ACTA STEREOL 1991; 10/1: 73-82
STERMAT'90
ORIGINAL SCIENTIFIC PAPER

GRAIN SIZE DISTRIBUTION

Marian Maliński, Jan Cwajna and Jacek Chrapoński

Institute of Materials Science and Engineering,
Silesian University of Technology
Graniczna 16, PL-40-018 KATOWICE, Poland

ABSTRACT

Statistical and geometrical distributions of the normalized area of plane grain cross-section for model microstructures with homogeneous grain size, built of truncated octahedra, Williams β-tetrakaidecahedra and rhombic dodecahedra have been presented in this work. These distributions can be taken as a basis to some new grain size homogeneity criteria and inhomogeneity measures. Accuracy of the mentioned measures was verified by the analysis of the stereological parameters of 2-D and 3-D computer-simulated microstructures with various inhomogeneity. It was stated-out in experimental studies on the 0H16 steel grade normal grain growth that the geometrical measure of the mean grain size is more sensitive to the heat treatment parameters than the statistical one. These studies and the model analysis indicate that the characteristic features of the polycrystalline microstructure can be described more accurately by geometrical distributions than by the statistical ones.

Key words: grain size, quantitative metallography, computer simulation and modelling.

INTRODUCTION

In our previous works (Maliński,1985; Cwajna et al., 1986; Cwajna et al., 1987a; Cwajna et al., 1987b; Maliński et al., 1988) some new stereological criteria and inhomogeneity measures were presented. They were based on the results of 2-D and 3-D simulation experiments with equilibrium model structures built of truncated octahedra (TO), Williams' β-tetrakaidecahedra (WT) and rhombic dodecahedra (RD) of the same size. For these microstructures distributions of the following stereological parameters of grain size and shape in 2-D space were evaluated:

- normalized plane section area (Maliński, 1985; Cwajna et al., 1986; Maliński et al., 1988),
- normalized plane section perimeter (Maliński, 1985),
- normalized intercept length chord of plane section (Cwajna et al.1987a; Cwajna et al. 1987b),
- shape factor of plane section (Maliński, 1985),
- number of sides of plane section (Maliński, 1985).

To verify the practical applicability of the grain size homogeneity criteria and inhomogeneity measures, a set of tests of grain growth was performed with OH16 ferritic steel grade heat-treated in different temperature and time conditions.
In these investigations the grain size was being characterized by standard distributions: frequency = f(normalized plane section area); as well as the distributions (relationships) of the type of area fraction = f(normalized plane section area). That was because it was revealed in our previous works (Cwajna et al., 1989 and Maliński et al., 1989) concerning the quantitative evaluation of dispersive phase particle size, that the distributions of $A_A = f(A/A_{max})$ type are more suitable to the description of the inhomogeneous microstructures. Above all, they make it possible to asses properly whether the presence of large grains in the microstructure is incidental or it is the microstructural feature.

In the $A_A = f(A/A_{max})$ distributions fractions of the grain area in successive size classes are equivalent to the probability (frequency) calculated with the use of geometrical definition (measure) of probability. It is suggested to call these distributions the "geometrical" ones, in contrast to the "statistical" distributions of the frequency = $f(A/A_{max})$ type.

