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REVERSED STEREOLOGY AND ITS APPLICATION TO INTERACTION BETWEEN SECOND PHASE AND GRAIN BOUNDARY

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ABSTRACT

'Reversed stereology' is a set of methods for quantitative characterization of feature / feature interaction(s) by applying the fundamental stereological principles in reverse order. Its basic idea is as follows:

Let Y_i and Y_j represent two sets of geometric features (e.g. points, lines, surfaces, volumes) embedded in a three-dimensional structure, X_3 , T_j be a set of isotropic uniform random test probes superimposed artificially onto the structure, where i and j denote the dimensionality of the corresponding features or probes. If the intersection sets $Y_i \cap Y_j$ and $Y_i \cap T_j$ are not empty, then the parameters $Q(Y_i \cap Y_j)$ and $Q(Y_i \cap T_j)$, where Q = surface area, line length, number of points or number of features, etc., can be combined into various estimators of the degree of randomness of the interaction between Y_i and Y_j .

Examples of application of reversed stereology to the quantitative characterization of interaction phenomena between geometric features related to second phase and grain faces, edges, and corners are systematically discussed in this paper.

Keywords: stereology, microstructure, grain boundary, second phase, nucleation and growth, feature / feature interaction, spatial distribution.

INTRODUCTION

Concerning with the tendency or preference for a set of geometric features to intersect geometrically with another set or even the same set, 'associations' of features with each other have been considered one kind of important characteristic of microstructural inhomogeneities in materials in geometry (DeHoff, 1982). Quantitative description of this kind of phenomena is of great significance in quantitative studies of microstructural evolution such as heterogeneous phase transformation and sintering processes, provides certain information of spatial distribution of features, and has drawn more and more attention. Aigeltinger and Exner (1977) studied quantitatively the pore surface / grain face interaction by comparing the real length density of the grain / grain / pore triple lines with that calculated by assuming a perfectly random interaction, although their expression was erroneous (Liu, 1993: Appendix B). Gokhale et al (1981) proposed a parameter aiming to describe the grain boudary affinity of precipitates using almost the same strategy as Aigeltinger and Exner did. Later, in their studies of either sintering processes and phase transformations, Liu et al (1990, 1991) found that the particle (or pore) / grain face interaction and the particle or pore / grain edge interaction can be separately described, and a corrected form of Aigeltinger and Exner's expression was also used to quantify the surface / surface interaction. A recent critical review (Liu, 1993) revealed that the exactly same idea, being simple but general, underlies all the above–mentioned studies, which is the 'reversed stereology'. Focusing on the particle / grain boundary interaction phenomena, more discussions of the reversed stereology and its application examples will be given in this paper.

BASIC IDEAS

'REVERSED STEREOLOGY' is a set of methods for quantitative evaluation of feature / feature interaction(s) by applying the fundamental stereological principles in reverse order. Its basic idea is as follows (Liu, 1993):

Let Y_i and Y_j represent two sets of geometric features (e.g. points, lines, surfaces, volumes) embedded in a three–dimensional structure, X_3 ; while T_j represents a set of isotropic uniform random (IUR) test probes superimposed artificially onto the structure, where i and j denote the dimensionality of the corresponding features or probes. If the intersection sets $Y_i \cap Y_j$ and $Y_i \cap T_j$ are not empty, then the parameters $Q(Y_i \cap Y_j)$ and $Q(Y_i \cap T_j)$, where Q = certain parameter related with surface area, line length, number of points or number of features, etc., can be combined into various estimators of the degree of randomness of the interaction between Y_i and Y_j .

To apply the reversed stereology principle, the key is to find the value of $Q(\text{random}) = Q(Y_i \cap T_j)$, i.e., the value of a given stereological parameter assuming a perfectly random feature / probe (feature) interaction, then compare it with the value of the same parameter corresponding to the true interaction, Q(exp), in order to determine the randomness or nonrandomness of the interaction. The Q(random) can be either experimentally determined by superimposing a set of IUR test probes, or simply calculated from existing data of other parameters assuming a perfectly random interaction.

The parameters directly used for quantitative characterization of feature / feature interactions may be defined in different forms, such as Q(exp) / Q(random), Q(exp) / [Q(exp)+Q(random)], or other kinds of combination of Q(exp) and Q(random), etc.

The practical value of the reversed stereology is at least two–folds: providing a set of methods for quantitative characterization of feature / feature interactions, and providing certain information about spatial distribution of given kind of features with another set of features as reference frame.

