The question of determination of grain size in researches of metal structures

Tatyana Danilenko

PhD in Technical Sciences
State Economy and Technology University of Transport
Kiev, Ukraine

Abstract
Analysis of the grain structures presented by standards G4, G5, G6 GOST 5639-82 showed that the set of flat grains of each standard is represented mainly by grains of three groups (82 ... 87%) – namely, those that correspond to the range of this group and two neighboring ones. Stereological reconstructed three-dimensional structures, plane sections of which correspond to the standards G4, G5, G6 GOST 5639-82, are more homogeneous, represented mainly by grains of two groups (81 ... 87%) and have larger (13 ... 17%) average diameter than their plane sections presented by standards.
Keywords: GRAIN STRUCTURE, GRAIN SIZE, THE STANDARDS GOST 5639-82, STEREOLOGICAL RECONSTRUCTION OF THREE-DIMENSIONAL GRAIN STRUCTURE

The size of grain is restricted for many types the steel products as grain structure determines significantly the number of properties, including mechanical. In standards GOST 5639-82 [1], ISO 643:2012 [2] for determination of grain size, grains of metals are separate crystals of polycrystalline conglomerate, i.e. grains are considered as three-dimensional objects; and at the same time, it is known that they have the form of the irregular polyhedrons [3]. At the same time “the grain size” in accordance with GOST 5639-82 is “the average size of accidental grains sections in the plane of metallographic specimen”, i.e. only average value and only in application to flat sections of three-dimensional grains is meant by concept of grain size (let us call these sections flat grains).

However, it is obvious that properties of product are affected by volumetric three-dimensional structure, but not its two-dimensional cut in metallographic specimen. The science stereology deals with ratios of parameters of three-dimensional structure and its flat cut; distribution of grains sizes and distributions of the sizes of flat and linear sections of grains are the main objects of the stereological analysis of polydisperse grain structures. Having established distribution of grains sizes, it is possible to calculate all the necessary quantitative characteristics of volumetric grain structure: average size of grains, amount of grains in unit of volume, characteristics of heterogeneity of structure, etc.

However, at the present time, research of grain structure of metallographic specimen continues to be generally accepted. In the existing standards [1, 2], the method of visual comparison of the flat grains seen under a microscope with scales standards presented in the form of the schematized net limiting the sizes of grains is one of the most widespread methods of determination of flat grains size.

In this paper, the following tasks have been set:

1) to analyze some standards of scales of GOST 5639-82, namely standards *G4, G5, G6*, for the purpose of establishment of distribution in relative fractions of P_k *(d)* of the sizes of flat grains in standards, i.e. establishment of size of flat grains for standard and quantitative characteristics of flat grains set;

2) to reconstruct distribution in relative fractions of $P(D_k)$ of the sizes of three-dimensional grains, which flat sections would correspond to the studied standards, by means of a stereological method on the basis of the established P_k *(d);*

3) to determine and compare characteristics of the reconstructed volume of grain structure and its flat cut in standards for the purpose of evaluation of degree of their distinction.

In accordance with GOST 5639-82, number of grain G is characterized by the average area of flat grains and the average diameter d_m, which is determined from ratio $d_m = 1/\sqrt{m}$ (where *m* – amount of grains for 1 mm^2 of the area of specimen), or if d_m is expressed by *a*, $d_m = 1/\sqrt{1/a} = \sqrt{a}$. At the same time, in the paper [4], it has been shown that it is correct to determine the diameter of flat grain-polyhedron as diameter equal to this flat grain of the circle d_S. In this case, if over any area S (which may be 1 mm^2) the amount of the counted grains is equal to *n*, the average area of grains can be found from a ratio $a_S = S/n$, and average diameter can be found from ratio $d_S = \sqrt{4a_S/\pi} = 1{,}1287\sqrt{a_S}$.

Therefore, the d_m is 1.1287 times less than d_S, i.e. distinction between them is about 13%; at the same time, d_S is the objective geometrical characteristic of the linear sizes of flat grain [4]. It should be considered at determination of grain size according to GOST 5639-82.

According to objective, the analysis of standards GOST 5639-82 assumed measurement of the area of each flat grain by a standard, calculation of its diameter *d* and distribution of diameters by the sizes, i.e. establishment of P_k *(d)* for each standard. At the same time, both d_S and d_m were determined.