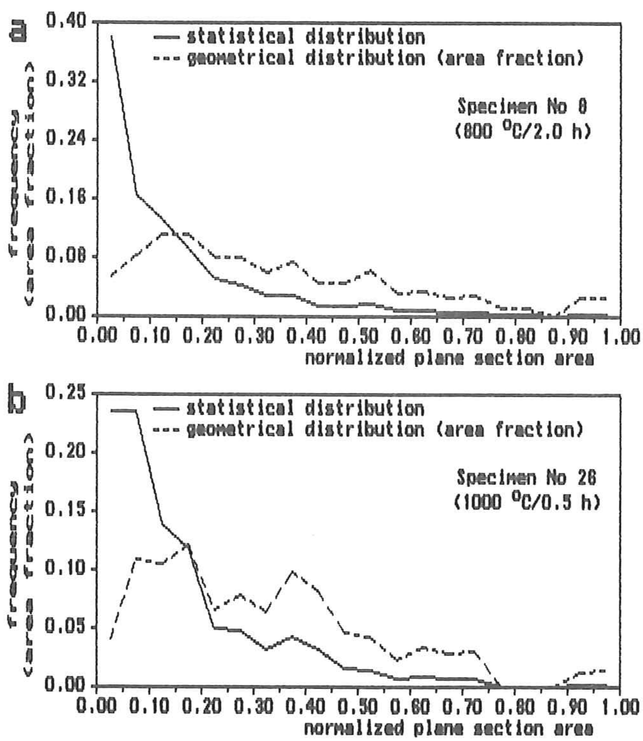


Fig.1. Statistical and geometrical distributions of the normalized plane section area (a - specimen No 8, b - specimen No 26).

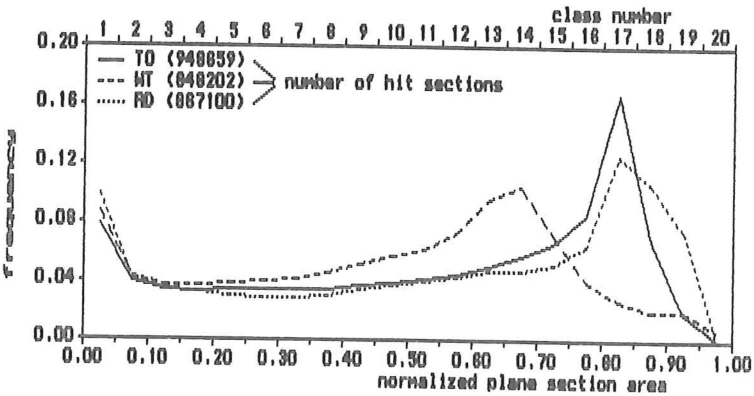
All the experimental works were performed with the MORPHOPERICOLOR automatic image analyser, with an application of morphological image modification techniques described in (Szala, 1990), and other software developed in our Stereological Laboratory.
It has been found that the analysis of either types of distributions can led to equivocal conclusions: The statistical distributions indicate that in normal grain growth in the polycrystalline microstructure fine grains are predominant (small value of grain plane section area) (fig.1a). On the other hand, the analysis of the

geometrical distributions shows that these are not the predominant cross-sections (fig 1b.).

Moreover, the geometrical distributions more distinctively demonstrate an effect of annealing temperature and time on the grain growth. However, for these distributions neither mean grain size nor its inhomogeneity measures have been hitherto proposed yet, that means a considerable limitation in their applicability in the investigations of the grain growth kinetics. That was the reason why, taking as a basis the computer simulation studies, the attempt was made to work out the mentioned measures and to verify objectively the homogeneity criteria and inhomogeneity measures of grain size.

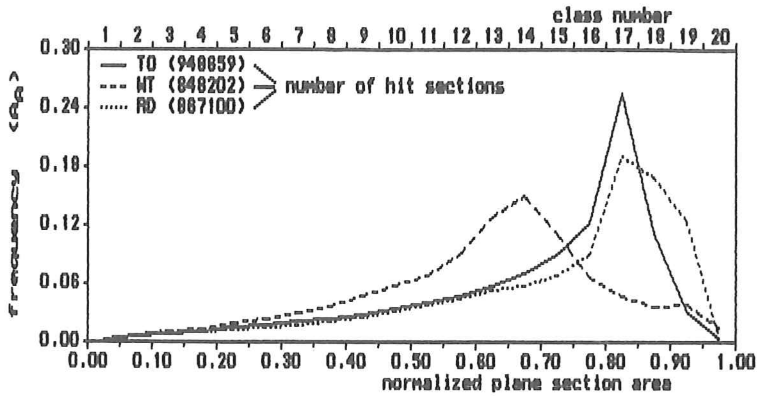
STATISTICAL AND GEOMETRICAL GRAIN SIZE HOMOGENEITY CRITERIA AND INHOMOGENEITY MEASURES

With an application of computer simulation technique described in detail in (Maliński, 1985), for the model microstructures (TO, WT and RD) the statistical and geometrical distributions with the random sections number of 1000000 were determined (fig.2 and 3). The geometrical distributions