APPLICATION TO INTERACTION BETWEEN SECOND PHASE AND GRAIN BOUNDARY

In materials science, usually the sets, $Y_i (i= 3, 2, 1)$, are related to the same given particle (or pore, precipitate) set embedded in the matrix of a microstructure, while $Y_j (j=2, 1, 0)$ are grain boundary subsets. Table 1 has summarized all the possible feature / feature interaction pairs and some of possible parameters which can be used to further define various parameters for quantifying the randomness of each corresponding interaction, although not necessarily all the suggested parameters are stereologically accessible. Table 2 has summarized some phenomena related to nucleation and growth (or annihilation and shrinkage) of second phase particles at the grain boundary which may be quantitatively characterized by reversed stereology. Combination of Tables 1 and 2 gives a rather clear picture, although not necessarily exhaustive, of what one can do in stereological evaluation of the interaction between the particle–related features and the grain boundary of the matrix. The original definition of some parameters in Tables 1 and

2, such as Y_j^{GB} and those of contiguity C_{GB}^{pp} and C_{3D}^{pp}, can be found in (Liu, 1993).

Now, let us see some examples. First set i=3 and j=2, then the reversed stereology can be used to quantitatively characterize particle volume / grain face interaction or the comprehensive preference of nucleation and growth of second phase at the grain boundary. It is based on the stereological evaluation of S_S, the area fraction occupied by the intersecting profiles of particles in the surface set Y_2, and A_A, that obtained on the IUR test plane set, T_2. The latter always equals to the volume fraction of second phase, V_V, in value. In this case, if one has statistically

$$S_S(\exp) = S_S(\text{random}) \tag{1}$$

i.e., $$S(Y_3 \cap Y_2) / S(Y_2) = A(Y_3 \cap T_2) / A(T_2) = V_V \tag{2}$$

then he knows in principle that the interaction between Y_3 and Y_2 is also perfectly random. Otherwise, the reverse is true: if $S_S(\exp) > S_S(\text{random})$, then the particles in Y_3 tend to intersect the surfaces in Y_2, and the difference between $S_S(\exp)$ and $S_S(\text{random})$ or their ratio represents the strongness of that tendency; if $S_S(\exp) < S_S(\text{random})$, then the particles tend to avoid the surfaces.

Again for i=3 and j=2, the reversed stereology method may also be used to characterize the preference of nucleation and that of growth of second phase at the grain boundary separately, as shown in Tables 1 and 2.

Set i=3 and j=1, then one may get another application example of the reversed stereology used to quantify particle volume/grain edge interaction and is based on the application of the relation

$$L_L(\exp) = L_L(\text{random}) = V_V \tag{3}$$

where L_L represents the length fraction of Y_1 or T_1 occupied by the particles in Y_3, no matter the elements of Y_1 or T_1 are straight or curved lines or linear features. If the left part of Eq.(3) is not true, then the interaction is not random, and the difference between $L_L(\exp)$ and $L_L(\text{random})$ will be an indicator of the nonrandomness of the interaction.

Set i=3 and j=0, then the reversed stereology may be used to quantify the particle volume/grain corner interaction if the point fraction of Y_0 occupied by the particles in Y_3 can be experimentally determined.

Set i=2 and j=2, then based on the application of an expression derived from the fundamental stereological relations by assuming a perfectly random interaction between the two sets of surfaces (Aigeltinger and Exner, 1977; Liu, 1993: Appendix A):

$$L_V^{pmm}(\text{random}) = \pi S_V^{GB}(\text{ext}) S_V^{pm} / 4 \tag{4}$$

the reversed stereology principles can be used to characterize surface/surface interaction, where L_V^{pmm} denotes the length density of the grain/grain/particle triple lines, S_V^{pm} is the surface density of grain/particle interfaces, while $S_V^{GB}(\text{ext})$ is the surface density of the 'extended grain boundary' (Liu, 1993).

Again for i=2 and j=2, other kinds of interactions including contiguity also may be quantified by the reversed stereology (Tables 1 and 2).

In principle, one can get at least one application example of reversed stereology for any (i,j) pair as long as $i+j > n$, where n is the dimensionality of the space concerned (cf. Tables 1 and 2). However, it should be pointed out that only those cases corresponding to i=3 can provide true information of the interaction between the particle volume or pore volume with another set of features (say, surfaces, linear features, point features). If the reversed stereology principle were used in a case of $i \neq 3$ to get such kind of information, the results may be very misleading. For example, for the case of i=2, j=2, and $L_V^{pmm}(\exp) = 0 \neq L_V^{pmm}(\text{random})$, the particles may occupy all the grain boundary faces, or may be no contact with the grain boundary at all. It has demonstrated well that the reversed stereology is there ready for use, but cautions should be taken with the physical meaning of each parameter correspondingly defined.

