www.czasopisma.pan.pl PAN www.journals.pan.pl

Arch. Metall. Mater. **62** (2017), 4, 2015-2019

DOI: 10.1515/amm-2017-0300

H. ADRIAN*[#] K. WIENCEK*

AUSTENITE GRAIN SIZE ESTIMTION FROM CHORD LENGTHS OF LOGARITHMIC-NORMAL DISTRIBUTION

Linear section of grains in polyhedral material microstructure is a system of chords. The mean length of chords is the linear grain size of the microstructure. For the prior austenite grains of low alloy structural steels, the chord length is a random variable of gamma- or logarithmic-normal distribution. The statistical grain size estimation belongs to the quantitative metallographic problems. The so-called point estimation is a well known procedure. The interval estimation (grain size confidence interval) for the gamma distribution was given elsewhere, but for the logarithmic-normal distribution is the subject of the present contribution. The statistical analysis is analogous to the one for the gamma distribution.

Keywords: Austenite grain size, linear section methods, modelling

1. Introduction

One of the most important factors affecting the phase transformations kinetics of undercooled austenite and mechanical properties of phase transformations products of heat treated structural steels is the austenite grain size. It depends on the temperature of austenitising and a chemical composition of steel. Lower austenite grain size decreases the hardenability of steel but improves the mechanical properties of quenched and tempered steels. In order to protect the steel against undesirable grain growth the microalloying elements (MA), such as Ti, Nb, V, showing high chemical affinity to interstitial (C, N) are added [1]. Compounds forming during reactions between MA and interstitials, carbides, nitrides and carbonitrides inhibit the austenite grain growth at high temperature. The relationship between the mean radius of austenite grains, R and parameters of compounds precipitations, (volume fraction, V_V, and mean radius, r) is described by the well known Smith-Zener equation [2]:

$$R = \frac{4r}{3V_V} \tag{1}$$

Parameters of compounds controlling austenite grain growth can be calculated using thermodynamic [3,4] and kinetic [5-7] models of compounds precipitation process.

Knowledge of austenite grain size is essential for prediction of heat treatment effect and for estimation of grain size different methods are used. One of them is measurements of chord lengths of austenite grains. In metallography, the chord lengths measurement for polyhedral microstructures may be reduced to simple counting measurements of n chords (or it end points) along a test line of length L – the so-called chord counting method [8-10,17]. In the prior austenite microstructure of some low alloy structural steels, the chord length is a random variable of logarithmic-normal distribution. The mean chord length, interpreted as linear austenite grain size, is a stereological characteristics of the material microstructure. The grain size interval estimation (confidence interval) is the main subject of this contribution. The statistical analysis is analogous to this one for the gamma distribution given in [10]. The aim of present work was development of accuracy estimation of austenite grain size using chord length measurement method in low alloy steels, where austenite chord lengths distribution is logarithmic-normal. The article includes two main parts. The first one presents the logarithmic-normal distribution; a simple approximate confidence interval for the linear grain size is proposed. The statistical independent grain chord lengths is of fundamental meaning. The second part contains the structural steels prior austenite grain chord lengths having the logarithmic-normal distribution [11-16]. The length of sequent chords were measured along test lines. With regard to the possibility of grain size interval estimation method given in part one, the statistical independence of the sequent chord lengths are analysed.

2. Statistics

If for a non-negative random variable X, the random variable $\ln X$ has the normal distribution with statistical parameters $m_{\ln X}$ and $\sigma_{\ln X}$, then X has the logarithmic-normal distribution with probability density function (PDF):

* AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, AL. A. MICKIEWICZA 30, 30-059 KRAKOW, POLAND

Corresponding author: adrian@agh.edu.pl

$$f(x) = \frac{1}{x\sigma_{\ln X}\sqrt{2\pi}} \exp\left(-\frac{\left(\ln x - m_{\ln X}\right)}{2\sigma_{\ln X}^2}\right) \quad x \in [0,\infty) \qquad (2)$$

and statistical parameters m_X and σ_X [8]. The PDF $f(x)$ is unimodal of positive skewness. The parameters: $\sigma_{\ln X}$ and the variation coefficient $v_X = \sigma_X / m_X$ fulfill the equation [8]:

$$\sigma_{\ln X}^2 = \ln\left(1 + v_X^2\right) \qquad (3)$$

For n statistical independent values (realizations) of X, i.e., the x_i ($i = 1,\ldots,n$), the arithmetic mean

$$\bar{x}_n = \frac{1}{n}\sum_{i=1}^{n} x_i \qquad (4)$$

is a value of the random variable (random mean) $\overline{X}_n$ which has a logarithmic-normal distribution with the PDF $f(\bar{x}_n)$, the mean m_X and standard deviation $\sigma_X/\sqrt{n}$.