Class number	Class interval	Distribution function [%] TO	WT	RD
1	0,00 ÷ 0,05	7,82	8,77	9,91
2	0,05 ÷ 0,10	11,76	13,02	14,19
3	0,10 ÷ 0,15	15,20	16,76	17,66
4	0,15 ÷ 0,20	18,48	20,45	20,90
5	0,20 ÷ 0,25	21,97	24,26	23,93
6	0,25 ÷ 0,30	25,35	28,27	26,85
7	0,30 ÷ 0,35	28,77	32,40	29,74
8	0,35 ÷ 0,40	32,19	37,01	32,75
9	0,40 ÷ 0,45	35,84	42,10	36,15
10	0,45 ÷ 0,50	39,76	47,91	39,89
11	0,50 ÷ 0,55	43,96	54,03	43,92
12	0,55 ÷ 0,60	48,44	61,35	48,23
13	0,60 ÷ 0,65	53,44	70,91	52,88
14	0,65 ÷ 0,70	59,16	81,30	57,62
15	0,70 ÷ 0,75	65,79	88,50	62,81
16	0,75 ÷ 0,80	74,30	92,65	69,17
17	0,80 ÷ 0,85	90,95	95,35	81,75
18	0,85 ÷ 0,90	97,86	97,29	92,34
19	0,90 ÷ 0,95	99,79	99,20	99,72
20	0,95 ÷ 1,00	100,00	100,00	100,00

Fig.2. Statistical distributions of the normalized plane section area of the model polyhedra.



Class number	Class interval	Distribution function [%] TO	WT	RD
1	0.00 ÷ 0.05	0.26	0.32	0.31
2	0.05 ÷ 0.10	0.80	0.99	0.89
3	0.10 ÷ 0.15	1.60	1.99	1.60
4	0.15 ÷ 0.20	2.66	3.37	2.71
5	0.20 ÷ 0.25	4.12	5.21	3.96
6	0.25 ÷ 0.30	5.85	7.56	5.44
7	0.30 ÷ 0.35	7.91	10.49	7.17
8	0.35 ÷ 0.40	10.29	14.13	9.24
9	0.40 ÷ 0.45	13.17	18.83	11.98
10	0.45 ÷ 0.50	16.62	24.64	15.17
11	0.50 ÷ 0.55	20.72	31.51	19.07
12	0.55 ÷ 0.60	25.50	40.53	23.62
13	0.60 ÷ 0.65	31.31	53.30	28.97
14	0.65 ÷ 0.70	38.48	68.37	34.86
15	0.70 ÷ 0.75	47.42	79.48	41.78
16	0.75 ÷ 0.80	59.68	86.28	50.86
17	0.80 ÷ 0.85	85.18	90.95	70.81
18	0.85 ÷ 0.90	96.34	94.57	87.02
19	0.90 ÷ 0.95	99.63	98.52	99.58
20	0.95 ÷ 1.00	100.00	100.00	100.00

Fig.3. Geometrical distributions of the normalized plane section area of the model polyhedra.

developed for model, homogeneous microstructures makes it possible to apply the homogeneity criteria and inhomogeneity measures, given in Table 1, to the grain size studies. Another tables, dealing with the inference about the grain size inhomogeneity from statistical distributions, were presented in (Maliński, 1985; Cwajna et al., 1986; Cwajna et al., 1987a).

It is significant that the statistical distributions for model microstructures with homogeneous grain size indicate relatively large number of grains in the first few classes of size (fig.2), that is also true for real materials (fig.1a). The geometrical distributions show, however, that it is not a predominant feature of the microstructure (fig.1b and 3).

VERIFICATION OF THE WORKED-OUT GRAIN SIZE HOMOGENEITY CRITERIA AND INHOMOGENEITY MEASURES

The verification of both statistical and geometrical grain size homogeneity criteria and inhomogeneity measures was carried out by studying of a few computer simulated microstruc-

Table 1. Criteria and measures of grain size inhomogeneity.