Table 1. A list of particle–related feature / grain boundary interaction pairs in a two phase microstructure and some possible parameters which may be used indirectly for quantification of each interaction. Letters m and p in superscripts stand for the matrix phase and the second phase (particles, pores, etc.), respectively; while the subscripts represent the dimensionality of corresponding feature sets. Capital letters V, S, L, N, and P in parameters denote volume, surface area, length, number of given features and number of points, respectively.

i+j	Feature–set Pair $Y_i \diagdown Y_j$	Interaction Type	Intersection Trace	Possible Parameters (Q)
5	$Y_3^p \diagdown Y_2^{GB}$(ext)	Particle volume ╲ Grain face	Particle profiles in grain faces	S_S, N_S^*, number of profiles per unit length of GB trace lines on 2D section, mean length of profile intercepts in grain faces, etc.
4	$Y_3^p \diagdown Y_1^{GB}$(ext)	Particle volume ╲ Grain edge	Segments of grain edges occupied by particles	L_L, N_L^*, etc.
3	$Y_3^p \diagdown Y_0^{GB}$(ext)	Particle volume ╲ Grain corner	Grain corners occupied by particles	P_P^*
4	$Y_2^{pm} \diagdown Y_2^{GB}$(ext)	Particle surface ╲ Grain face	p–m–m triple lines	L_V, L_S, etc.
4	$Y_2^{pp} \diagdown Y_2^{GB}$(ext)	Interparticle surface╲Ficticious grain face	Ficticious m–m–p triple lines	L_V, L_S, etc.
4	$Y_2^{pm+pp} \diagdown Y_2^{GB}$(ext)	Total particle surface╲ Grain face	Union of true and fict. m–m–p lines	L_V, L_S, Contiguity, etc.
3	$Y_2^{pm} \diagdown Y_1^{GB}$(ext)	Particle surface ╲ Grain edge	p–m–m–m qradruple points	P_V^*, etc.
3	$Y_2^{pp} \diagdown Y_1^{GB}$(ext)	Interparticle surface╲Ficticious grain edge	Ficticious points	P_V^*, etc.
3	$Y_1^{ppm} \diagdown Y_2^{GB}$(ext)	p–p–m triple line ╲Grain face	p–p–m–m qradruple points	P_V^*, P_S^*, etc.
3	$Y_1^{ppp} \diagdown Y_2^{GB}$(ext)	p–p–p triple line ╲ Ficticious grain face	Ficticious points	P_V^*, P_S^*, etc.
3	$Y_1^{pmm} \diagdown Y_2^{GB}$(ext)	Already considered in $Y_2^{pm} \diagdown Y_2^{GB}$(ext) interaction		

*: Stereologically inaccessible.

Table 2. Quantitative characterization of various phenomena related to nucleation and growth of second phase particles (p) at the grain boundary (GB) of matrix phase (m)

Nucleation and Growth Sites	Type of Phenomenon	Possible Parameters, Q(exp) or Q(exp) / Q(random)	Reversed Stereology Related ?
At grain faces	Degree of site saturation	Area fraction occupied by particles, $S_S^p(GB)$	No
	Comprehensive preference of nucleation and growth at grain faces	$S_S^p(GB) / A_A^p(\text{random})$, etc.	Yes
	Preference of nucleation at grain faces	(Number of particle profiles per unit length of GB trace) / (number of particles per unit length of IUR test lines in 3D)	Yes
	Preference of growth at grain faces	(Mean intercept length of particles in grain faces) / (mean intercept length of particles in 3D)**	Yes
	Preference of contiguity at grain faces	$C_{GB}^{pp} / C_{3D}^{pp}$	Yes
	Preference of particle surface \ grain face interaction	$L_V^{pmm}(\text{exp}) / L_V^{pmm}(\text{random})$	Yes
	Homogeneity of particle distribution over grain faces	Limited information from C_{GB}^{pp}	No
At grain edges	Degree of site saturation	Length fraction occupied by particles, $L_L^p(GB)$	No
	Comprehensive preference of nucleation and growth at grain edges	$L_L^p(GB) / L_L^p(\text{random})$, etc.	Yes
	Preference of nucleation at grain edges	(Number of particles per unit length of grain edge)* / (number of particles per unit length of IUR test lines in 3D)	Yes
	Preference of growth along grain edges	(Mean intercept length of particles along grain edge)* / (mean intercept length of paticles in 3D)**	Yes
At grain corners	Degree of site saturation	Point fraction occupied by particles*; limited information from edge saturation	No
	Comprehensive preference of nucleation and growth at grain corners	$P_P^p(GB)^* / P_P^p(\text{random})$, etc.	Yes

*: Stereologically inaccessible; **: Also depending on nucleation preference.

CONCLUSIONS

From the practical point of view, this paper may be considered a complement of author's previous work (Liu, 1993). The discussion has been expanded into a systematic analysis of various kinds of interactions between geometric features related to the second phase (particles, pores, precipitates) and grain faces, grain edges, grain corners (cf. Table 1); and into the possible approaches to apply the reversed stereology to quantitative characterization of various phenomena related to nucleation and growth (or annihilation and shrinkage) of the second phase at different kinds of grain boundary sites of the matrix phase (cf. Table 2). The reversed stereology also seems to be able to provide certain information of the spatial distribution of second phase particles with the grain boundary of matrix phase as the reference frame.

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