As number of grain G is characterized not only by the average area of flat grains and average diameter d_m, but also and the minimum, average and maximum number of grains for 1 mm^2, in this research the minimum, average and maximum diameters of d_S and d_m of grains of certain standard have been determined.

The results of calculations presented in Table 1 have shown that the maximum d_m value admissible in a standard is the closest to the actual average diameter d_S in each standard.

Further, schemes of the standards of numbers G4, G5 and G6 were selected for the analysis by the computer program "Compass". The grains, which are completely satisfy limits of the circle with a diameter of 79.8 mm, accepted in GOST for standards as well as grains crossed by this circle have been included to this scheme; but their borders are clearly visible in a standard. In Figure 1, the scheme of a standard G5 is presented.

Table 1. Results of calculations of diameters of d_S and d_m grains of standards GOST 5639-82

Grain *G* No	Grain diameter d_m, *microns*			Grain diameter d_S, *microns*		
	minimum	average	maximum	minimum	average	maximum
-3	817	1000	1155	922	1129	1304
-2	577	707	817	651	798	922
-1	408	500	577	461	564	651
0	289	354	408	326	400	461
1	204	250	289	230	282	326
2	144	177	204	163	200	230
3	102,1	125	144	115,2	141	163
4	72,2	88,4	102,1	81,5	99,8	115,2
5	51,0	62,5	72,2	57,6	70,5	81,5
6	36,1	44,2	51,0	40,7	49,9	57,6
7	25,5	31,2	36,1	28,8	35,2	40,7
8	18,0	22,1	25,5	20,3	24,9	28,8
9	12,8	15,6	18,0	14,4	17,6	20,3
10	9,0	11,0	12,8	10,2	12,4	14,4
11	6,4	7,8	9,0	7,2	8,8	10,2
12	4,5	5,5	6,4	5,1	6,2	7,2
13	3,2	3,9	4,5	3,6	4,4	5,1
14	2,3	2,8	3,2	2,6	3,2	3,6

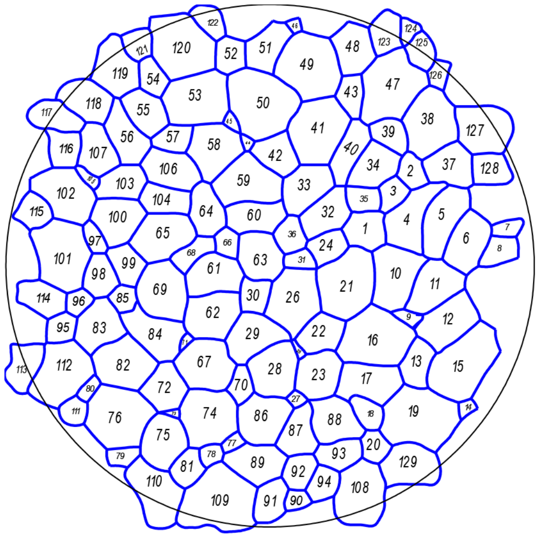


Figure 1. The scheme of standard G5 of GOST 5639-82 with enumerated grains

Grains of standards were enumerated and their areas were measured by means of the program "Compass", and then diameter d_S of each grain is determined as diameter of an equal circle; and diameters are distributed by the sizes in the scale measured for d_S, i.e. dimensional intervals (5.1…7.2 *microns*); (7.2…10.2 *microns*);…; (115.2…163 *microns*) have been accepted (see Table 1).

It has been established that in the analyzed standards the following range of diameters of flat grains is observed: in G4 standard – from G3 to G7; in G5 standard – from G4 to G9; in G6 standard – from G5 to G12. The obtained distributions of diameters of flat grains *Pk (d)* given in Table 2 have shown that the amount of the flat grains with size which is within number range (G4, G5, G6 respectively) is 35.2 … 39.6%; grains which are within previous interval with larger grains (for example, G3 for number G4) – 24,5 … 25,7%, and grains which are smaller of the subsequent interval (for example, G5 for number G4) – 20.7 … 23.3%; other grains are smaller. Thus, set of flat grains of each standard is presented generally by grains of three numbers (82 … 87%); namely, those that correspond to the range of this standard and two neighboring it.