KOLMOGOROV'S test of goodness of fit	
Statistical Criterion $\lambda = \mathrm{sus}_x \left| F_S(x_i) - \frac{n_i^{cu}}{n} \right| n$	Geometrical Criterion $\lambda = \sup_x \left| F_G(x_i) - (A_A)_i^{cu} \right| n$
Notes	$F_S(x_i)$ - hypothetical value of distribution function for i^{th} class (fig.2) $F_G(x_i)$ - hypothetical value of distribution function for i^{th} class (fig.3) n_i^{cu} - cumulative size to i^{th} class $(A_A)_i^{cu}$ - cumulative area fraction to i^{th} class n - number of analysed grains λ_α - critical value of KOLMOGOROV'S distribution α - significance level ($\alpha = 0.05$; $\lambda_\alpha = 1.358$) I N F E R E N C E a) $\lambda < \lambda_\alpha \longrightarrow$ grain size homogeneity b) $\lambda \geq \lambda_\alpha \longrightarrow$ grain size inhomogeneity
Statistical (geometrical) Measure $M_{S,G} = \lambda - \lambda_\alpha$ I N F E R E N C E Grain size inhomogeneity grows whith the $M_{S,G}$ growth	

tures. A characteristics of the microstructure B, that consists exclusively of truncated octahedra and is the best approximation of the homogeneous microstructure, as well as the most inhomogeneous microstructure A (with the highest coefficients of the grain volume variability), are shown in Table 2.
The results of the statistical tests indicate that there is a significant difference in the grain size inhomogeneity between the investigated microstructures (fig.4 and 5).

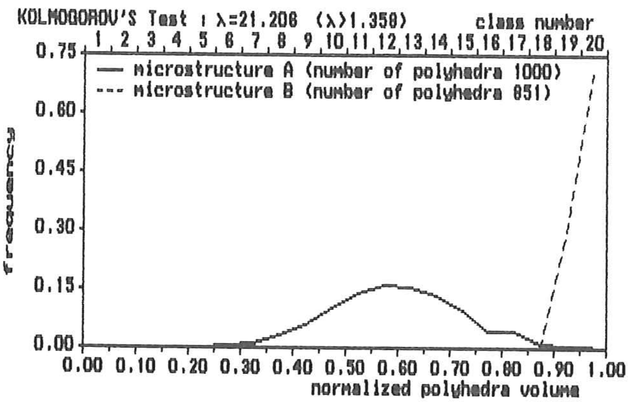


Fig.4. The results of the application of Kolmogorov test to the comparison of the statistical distributions of the normalized volume of the polyhedra of A and B microstructure.

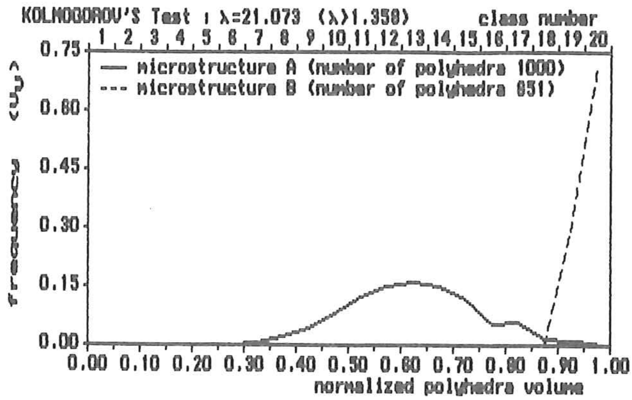


Fig.5. The results of the application of Kolmogorov test to the comparison of the geometrical distributions of the normalized volume of the polyhedra of A and B microstructure.

Table 2. Selected parameters characterizing the computer simulated microstructures.

Parameter	Distribution: Statistical		Distribution: Geometrical	
	Mean value	Coefficient of variation	Mean value	Coefficient of variation
Microstructure A				
Normalized polyhedra volume	0.6016	20.30 %	0.6263	19.54 %
Shape factor 3-D	0.9166	1.73 %	0.9194	1.76 %
Number of faces	15.46	14.70 %	15.77	14.33 %
S_V = 3.2422 [mm^2/mm^3] N_V = 994.8 [mm^{-3}]				
Microstructure B				
Normalized polyhedra volume	0.9595	1.52 %	0.9604	2.37 %
Shape factor 3-D	0.9538	0.06 %	0.9750	< 0.01 %
Number of faces	14.00	0 %	14.00	0 %
S_V = 1.7029 [mm^2/mm^3] N_V = 839.8 [mm^{-3}]				

Both statistical and geometrical distributions of the grain plane section area for the investigated model microstructures are given in fig.6. The conclusions that can be drawn from the application of the developed grain size homogeneity criteria and inhomogeneity measures for the 2-D space (fig.6) are entirely consistent with those taken from the direct comparison of the distributions describing the microstructure of the generated microstructures in the 3-D space (fig.4 and 5).