The random variable $\overline{X}_n$ is a statistical estimator for m_X. For given $\overline{x_n}$ the point estimation is:

$$m_x \approx \overline{x_n} \qquad (5)$$

Again, the normally distributed random variable $\ln\overline{X}_n$ leads to the confidence interval for m_X. In the simplest case when the parameter $\sigma_{\ln X}$ is known and for sufficiently large n (n>50), an approximative confidence interval for m_X may be written:

$$\left(\overline{X}_n \exp\left(-u_\alpha \frac{\sigma_{\ln X}}{\sqrt{n}}\right), \overline{X}_n \exp\left(u_\alpha \frac{\sigma_{\ln X}}{\sqrt{n}}\right)\right) \qquad (6)$$

where u_α has the usual statistical meaning ($\alpha < 0{,}10$).

For given n and α, because of $\overline{X}_n$, the confidence interval is a random variable. The end points of the confidence interval in Eq. (6) are random variables with the means a_1 and a_2 respectively. The a_1, a_2 as functions of n characterise the Eq. (6) confidence intervals. As example, these functions for $m_X = 12$ and $\sigma_{\ln X} = 0{,}5$ (a typical values for the austenite chord lengths of the analyzed steels in chapter 3 – Experimental) are shown in Fig. 1. The figure shows that up to ca. $n = 200$ the length of the interval $[a_1, a_2]$ decreases rapidly; then (here up to $n = 1000$) it converges slowly to m_X.

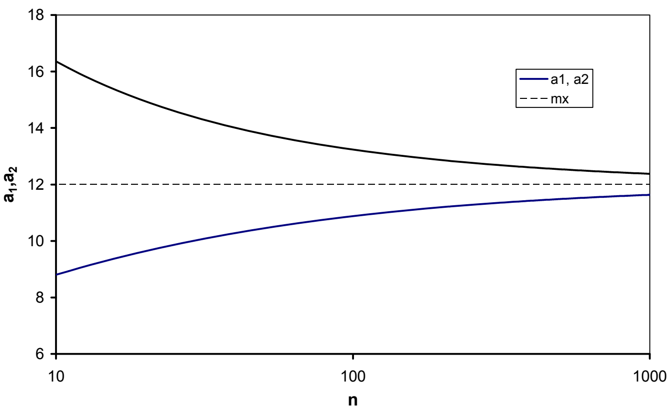

Fig. 1. The a_1, a_2 variables as function of chord number n for the m_X – confidence intervals of typical chord lengths of logarithmic-normal distribution ($m_X = 12$, $\sigma_{\ln x} = 0{,}5$)

3. Experimental

The prior austenite microstructure of low alloy structural steels (of different chemical composition and heat treatment) was investigated in [11-13]). The quantitative metallography was made on steel specimen polished surface (after etching in saturated aqueous picric acid solution, Fig. 2) by linear sections of the microstructure on sequent chords along the test line.

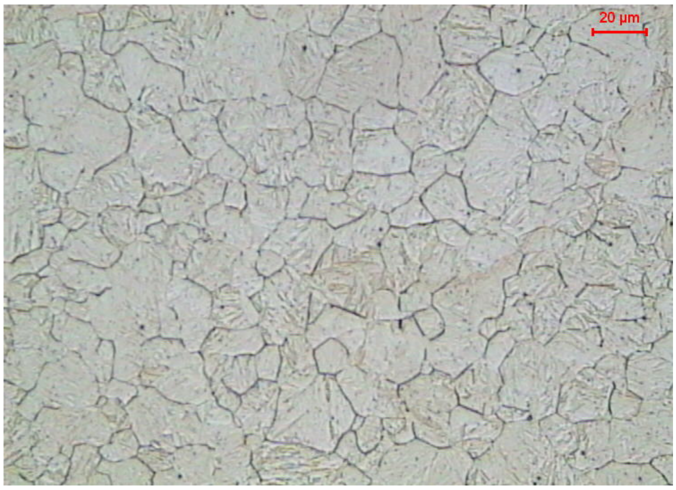

Fig. 2. Typical prior austenite microstructure of a structural steel

For each steel the length of N ($N > 990$) chords was measured. The measurement results are $x_1, x_2, \ldots, x_N$. For the data sets the austenite chord lengths distribution correspond to the logarithmic-normal one given in Eq. 2, [11-13], Fig. 3. Here, for the subsequent statistical analysis 18 homogeneous data-sets (denoted as L1,...,L18) are chosen (Table 1).