On the basis of obtained distributions of $P_k(d)$, the average diameter of set of flat grains of each standard d_{av} was calculated by formula

$$d_{av} = \sum_{k}^{n} d_{Sk} P_k(d) \, ,$$

At that, the average value of dimensional interval was taken as d_{Sk} (for example, 141 microns for the interval G3). The calculated d_{av} is close to average d_S value of the analyzed number of G (see Table 1); and at this, d_{av} is less than d_S by 3.0 … 4.2 microns; and d_{av} is higher than d_m by 2.7 … 8 microns. The number of flat grains in 1 mm^2 is close to average value of *m* in accordance with GOST, but exceeds it a little (by 7 … 13%) according to all the standards; obviously, this is explained by the fact that when calculating, d_{av} of value d_{Si} of each interval was carried to the middle of interval.

Table 2. Distribution of diameters d_S of flat grains in standards GOST 5639-82 and parameters of structures

Grain *G* No	Range of diameters d_S in interval, micron	Distribution of diameters in standard *G4*		Distribution of diameters in standard *G5*		Distribution of diameters in standard *G6*	
		items	$P_k(d)$	items	$P_k(d)$ *	items	$P_k(d)$
3	115,2…163	13	0,245	-	-	-	-
4	81,5…115,2	21	0,396	32	0,248062	-	-
5	57,6…81,5	12	0,226	46	0,356589	67	0,257
6	40,7…57,6	6	0,113	30	0,232558	92	0,353
7	28,8…40,7	1	0,019	12	0,093023	54	0,207
8	20,3…28,8	-	-	6	0,046512	30	0,115
9	14,4…20,3	-	-	3	0,023256	11	0,042
10	10,2…14,4	-	-	-	-	4	0,015
11	7,2…10,2	-	-	-	-	2	0,008
12	5,1…7,2	-	-	-	-	1	0,004
Average diameter of grains of standard d_{av}, micron		96,4		66,3		46,9	
Standard deviation σ_d, micron		31,0		23,9		17,4	
Variation coefficient K_d, %		32,2		36,1		37,1	
Number of flat grains for1 mm^2		137		290		579	
Number of flat grains for1 mm^2 according to GOST		128		256		512	

*For standard G5, $P_k(d)$ with higher accuracy is given as they will be used in course of further calculations

The parameters characterizing degree of structure uniformity – standard deviation σ_d and coefficient of variation K_d, which have shown sufficient uniformity of structures were also calculated.

For performance of the specified and all the subsequent calculations, the computer program has been developed and applied.

Further, with the use of established P_k *(d)* as initial information, distribution reconstruction of the sizes of three-dimensional $P(D_k)$ grains, which flat sections would correspond to the studied standards, was performed. Allowing performance the specified reconstruction, the new method of determination of key parameters of three-dimensional structure of metal materials [5] has been applied for this purpose. In the method, as initial information for reconstruction, it is possible to use both distribution of diameters of flat P_k *(d)* grains and distribution of linear sections (chords) P_k *(l)* obtained as a result of application of structure of transversal lines and their crossing with borders of flat grains to the images. For distribution of $P(D_k)$, $P_k(d)$, $P_k(l)$, the same dimensional scale with *n* intervals is used.

Working formulas of method for non-uniform dimensional scale corresponding to scale of the existing standards [1, 2] and representing geometric progression with a multiplier $\sqrt{2}$ are of the form:

$$P_k(d) = \alpha_1 P(D_k)\sqrt{D_k^2 - d_{k-1}^2} + \alpha_2 P(D_{k+1})(\sqrt{D_{k+1}^2 - d_{k-1}^2} - \sqrt{D_{k+1}^2 - d_k^2}) + \ldots + \alpha_n P(D_n)(\sqrt{D_n^2 - d_{k-1}^2} - \sqrt{D_n^2 - d_k^2}); \quad (1)$$