Therefore, it can be assumed that the simulation studies have confirmed both accuracy and practical applicability of the proposed criteria and measures. The final results of the quantitative evaluation of the grain size inhomogeneity depends to great extent on the homogeneous microstructure model selected to the comparison with the investigated microstructure.

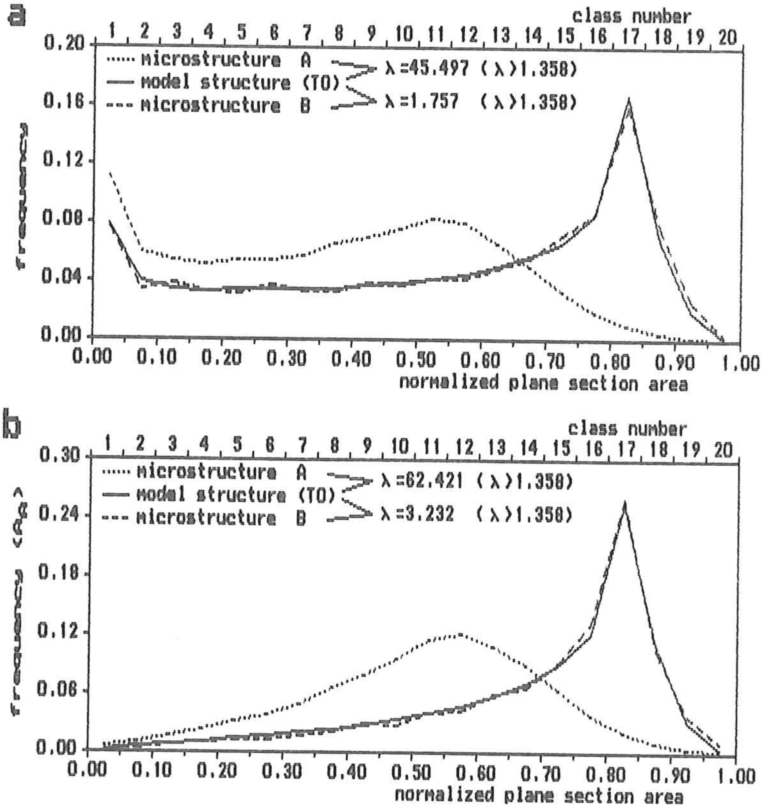


Fig.6. The results of the application of Kolmogorov test to the comparison of the statistical (a) and geometrical (b) distributions of the normalized plane section area for computer generated microstructure A and model structure TO as an example.

EXAMPLES OF AN APPLICATION OF THE GRAIN SIZE HOMOGENEITY CRITERIA AND INHOMOGENEITY MEASURES IN THE STUDIES OF 0H16 STEEL GRADE GRAIN GROWTH

In the course of annealing of the specimens of 0H16 steel grade in the temperature range of 750 ^oC to 1100 ^oC for 0.5 to 10 h the normal grain growth have been revealed. As a result of this process the mean grain size has increased while the grain size inhomogeneity dropped in the ranges given in Table 3.

Table 3. Variability range of the grain size distribution parameters and inhomogeneity measures in the specimens of 0H16 steel grade.

Distribution type	A/A_{max}	$v(A/A_{max})$ [%]	Inhomogeneity Measure TO	WT	DR
Statistical	2334.0 ÷ 22816.5	72.50 ÷ 160.10	7.539 ÷ 27.938	6.492 ÷ 27.367	7.484 ÷ 27.036
Geometrical	5911.5 ÷ 62199.5	53.98 ÷ 120.36	7.703 ÷ 30.668	6.102 ÷ 30.037	8.155 ÷ 30.817

A significantly wider variability range of the grain size geometrical measure testifies its higher sensitivity to the annealing parameters variations than that of the statistical measure. In fig.7 the grain size characteristics of the specimen with the most irregular grain is given and in fig.8 that of the specimen with the most homogeneous grain structure. The analysis of the statistical and geometrical distributions presented in these figures can lead to a conclusion that the geometrical distributions reflect better the fine grain dissolution and large grain growth at the annealing.

