ACTA STEREOL 1994; 13/2: 281-286
PROC 6ECS PRAGUE, 1993
ORIGINAL SCIENTIFIC PAPER

EFFICIENT AND UNBIASED EVALUATION OF SIZE AND TOPOLOGY OF SPACE–FILLING GRAINS

Guoquan LIU, Haibo YU, Wenqing LI

Department of Materials Science and Engineering
University of Science and Technology Beijing, 100083 CHINA

ABSTRACT

The applications of the new stereological methods such as the disector and the point–sampled intercepts have been mainly published in studies of non–space–filling particle systems with few exceptions. This paper demonstrates the feasibility of using the disector and the point–sampled intercepts as unbiased and efficient methods for size and topology evaluation of space–filling grains, with limited information on the distribution, in engineering materials. The rules of identifying and counting of grains and grain corners are given for the disector analysis. The study of the size and the topology of grains in an austenite grain growth series of microalloyed low–carbon steel samples has been used as an example.

Keywords: grain size, topology, grain growth, space–filling grains, stereology, disector, point–sampled intercepts.

INTRODUCTION

A single–phase space–filling grain structure consists of 4 subsets of geometrical elements: grain corners (quadruple points), grain edges (triple lines), grain faces, and grain volumes, which essentially follow the '2–2, 3–1, 4–0 rules' (Liu, 1993). Underwood (1970) made an excellent review on the 3D studies of such space–filling cells or grains up to 1968, where some 3D pictures of grains from metals and alloys, cells from plant, human fat tissue and bubble froth were shown as examples. The serial section analysis was and has been continuously used as one of the most common methods to study the size, the topology, and the shape of 3D space–filling grains or cells (Rhines, 1967; Underwood, 1970; Rhines et al., 1974; Rhines and Patterson, 1982; Liu, 1984; Nunez and Domingo, 1988), and the necessary analysis rules along with the fundamental principle of treating each feature as a point to avoid the boundary bias in the 3D feature counting have been systematically established (Rhines et al., 1976). However, the low efficiency of the serial section analysis frightens many researchers away, so that new experimental methods of higher efficiency have been sought after for the unbiased estimation of grain size and topology.

In this paper, the problem has been solved by reducing the number of the dependent serial sections to 2 with modified counting rules of both grains and quadruple points, which implies that in principle the analysis has become that of the disector described by Sterio (1984). The disector with an unbiased counting frame improves the efficiency significantly because not only the number of the dependent section planes needed

has been reduced, but also it is no longer necessary to give an individual identification number to each grain as a must in the serial section analysis. On the other hand, the disector and the point–sampled intercept methods have been combined to get certain information of the grain volume distribution to make compensation for the disadvantage of the disector analysis which tells nothing of the distribution of grain volumes, faces, edges, and corners.

MATERIAL AND EXPERIMENTAL METHODS

The chemical compositions (wt.%) of the experimental steel were: C.18, Si.29, Mn1.14, Cr1.06, V.11, Ti.062, B.0027, Al.031, N.0044, P.024, S.020. Five samples of 10 mm in diameter and 12 mm in height were prepared from forged rods of 13 mm in diameter, isothermally treated at 1050 ℃ for 1 / 2, 1, 10, 30, and 60 min, respectively, then water quenched.

The disector of known volume (Sterio, 1984) has been used here for unbiased estimation of not only the numerical density of grains, N_V, but also of the quadruple–point (grain corner) density, C_V, from which some selected parameters such as the number–weighted mean grain volume, v_N, the numerical density of grain edges, E_V, the numerical density of grain faces, F_V, the number of corners per grain, C_g, the number of edges per grain, E_g, and the number of faces per grain, F_g, are further derived according to the relationships given in the references (Underwood, 1970; Rhines et al., 1976; Liu et al., 1993).

In the serial section analysis, the identifying and counting of grain corners or quadruple points are carried out by classifying them into three types, which correspond to three topological configurations that occur upon two successive sections (Rhines et al., 1976): Either of types ① or ② (Fig. 1a) corresponds to a grain corner and to the appearance or the disappearance of a grain in between the sections, while type ③ (Fig. 1b) corresponds to a grain corner without appearance or disappearance of grains. The same applies in the disector analysis (Liu et al., 1993): Type ① serves as 'top points' of grains used to acquire the numerical density of grains. Moreover, the sum of the numbers of all the three types are used to estimate the numerical density of grain corners in the material.