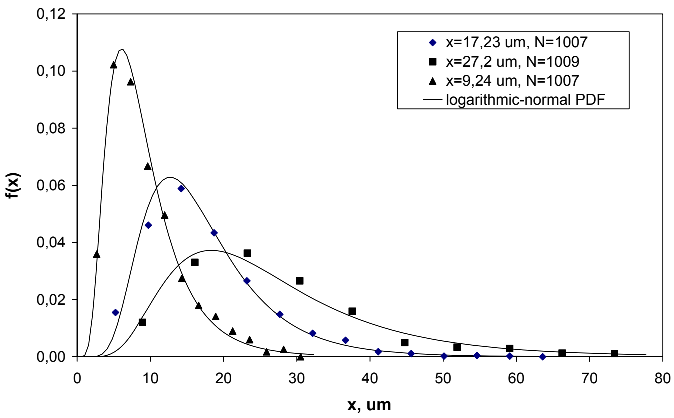

Fig. 3. Austenite grain chord length distribution (PDF $f(x)$) for different grain size $x = \bar{x}_N$ and the approximation with logarithmic-normal PDFs

In Table 1, for given data-set of N elements, the austenite chord lengths characteristics are determined, in particular, the statistical parameters (the arithmetic mean $\bar{x}_N$, the standard deviation s_x and the variation coefficient $v_x = s_x/\bar{x}_N$) and then, the parameter $s_{\ln x}$ of the suitable logarithmic-normal distribution –

TABLE 1

Austenite grain chord characteristics

Data set	$\overline{x}_N$, µm	s_x, µm	v_x, µm[o]	$s_{\ln x}$, µm[o]	N
L1	7,23	3,58	0,50	0,47	1010
L2	7,25	3,53	0,49	0,46	1006
L3	8,54	3,96	0,46	0,44	1016
L4	9,24	5,26	0,57	0,53	1007
L5	9,79	5,96	0,61	0,56	1006
L6	10,75	6,40	0,60	0,55	1007
L7	11,58	5,78	0,50	0,47	1013
L8	11,82	6,91	0,58	0,54	1018
L9	11,97	6,13	0,51	0,48	1002
L10	12,02	6,71	0,56	0,52	995
L11	12,22	7,45	0,61	0,56	999
L12	12,42	5,69	0,46	0,44	1011
L13	13,19	6,34	0,48	0,46	1007
L14	13,65	7,05	0,52	0,49	1009
L15	13,75	6,41	0,47	0,44	1007
L16	17,23	8,19	0,48	0,45	1007
L17	20,12	11,34	0,56	0,53	1044
L18	27,20	14,90	0,55	0,51	1009

calculated using Eq. (3) for the empirical variation coefficient v_x. From Table 1 it results:

(i) The changing area of $\overline{x}_N$ is relatively large, the values belong to the interval [7,23; 27,20 in µm] – it is an austenite of medium grain size [17];

(ii) The v_x – parameter is approximately constant, it values belong to the interval [0,46; 0,61 in m^0] (it is similar to the v_x – properties of the austenite chords in [10];

(iii) From Eq. (3) it results that the $s_{\ln x}$ – parameter is also approximately constant, it values belong to the interval [0,44; 0,56 in m^0], Fig. 4. The arithmetic mean $<s_{\ln x}>$ calculated for the $s_{\ln x}$ – values of the particular data-sets is $<s_{\ln x}> = 0{,}50$.

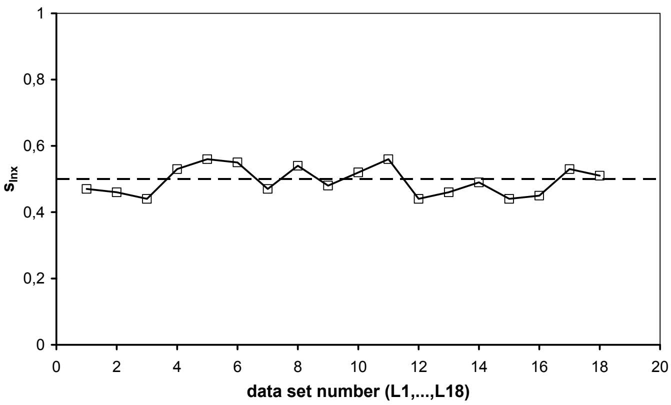

Fig. 4. Austenite grain chord length parameter $s_{\ln x}$ in relation of the number of data-sets L1,...,L18

3.1. Independence of chord lengths

The statistical independence of the length of sequent chords (along a test line) is a condition for the austenite grain size interval estimation by Eq. (6).

