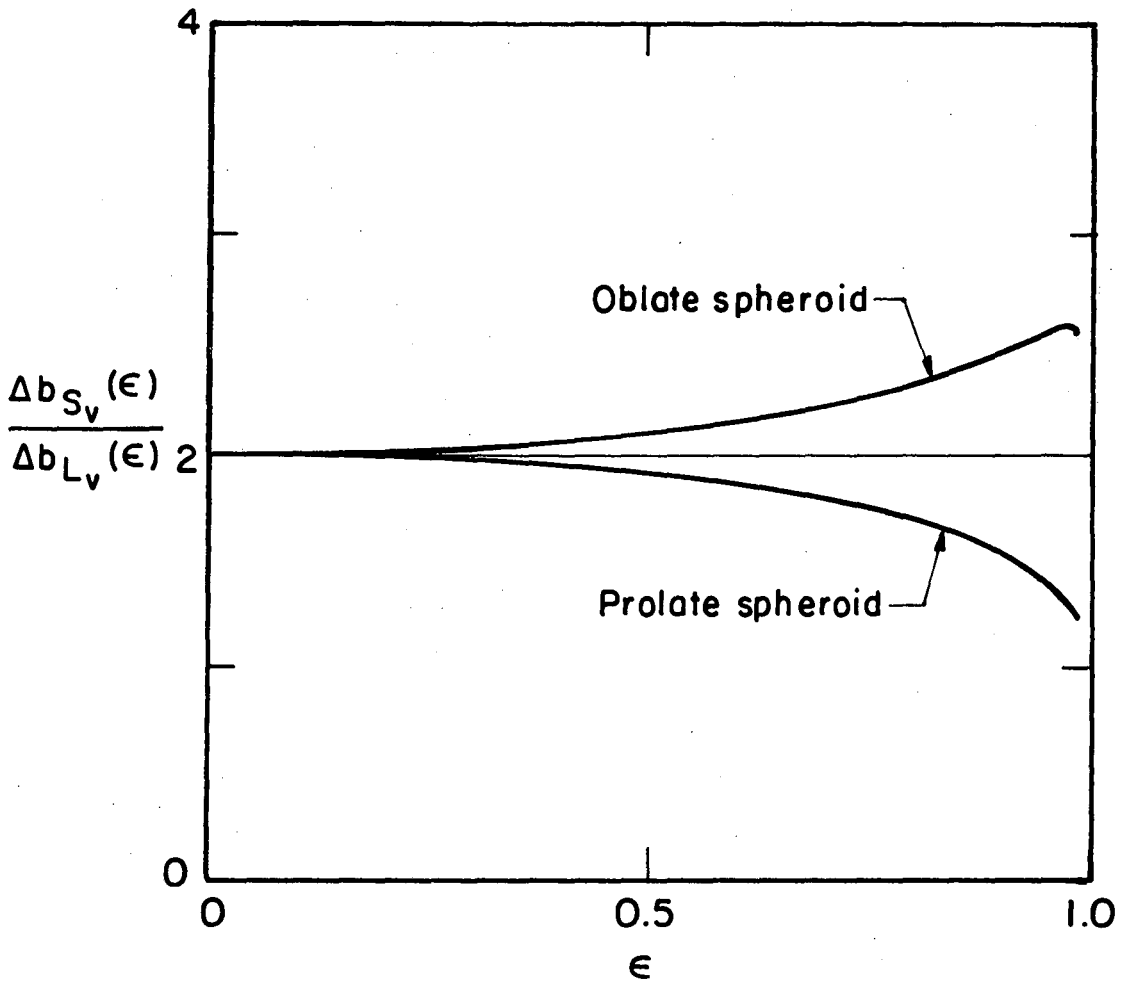
4
Oblate spheroid
$\frac{\Delta b_{S_v}(\epsilon)}{\Delta b_{L_v}(\epsilon)}$
2
Prolate spheroid
0
0
0.5
1.0
ϵ
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FIG. 3

6. A METHOD FOR EVALUATING N_V

To determine the average grain density in a three-dimensional microstructure from two-dimensional sections we suggest using equation 7 with Meijering's "cell model" coefficients ($k_{L_V} = 1.2075$ and $k_{S_V} = 1.4552$). Then b is given by

$$b=2.4150\sqrt{\bar{a}}-1.4552\bar{l} \tag{13a}$$

Since $N_V=b^{-3}$, $N_A=1/\bar{a}$ and $N_L=1/\bar{l}$, we get

$$N_V=(\frac{2.4150}{\sqrt{N_A}}-\frac{1.4552}{N_L})^{-3} \tag{13b}$$

As previously indicated, the sample plane(s) must be randomly orientated with respect to the microstructure.