The following new counting rules have been designed in order to implement Rhines' topological configuration analysis to the unbiased evaluation of the size and the topology of space–filling grains with the disector. Place the unbiased counting frame (Gundersen, 1977) in the reference plane of the disector. Then,

i) the top point of a grain (a grain corner corresponding to the configuration of type ①) is counted only when the grain profile, usually 3–sided, newly appearing in the reference plane falls at least partially within the area of the frame without crossing over the 'forbiden lines' of the frame. Very rarely, the new grain profile in the reference plane may appear as 2–sided (Rhines et al, 1976), which corresponds to a top point of a new grain but not to a grain corner;

ii) a grain corner corresponding to the configuration of type ② is counted only when the corresponding triple point in the reference plane falls in the frame area;

iii) for that corresponding to type ③, the configuration is counted as a grain corner only when its central line segment falls at least partially within the frame area without crossing over the 'forbiden lines';

iv) some complicated configurations formed by combination of type ③ with type ① or ② may appear, although in our practice they are very rare and each is composed of no more than 2 or 3 basic configurations mentioned above because the 'thickness' of the disector was taken as only 1 / 15 to 1 / 30 of the average grain intercept in this work.

An example is shown in Fig. 1c. In such cases, the top point of a grain is counted only if a grain profile newly appears in the reference plane which does not violate Rule (i), regardless of the number of sides of the profile. The number of grain corners are taken as the difference of the numbers of the profile sides contained in a complicated configuration that occurs upon the two section planes, $\triangle n$, minus 2. When the arrow points to the right, the configuration shown in Fig. 1c represents 2 grain corners, one of which is the top point of a grain. When the arrow points to the left, it represents 2 grain corners with no top point. However, if a 2–sided profile appears in the look–up plane, while the corresponding profile in the reference plane is n–sided, then the configuration is composed of $\triangle n$ basic ones of type ③ and so counted.

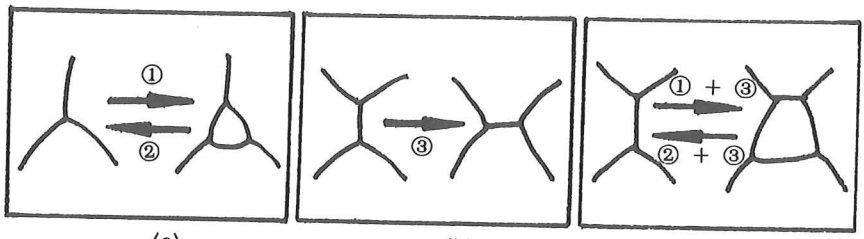

Fig. 1. Three basic types of topological configurations that occur upon two successive sections in the serial section analysis or the disector analysis (a and b), and examples of complicated ones (c). When used in the disector analysis, the direction of arraws labelled in the configurations always represents that from the look–up plane to the reference plane. See the text for details.

At least 2000 grain profiles have been observed for each sample studied in this work to ensure the statistical representativity of the evaluation results. The relative error of N_V for all the samples is at least less than 6% .

Systematic errors in the N_V estimation of grains in a space–filling polycrystal by using the disector may come from (1) overestimation, resulted from possible multi–profile appearance of a grain in a section plane due to the concavity of the grain surface or of the grain edge; (2) underestimation, resulted from small grains situated in between the planes of the disector but appearing in neither of the two planes; this may also result from mistakenly attributing 2 equal–sided profiles of 2 grains appearing in the two planes to the same grain (Rhines et al., 1976); and (3) the boundary (or edge) errors. Obviously, the edge errors have been completely removed by using the unbiased counting frame and the corresponding counting rules. Because the disector used here is relatively thin, for example, its thickness for No.1 sample is only about 1 / 26 of the average mean caliper diameter of the grains, about 1 / 10 of the mean segment length of grain edges, or less than 1 / 16 of the mean Feret diameter of grain faces in their own 'planes', hence the second type of errors should be negligible. It is also found that the first type of errors is less than 1% for the samples studied in this work. Obviously, this type of errors approaches zero when the faces and edges approach planes and straight lines, respectively. Moreover, when two 3–sided profiles newly appeared in the reference section locate near by each other, they may be from the same grain or may not. When this happened, a third section was made to further reduce the first type of errors.

The point–sampled intercept method (Gundersen and Jensen, 1985) has been used extensively in dispersed particle systems (Cruz–Orive and Weibel, 1990), with very rare reports for space–filling grains (Kurzydlowski and Bucki, 1992). The method has

been used here mainly for unbiased estimation of the volume–weighted mean grain volume, v_V, of the coefficient of variation of grain volume in the number frequency distribution, $CV_N(v)$, and for estimation of v_N when $CV_N(v)$ is known. The experimental procedure was so arranged that the relative error of v_V is less than 5% .