$$P_k(l) = \beta_1 P(d_k)\sqrt{d_k^2 - l_{k-1}^2} + \beta_2 P(d_{k+1})(\sqrt{d_{k+1}^2 - l_{k-1}^2} - \sqrt{d_k^2 - l_k^2}) + \ldots \beta_n P(d_n)(\sqrt{d_n^2 - l_{k-1}^2} - \sqrt{d_n^2 - l_k^2}), \quad (2)$$

where: α_1; α_2;...; α_n – the coefficients of form of three-dimensional grains considering distinction of polyhedral form of average grain from sphere form according to ratio: $\alpha_k = P_k(d)_{grain} / P_k(d)_{sphere}$; β_1; β_2;...; β_n – the coefficients of a form of flat grains considering difference of form of average flat grain from circle form according to ratio: $\beta_k = P_k(l)_{flat\ grain} / P_k(l)_{circle}$

In paper [5], coefficients of the form α and β have been established for the austenitic recrystallized structures for the specified dimensional scale of 20 intervals. If the maximum value in this scale is accepted as equal to 1 *conditional unit of length*, the scale takes the following form:

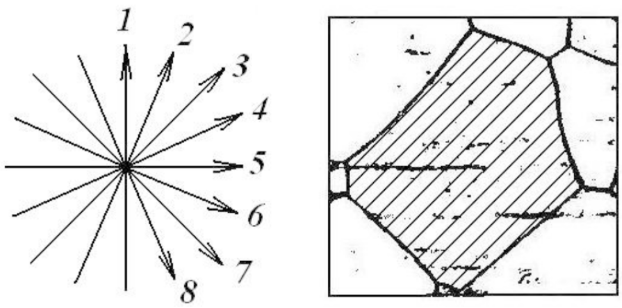
0,0010; 0,0014; 0,0020; 0,0028; 0,0039;
0,0055; 0,0078; 0,0110; 0,0156; 0,0221;
0,0312; 0,0442; 0,0625; 0,0884; 0,125; 0,177;
0,25; 0,354; 0,5; 0,707; 1.

Distributions $P_k(d)_{sphere}$ and $P_k(l)_{circle}$ are identical, it follows from simple geometrical ratios of sphere and circle. These distributions have been calculated for a circle with diameter equal to 1 *conditional unit* according to a formula:

$$P_k(l) = \sqrt{1 - l_{k-1}^2} - \sqrt{1 - l_k^2}.$$

Distributions $P_k(l)_{flat\ grain}$ and $P_k(d)_{grain}$ have been established experimentally, the first – using 40 flat grains of the different size but typical in shape; and the second – using 20 flat grains typical in shape, but of the maximum sizes observed in specimen, at the same time, accepting that each of them is the maximum flat section for that grain from which it is obtained. 8 groups of parallel equidistant transversal lines were applied in the program "Compass" at the equal angle to each other on images of each grain, as shown in Figure 2 (sufficiency of such quantity of transverses is shown in papers [5, 6]); the chords obtained as the results of crossing of lines by grains borders were measured; the maximum chord l_{max} was determined; the dimensional scale in a relative form (l_1/l_{max}; l_2/l_{max}; ... l_k/l_{max}; ... l_n/l_{max}) was composed; and chords in relative fractions according to dimensional intervals of scale were distributed. Further, average relative fractions of chords $P_k(l)_{flat\ grain}$ were calculated for 40 and 20 flat grains respectively for each dimensional interval; and also coefficients β and α were calculated.

At that, for calculation of coefficients of form α, the average flat grain distribution P_k *(l)* of 20 flat grains of the maximum sizes, typical in a form and observed on specimens was accepted as distribution *Pk (d)* of grain, inasmuch as it was shown in papers of



a b

Figure 2. The directions (1 - 8) of applying of eight groups of parallel equidistant transversal lines (a) to the flat grain and an example (b) of applying transverses of the direction 3 to grain for establishment of distribution $P_k(l)$

the author [5; 6; 8], for establishment of distribution of flat sections diameters P_k *(d)* from grain-polyhedron, it is enough to establish distribution of chords P_k *(l)* on the maximum flat section of this grain and accept it as $P_k(d)$.

Coefficients of the form *β* and *α* for the austenitic recrystallized structures are presented in Tables 3 and 4.