$\bar{a}$ can be measured by first selecting a random rectangular area on the test plane. Then counting the number of grains in the test area, counting grains that intersect the edges and corners as half and quarter grains respectively. $\bar{a}$ is found by dividing the test area by the number of grains. $\bar{l}$ can be measured by first drawing lines across the rectangle. Then counting the number of line intercepts, i. e., where the scribed lines intersect grain boundaries. $\bar{l}$ is the total test line length divided by the number of intersections.

7. DISCUSSION

Much controversy has existed regarding whether grain size should be measured using $\bar{a}$ (ASTM and Jeffery's method) or $\bar{l}$ (Heyn's method). The former method is related to L_V while the later is related to S_V. Neither method can be directly correlated to grain density (see table 1) because of varying cell shape geometries encountered in different microstructures. However, we have presented a method using both parameters, L_V and S_V, to more precisely estimate N_V. Inversely, this additional knowledge combined with L_V and S_V should provide

useful information about microstructure geometries and stability. For example, the stability of microstructures can be correlated with surface area per grain, i. e., a microstructure with high surface area per grain has a corresponding high surface energy per grain. It should prove instructive to study phenomenon such as grain growth with respect to stored surface energy per grain.

In general, equation 13 enables three-dimensional grain density, N_V, to be estimated from two-dimensional quantities, $\bar{a}$ and $\bar{l}$. While stereologists can estimate L_V and S_V from these two-dimensional parameters, we have exhibited a method for also estimating the three-dimensional grain density, N_V. In contrast to previous methods [1, 4, 5, 6], this procedure does not require information about shape or size distribution of the features.

This procedure also holds for certain non space filling features, i. e., where the volume fraction is < 1. For example, if any of the microstructures considered herein were allowed to dissociate leaving free space between grains, $\bar{a}$ and $\bar{l}$ for these particles would not change, enabling application of equation 13a. If equally sized spherical inclusions are considered, equation 13a predicts a sphere size within 3.6% of the actual size.

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APPENDIX

Determination of L_V and S_V at constant volume during compression and elongation as a function of deformation can be done by specifing and solving equivalent problems. First we consider compression where the axes orthogonal to the compressed axis expand uniformly to maintain constant volume (and thus constant cell density). If we consider a volume element that contains randomly oriented surfaces, the expected surface density for any given orientation is identical to that of any other orientation. Another geometry that has the equivalent property is the surface of a sphere (or hemisphere). Therefore, we can insert a sphere of unit area inside the volume element and relocate all grain boundary surfaces to portions of the sphere having the same orientation without altering the density of surfaces with respect to orientation. If the volume element including the sphere is compressed uniformly at constant volume, then the sphere will be transformed into an oblate spheroid having the same volume as the initial sphere. The surface area and volume of the oblate spheroid are

$$surface = 2\pi a^2 + \pi \frac{b^2}{\epsilon} \ln((1+\epsilon)/(1-\epsilon)) \tag{A1}$$

$$volume = \frac{4}{3}\pi a^2 b \tag{A2}$$

where

$$\epsilon = \sqrt{1-(\frac{b}{a})^2}$$

then,

$$\frac{S_V(\epsilon)}{S_V(0)} = \frac{1}{2}\left\{(1-\epsilon^2)^{-\frac{1}{3}} + (1-\epsilon^2)^{\frac{2}{3}} \frac{\ln((1+\epsilon)/(1-\epsilon))}{2\epsilon}\right\} \tag{A3}$$

where a and b are the major and minor axis respectively of the oblate spheroid. (Note, a and b have different meaning in this appendix, and should not be confused with the main text.)

A similar analysis for an elongated prolate spheroid yields

$$\frac{S_V(\epsilon)}{S_V(0)} = \frac{1}{2}\left\{(1-\epsilon^2)^{\frac{1}{3}} + (1-\epsilon^2)^{-\frac{1}{6}} \frac{\sin^{-1}\epsilon}{\epsilon}\right\} \tag{A4}$$

where a and b are the major and minor axis respectively of the prolate spheroid.

To calculate the change in L_V we use a similar analysis. Again we consider a volume element where lines in three-dimensional space are randomly oriented. If we consider a sphere where the density of diameters is everywhere equally probable, then the normalized density of diameters as a function of θ is $\cos\theta$, where θ is the angle between a diameter and the plane perpendicular to the axis of compression (see figure 4). Again the density of diameters with respect to orientation is random. If the sphere is compressed at constant volume, the density of diameters is given by

$$\frac{L_V(\epsilon)}{L_V(0)}=\int_0^{\frac{\pi}{2}}\cos\theta\sqrt{(a\cos\theta)^2+(b\sin\theta)^2}\,d\theta=\frac{b}{2}+a\frac{\sin^{-1}\epsilon}{2\epsilon} \tag{A5}$$

where

$$\epsilon=\sqrt{1-(\frac{b}{a})^2}$$

Recasting this equation solely in terms of ϵ yields

$$\frac{L_V(\epsilon)}{L_V(0)}=\frac{1}{2}\left\{(1-\epsilon^2)^{\frac{1}{3}}+(1-\epsilon^2)^{-\frac{1}{6}}\frac{\sin^{-1}\epsilon}{\epsilon}\right\} \tag{A6}$$