RESULTS AND DISCUSSION

The experimental data acquired have been summarized in Table 1, on these data an outline is given on the global topological change of grains during the austenite grain growth, on the feasibility of approximating v_N directly or indirectly using the v_V data, etc.

The only quantitative information in the literature on the topological change of metal grains as a function of isothermal grain growth time is from Rhines et al. (1974) for steady–state grain growth in high purity aluminum. It demonstrated the average number of corners per grain C_g to approach 24, the average number of edges per grain E_g to approach 36, and the average number of faces per grain F_g to approach 14 for separated grains. I.e., all data approach those describing topologically the tetrakaidecahedron or the truncated octahedron. The data in Table 1 not only show a similar tendency of topological change in isothermal austenite grain growth in the microalloyed steel, but also show such a tendency to be maintained even in the *non–steady–state* stage of a grain growth process in which $CV_N(v)$ varies.

Table 1. Experimental data of selected topological and metric parameters for the steel samples undergoing isothermal grain growth at 1050 ℃

Sample	No. 1	No. 2	No. 3	No. 4	No. 5
Time at 1050℃, min	0.5	1.0	10.0	30.0	50.0
C_g	21.15	21.25	21.97	22.06	22.76
E_g	31.73	31.87	32.96	33.08	34.13
F_g	12.58	12.62	12.99	13.03	13.38
C_V, $x10^4\ mm^{-3}$	8.30	6.53	5.11	4.41	3.92
E_V, $x10^4\ mm^{-3}$	16.61	13.07	10.22	8.82	7.85
F_V, $x10^4\ mm^{-3}$	9.88	7.76	6.04	5.21	4.61
N_V, $x10^4\ mm^{-3}$	1.57	1.23	0.93	0.80	0.69
v_N, $x10^{-4}\ mm^3$	0.64	0.81	1.08	1.25	1.45
v_V, $x10^{-4}\ mm^3$	1.18	1.48	1.96	2.73	3.54
$CV_N(v)$	0.92	0.90	0.91	1.09	1.12
s_F, $x10^{-4}\ mm^2$	6.57	7.50	8.17	8.99	9.93
d_{eq}, $x10^{-3}$ mm	49.63	53.68	59.08	62.04	65.18

It follows from the relation

$$CV_N(v) = [(v_V / v_N) - 1]^{1/2} \qquad (1)$$

that the relative difference between v_V and v_N for the same set of grains is less that 10% when $CV_N(v) < 0.3$. In a biomedical context, sometimes such low $CV_N(v)$ values are found for non–space–filling features so that v_V can be used as a first approximation of v_N (Yang, 1989). In single–phase polycrystalline metals or alloys, the $CV_N(v)$ of grain volume distribution is usually relatively large. For example, $CV_N(v) = 1.09$ to 2.13 for the

7 samples of annealed high purity aluminum studied by Rhines and Patterson (1982), $CV_N(v) = 0.87$ for an annealed pure alpha–iron sample studied by Liu (1984), and $CV_N(v) = 0.90$ to 1.12 for the austenite grains in the 5 steel samples studied in this work. For these samples just mentioned, the v_V is larger than the corresponding v_N by at least 75%, at most by 450%. Therefore, it is inappropriate in such cases to use v_V as an approximation of v_N . However, this does not imply that one has no opportunity to employ the point–sampled intercept method to estimate v_N of metal grains indirectly. For example, the $CV_N(v)$ keeps constant in the steady–state grain growth process in single–phase metals (Atkinson, 1988). Therefore, once the $CV_N(v)$ of one of the samples in the steady–state grain growth series has been estimated experimentally, the v_N data for all other samples in the same series can be calculated based on Eq.(1) from the data of v_V acquired on independent sections using the point–sampled intercept method. The validity of this strategy has been confirmed by the experimental data of the first 3 samples which came from the steady–state stage of the grain growth: the maximum difference between the v_N value so calculated and that acquired by the disector is only 1.51% .

It may be worthy of note that the information on the grain volume distribution does no help to estimate the corresponding distribution information of grain faces, edges, and corners, because only those relations between the distributions of the grain diameter (not volume!) and the grain topological parameters have been experimentally found and theoretically confirmed (Liu, 1993).