On the basis of the given working formula (1) for a standard G5, where flat grains are presented in 6 dimensional intervals, the equations for each $P_k(d)$ have been worked out; at that, values *D* and *d* corresponded to average values of dimensional intervals from Table 1, i.e. for example, all the flat sections of an interval (81.5 … 115.2 microns) were referred to the size of 99.8 microns.

$P_k(d)$ were the known members of the equations (see Table 2). Unknown $P(D_k)$ were calculated as a result of solution of equation system:

$$P_6(d)=\alpha_1 P_6(D)\sqrt{D_6^2-d_5^2}\ ;$$

$$P_5(d)=\alpha_1 P_5(D)\sqrt{D_5^2-d_4^2}+\alpha_2 P_6(D)(\sqrt{D_6^2-d_4^2}-\sqrt{D_6^2-d_5^2})\ ;$$

$$P_4(d)=\alpha_1 P_4(d)\sqrt{D_4^2-d_3^2}+\alpha_2 P_5(D)(\sqrt{D_5^2-d_3^2}-\sqrt{D_5^2-d_4^2})+\alpha_3 P_6(D)(\sqrt{D_6^2-d_3^2}-\sqrt{D_6^2-d_4^2})\ ;$$

……………..

$$P_1(d)=\alpha_1 P_1(D)\sqrt{D_1^2-d_0^2}+\alpha_2 P_2(D)(\sqrt{D_2^2-d_0^2}-\sqrt{D_2^2-d_1^2})+\ldots+\alpha_6 P_6(D)(\sqrt{D_6^2-d_0^2}-\sqrt{D_6^2-d_1^2})\ .$$

After substitution of values of the known components of the equations, the following system was obtained:

$$0,248062=0,676P(D_6)\sqrt{99,8^2-70,5^2}\ ;$$

$$0,356589=0,676P(D_5)\sqrt{70,5^2-49,9^2}+1,638P(D_6)(\sqrt{99,8^2-49,9^2}-\sqrt{99,8^2-70,5^2})\ ;$$

$$0,232558=0,676P(D_4)\sqrt{49,9^2-35,2^2}+1,638P(D_5)(\sqrt{70,5^2-35,2^2}-\sqrt{70,5^2-49,9^2})+ \\ +1,503P(D_6)(\sqrt{99,8^2-35,2^2}-\sqrt{99,8^2-49,9^2})\ ;$$

…………….

$$0,023256=0,676P(D_1)\sqrt{17,2^2-12,4^2}+1,638P(D_2)(\sqrt{24,9^2-12,4^2}-\sqrt{24,9^2-17,6^2})+ \\ +1,503P(D_3)(\sqrt{35,2^2-12,4^2}-\sqrt{35,2^2-17,6^2})+1,572P(D_4)(\sqrt{49,9^2-12,4^2}-\sqrt{49,9^2-17,6^2})+ \\ +2,038P(D_5)(\sqrt{70,5^2-12,4^2}-\sqrt{70,5^2-17,6^2})+3,253P(D_6)(\sqrt{99,8^2-12,4^2}-\sqrt{99,8^2-17,6^2})$$

From the first equation of the given system, $P(D_6)$ were calculated and then all the others $P(D_k)$ were calculated successively. At that, $P(D_k)$ with negative values ($P(D_3)$, $P(D_4)$, $P(D_5)$) were accepted equal to zero. The calculated $P(D_k)$ according to a method [8] were standardized according to a formula

$$P'(D_k)=P(D_k)/\sum_{k=1}^{n}P(D_k)\ .$$

Table 3. Values of coefficients of form α of three-dimensional grains

α_1	α_2	α_3	α_4	α_5	α_6	α_7	α_8	α_9	α_{10}	α_{11}	α_{12}
0,676	1,638	1,503	1,572	2,038	3,253	4,752	5,988	8,240	6,128	6,996	9,936

α_{13}	α_{14}	α_{15}	α_{16}	α_{17}	α_{18}	α_{19}	α_{20}
14,924	20,240	29,843	40,480	61,187	79,281	111,912	158,542

Table 4. Values of coefficients of form *β* of flat grains

β_1	β_2	β_3	β_4	β_5	β_6	β_7	β_8	β_9	β_{10}	β_{11}	β_{12}
0,600	1,857	1,609	1,821	2,508	3,474	4,543	5,765	7,169	7,588	10,021	14,015