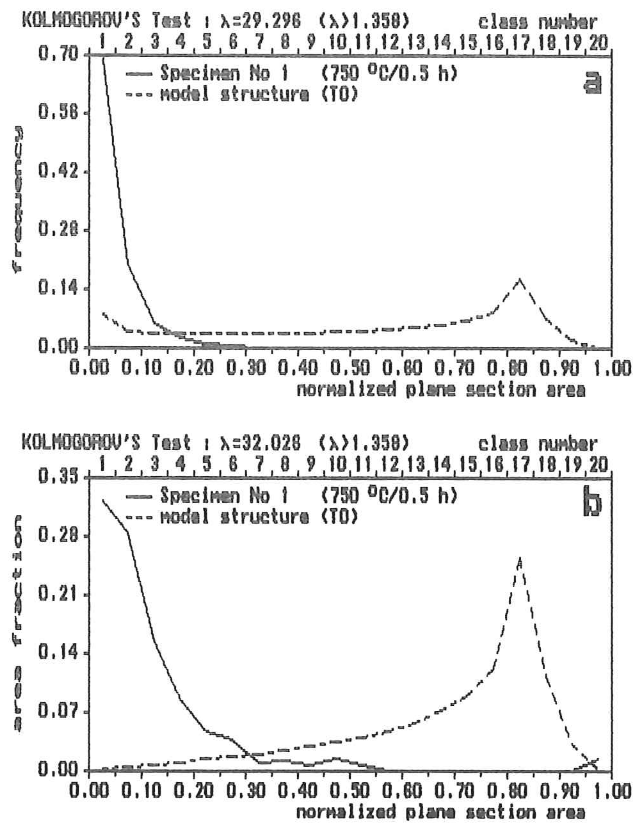


Fig.7. The results of the application of Kolmogorov test to the comparison of the statistical (a) and geometrical (b) distributions of normalized plane section area for the model structure TO and microstructure 0H16 steel annealed at 750 °C for 0.5 h

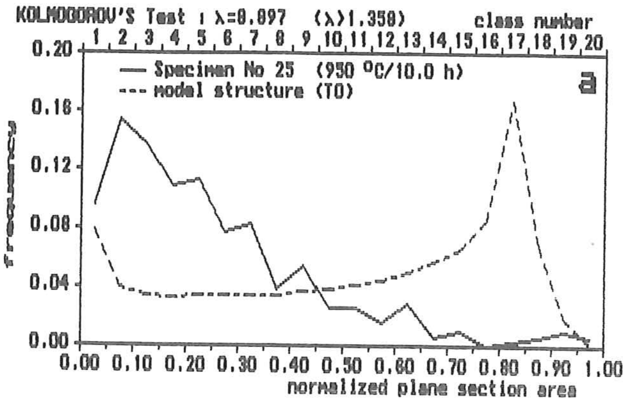
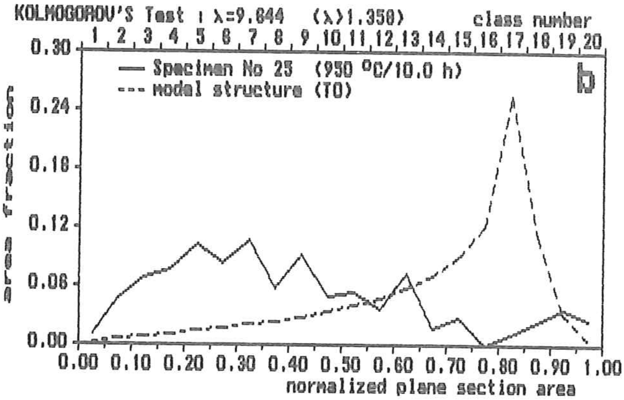


Fig.8. The results of the application of Kolmogorov test to the comparison of the statistical (a) and geometrical (b) distributions of normalized plane section area for the model structure TO and microstructure 0H16 steel annealed at 950 ^{o}C for 10.0 h



The results of the comparison of the distributions of grain size in the specimens of similar microstructure depend on the choice of the distributions being compared (fig.9 and 10). In the carried out comparative studies all possible combinations of significance and non-significance of the differences between the particular types of the distribution. That indicates that statistical and geometrical distributions contain significantly different information concerning the investigated microstructure.