The data of the mean area of grain faces, s_F, derived from both the data of the grain surface density and the numerical density of grain faces also have been listed in Table 1. A linear regression analysis of the data shows that s_F is essentially proportional to the second power of the so–call 'mean equivalent grain diamenter', $d_{eq} = (6\ v_N / \pi)^{1/3}$, with a correlation coefficient as high as 0.991. Nevertheless, the d_{eq} , so defined, is always larger than the average value of the true equivalent diameter of individual grains except for equal–size grain systems.

CONCLUSIONS

1. By implanting Rhines' rules of identifying grain corners used in the serial section analysis (Rhines et al., 1976) into the disector method, the unbiased estimation of grain volume and the topology in a group of microalloyed steel samples has been carried out. The samples belong to the same isothermal austenite grain growth series, and are all space–filling grain aggregates. Corresponding rules for unbiased counting of grains and grain corners have been designed. The disector method owns both the advantage of higher efficiency and the disadvantage of telling nothing of the distributions of grain volumes, faces, edges, corners, and shapes. The latter has been partially compensated by a combined application of the disector and the point–sampled intercepts to obtain $CV_N(v)$ in this work.

2. The mean grain volume number–weighted, v_N, is much smaller than that volume–weighted, v_V, in all the samples studied, so that the former cannot even be roughly approximated directly by the latter.

3. The characteristic of constant width of grain size distribution during the steady–state grain growth process in single–phase materials (Atkinson, 1988) simplifies the procedure of the N_V estimation. That is, in the study of such a process, once the $CV_N(v)$ for one of the samples has been known, the numerical density or the mean volume of grains for all the other samples in the same grain growth series can be estimated on independent sections with the point–sampled intercept method, without using the disector or other 3D sampling techniques. The results in the present work form

preliminarily evidence for such a conclusion.

4. The experimental results have shown that the average number of faces, edges, and corners per grain always increase monotonically during grain growth, and seems to approach 14, 36, and 24, respectively, regardless of the variation of the width of grain size distribution in the non–steady–state grain growth process studied.

ACKNOWLEDGMENTS

The research has been founded by the National Natural Science Foundation of China (project No. 59271063), to which the authors wish to express their appreciation.

REFERENCES

Atkinson HV. Theories of normal grain growth in pure single phase systems. Acta Metall 1988; 36: 469–491.

Cruz–Orive LM, Weibel ER. Recent stereological methods for cell biology: a brief survey. Am J Physiol 1990; 258: L148–L156.

Gundersen HJG. Notes on the estimation of the numerical density of arbitrary profiles: the edge effect. J. Microsc 1977; 111: 219–223.

Gundersen HJG, Jensen EB. Stereological estimation of the volume–weighted mean volume of arbitrary particles observed on random sections. J Microsc 1985; 138: 127–142.

Kurzydlowski KJ, Bucki JJ. A method for grain size and grain size uniformity estimation – applications to polycrystalline materials. Scripta Metall Mater 1992; 27: 117–120.

Liu G. Quantitative characterization of grain structure in pure iron. Ph.D. Dissertation, University of Florida, Gainesville, 1984.

Liu G. Invited review: Applied stereology in materials science and engineering. J Microsc 1993; 171: 57–68.

Liu G, Yu H, Li D, Li W. Unbiased estimation of spatial topological parameters of polycrystals (in Chinese with English abstract). Proc 6th Conf on Stereology and Image Analysis of China, Beijing 1993: 265–270.

Nunez C, Domingo S. Grain shape and its influence on the experimental measurement of grain size. Metall Trans A 1988; 19A: 933–940.

Rhines FN. Measurement of topological parameters. Proc 2nd International Congress for Stereology (ed. Elias H). Chicago, April 8–13, 1967: 247.

Rhines FN, Craig KR, DeHoff RT. Mechanism of steady–state grain growth in aluminum. Metall Trans 1974; 5: 413–425.

Rhines FN, Craig KR, and Rousse DA. Measurement of average grain volume and certain topological parameters by serial section analysis. Metall Trans A 1976; 7A: 1729 –1734.

Rhines FN, Patterson BR. Effect of the degree of prior cold work on the grain volume distribution and the rate of grain growth of recrystallized aluminum. Metall Trans A 1982; 13A: 985–993.

Sterio DC. The unbiased estimation of number and size of arbitrary particles using the disector. J Microsc 1984; 134: 127–136.

Underwood EE. Quantitative stereology. Addison–Wesley, Massachusetts, 1970. Section 7.6.3.

Yang ZW. Applied stereology in medicine and biology (in Chinese). Sichuan Society of Anatomy, 1989: 73.