A similar expression for elongated material where a and b are the major and minor axis of the prolate spheroid yields

$$\frac{L_V(\epsilon)}{L_V(0)}=\frac{1}{2}\left\{(1-\epsilon^2)^{-\frac{1}{3}}+(1-\epsilon^2)^{\frac{2}{3}}\frac{\ln(1-\epsilon)-\frac{1}{2}\ln(1-\epsilon^2)}{\epsilon}\right\} \tag{A7}$$

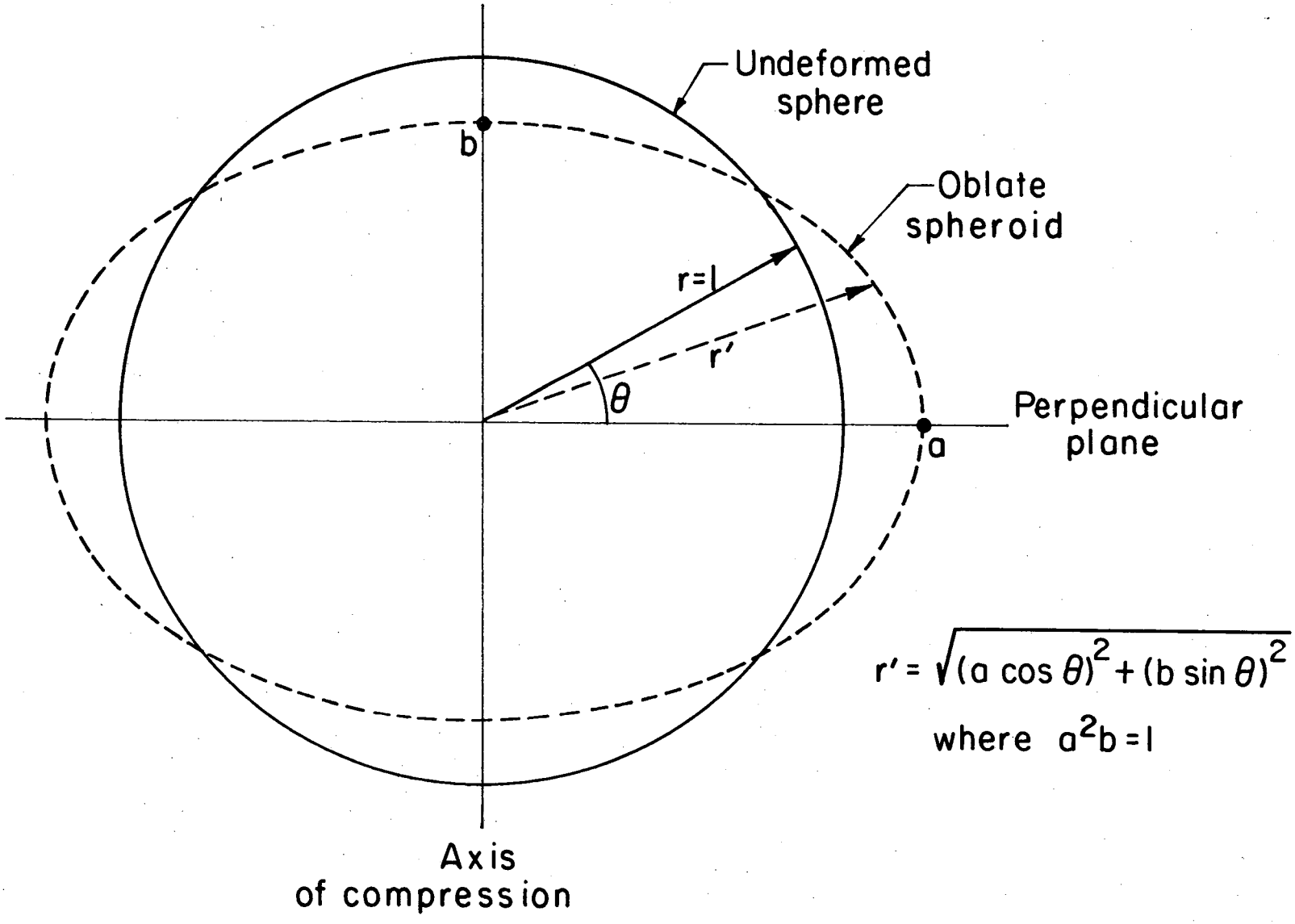
Undeformed
sphere
b
Oblate
spheroid
r=1
r'
θ
Perpendicular
plane
a
Axis
of compression
$r' = \sqrt{(a\cos\theta)^2 + (b\sin\theta)^2}$
where $a^2b = 1$
XBL 786-5138

FIG. 4

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