β_{13}	β_{14}	β_{15}	β_{16}	β_{17}	β_{18}	β_{19}	β_{20}
19,864	28,110	37,995	59,734	78,770	110,099	155,608	220,229

The results of calculations given in Table 5 have shown that in the reconstructed three-dimensional structures with numbers G4 and G6 of grain are presented only at four-dimensional intervals, structure G5 – at three-dimensional intervals; whereas, at their flat cuts, i.e. in standards, the range of the sizes of flat grains is from five (for *G4*) to eight (for *G6*) dimensional intervals (see Table 2). The amount of the flat grains with the size which corresponds to number range (G4, G5, G6 respectively) is larger than for set of their flat sections in a standard and is 43.3 … 52.0%; the amount of grains which were in the previous interval with larger grains is also larger than their flat sections in a standard – 34.5 … 37.3%, and the amount of grains of smaller size of the subsequent interval is significantly smaller – 8.7…16.6%; and even the smaller amount of them is in the last interval with the finest grains. Thus, the reconstructed set of three-dimensional grains is generally presented by grains of two numbers (81 … 87%), namely, those that correspond to the range of this number of grain and numbers with larger grains.

Average diameter of set of three-dimensional grains D_{av} exceeds the average diameter of their flat sections d_{av} by 13…17% and is very close to the upper bound of range of a standard diameters; and indicators of varied grain sizes are significantly lower for three-dimensional structure in comparison with plane structure of standards: the standard deviation is less by 15…24%, variation coefficient is lower by 25…35%, i.e. the volume structure is much more uniform than it is seen in a flat cut.

According to number of grains in 1 mm^3, volume structures of all the standards are close to the parameter N_v GOST 5639-82 (see Table 5), the difference is 1 … 6%.

Thus, the researches have shown the following:

- in course of determination of grain size in accordance with GOST 5639-82 it is necessary to consider that the average diameter of d_m grains is less by 1.1287 times (i.e. on ~ 13%) than objective diameter of d_S equal to average grain of circle;
- the reconstructed volume structures, which flat sections correspond to standards of G4, G5, G6 GOST 5639-82, are more uniform and are of larger average diameter (by 13 … 17%) than their flat cuts presented by standards that should to be considered in the researches of influence of grain size on properties of steel products.

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Table 5. The reconstructed distributions of diameters *P'(D)* of three-dimensional grains, the corresponding sets of flat grains of standards GOST 5639-82 and parameters of volume structures

Number of grain *G*	Diameters range *D* In the interval, *micron*	Diameters distribution *P'*(*D*), calculated by standard *G4*	Diameters distribution *P'*(*D*), calculated by standard *G5*	Diameters distribution *P'*(*D*), calculated by standard *G6*
3	115,2…163	0,345	-	-
4	81,5…115,2	0,520	0,367	-
5	57,6…81,5	0,087	0,467	0,373
6	40,7…57,6	0,048	0, 166	0,433
7	28,8…40,7	-	-	0,107
8	20,3…28,8	-	-	0,087
9	14,4…20,3	-	-	-
10	10,2…14,4	-	-	-
11	7,2…10,2	-	-	-
12	5,1…7,2		-	-
Average diameter D_{av}, *micron*		109,1	77,8	53,8
Standard deviation σ_D, *micron*		26,4	18,2	14,8
Variation coefficient K_D, %		24,2	23,4	27,6
Grains amount in 1 mm^3, *pcs*		1471	4058	12271
Average number of grains in 1 mm^3 according to GOST, N_v, *pcs*		1448	4096	11585

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Peculiarities of protective coating of constructional details with powder obtained from industrial wastes

Oleksandr Povstyanoy

PhD in Technical Sciences, associate professor
Lutsk National Technical University
Lutsk, Ukraine
E-mail: povstjanoi@mail.ru

Nataliia Imbirovuch

PhD in Technical Sciences, associate professor
Lutsk National Technical University
Lutsk, Ukraine
E-mail: povstiana@yandex.ru

Yuliia Kuts

Ph.D. student
Lutsk National Technical University
Lutsk, Ukraine
E-mail: pywochek@gmail.com