Therefore, it seems necessary to supplement the hitherto applied description of the grain size by means of the statistical distribution of the normalized area of the grain plane

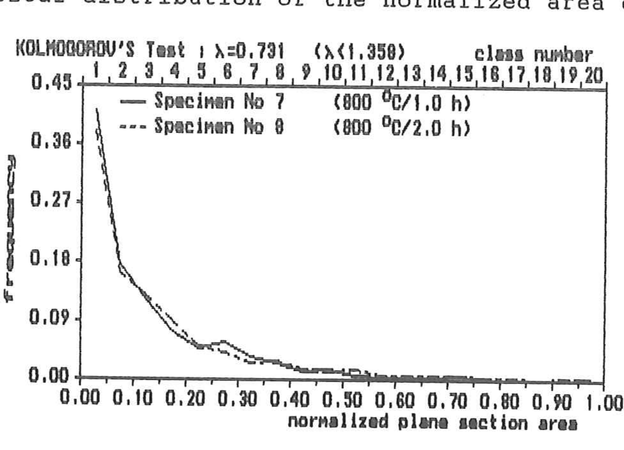


Fig.9. The result of the application of Kolmogorov test to the comparison of the statistical distributions of the normalized plane section area for the selected specimens of 0H16 steel

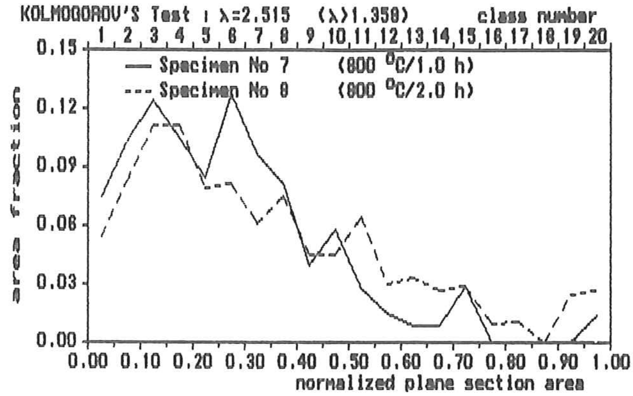


Fig.10. The result of the application of Kolmogorov test to the comparison of the geometrical distributions of the normalized plane section area for the selected specimens of 0H16 steel

section with the geometrical distribution of this stereological parameter, and that is the main conclusion drawn from the performed studies.

REFERENCES

Cwajna J, Maciejny A, Maliński M. Stereological criteria of grain size homogeneity. In: Proc. 7th Riso International Symposium on Metallurgy and Materials Science, Roskilde, Denmark, 1986: 273-278.

Cwajna J, Maciejny A, Maliński M. Stereological criteria and measures of structural stability of one-phase polycrystalline materials. In: Proc. ICS VII, Caen-France 1987a, Acta Stereol., 1987; 6/III: 261-266.

Cwajna J, Maliński M, Maciejny A. Stereological criteria of grain size homogeneity of polycrystalline materials. In: Proc. XIIth Conference on Physical Metallurgy. Katowice-Kozubnik 1987b; V.2: 205-210.

Cwajna J, Maliński M, Szala J. Quantitative metallography of high-speed steels. Acta Stereol., 1989; 7/2: 121-136.

Maliński M. Stereological criteria of grain size homogeneity of one-phase materials. Doctor's Thesis. Silesian University of Technology, Faculty of Metallurgy. Katowice, Poland, 1985.

Maliński M, Cwajna J, Maciejny A. Stereological criteria and measures of grain size homogeneity. Papers of Commission of Metallurgy and Foundry, Cracow 1988; Metallurgy No 35: 43-56.

Maliński M, Cwajna J, Richter J. Carbide particles size and shape in large diameter high-speed steel bars. In: Proc. 5th European Congress for Stereology. Freiburg 1989, Acta Stereol., 1989; 8/2: 269-274.

Szala J, Maliński M. Some examples of modifying the binary images. In: Proc. 3rd Conference "Stereology in Materials Science", STERMAT '90, Cracow-Katowice, October 1990; 278-281.