For the data-sets L1,...,L18 statistical independence of chord lengths was analysed by the random mean $\overline{X}_n$ distribution (Chapter 2 – Statistics). If the austenite sequent chord lengths along the test line: $x_1, x_2, \ldots, x_N$ are independent values (realizations) of the logarithmic-normally distributed random variable X, then, for a given $n < N$, the arithmetic mean $\overline{x}_n$ is a value of the logarithmic-normally distributed random mean $\overline{X}_n$. The statistical independence of the chord lengths may be analysed by the PDF $f(\overline{x}_n)$ of the random mean $\overline{X}_n$. In a data-set for given number n (n=2,3,4,5), there are at most r_n disjoint subsets of n elements. The arithmetic mean distribution of the r_n subsets are characterised by the empirical PDF $f(\overline{x}_n)$. The chi-square test for representative data-sets shows that the empirical PDFs $f(\overline{x}_n)$ are consistent with the corresponding PDF of the logarithmic- normal distribution of $\overline{X}_n$. As an example, fig. 5 shows the comparison of empirical $f(\overline{x}_n)$ functions with the corresponding logarithmic-normal functions for the data set L6 ($N = 1007$; $n = 2$ and $n = 5$).

From the independence analysis results, that the sequent chord lengths in the data-sets L1,...,L18 may be interpreted as approximately independent values (realizations) of the corresponding logarithmic-normal distributed random variable X. This result make possible the statistical grain size interval estimation by the Eq. (6).

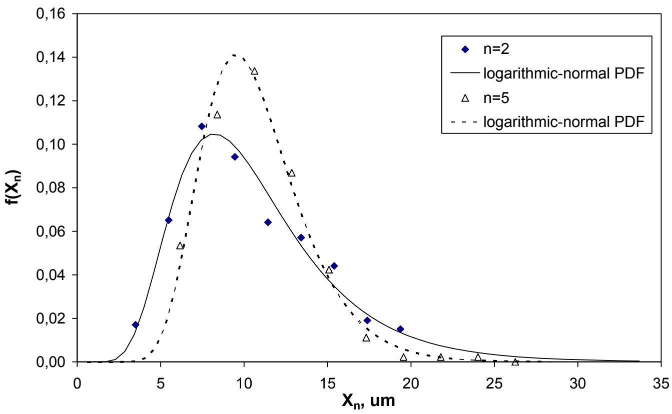

Fig. 5. Comparison of the austenite chord length PDFs $f(\overline{x}_n)$ with the logarithmic-normal PDFs for n = 2 and n = 5 (data set L6: $\overline{x}_n$ =10,75 mm, N = 1007)

3.2. Estimation

The length of sequent austenite chords along a test line are statistically independent, so the arithmetic mean $\overline{x}_n$, in Eq. (4), may be estimated by simple chord counting measurements made on linear sections [8,9,17]. In the case when a test line of length L includes n chords then

$$\overline{x}_n = \frac{L}{n} \tag{7}$$

The arithmetic mean $\overline{x}_n$ in Eq. (7) is a value (realization) of the logarithmic-normally random mean $\overline{X}_n$. The $\overline{X}_n$ is an estimator for m_X, so for given $\overline{X}_n$ the point estimation of m_x is presented by Eq. (5). In particular, for the data sets L1,..., L18 in Table 1 it is $n = N$ and $m_X \approx \overline{x}_N$

Then, the interval estimation will be given for two cases, the first one (i) when the $\sigma_{\ln X}$ – parameter is known, and the second one (ii) when the $\sigma_{\ln X}$ parameter is unknown.

3.2.1. Confidence interval for m_X when $\sigma_{\ln X}$ is known

Because of large N in the data-sets L1,...,L18 (Table 1):

$$\sigma_{\ln X} \approx s_{\ln x} \tag{8}$$

So, it can be assumed that for the analysed data-sets the parameter $\sigma_{\ln X}$ is known and for given $n < N$, Eq. (6) gives the confidence interval for m_X. Fig. 6 presents the end point coordinates of the confidence interval values (here denoted as w_1 and w_2) and the approximate expected values a_1 and a_2 as a function of n for the data-set L18. From fig. 6 it results, that up to ca. $n = 250$ the length $w_2 - w_1$ of the interval $[w_1, w_2]$ decreases; then (here up to $n = 1000$) it is approximately constant, the half length of the relative confidence interval length (a possible measure of the estimation precision) is less than 10% – it may be of practical significance. The scatter of the confidence interval values is large (depending mainly of the variance of the random mean $\overline{X}_n$).

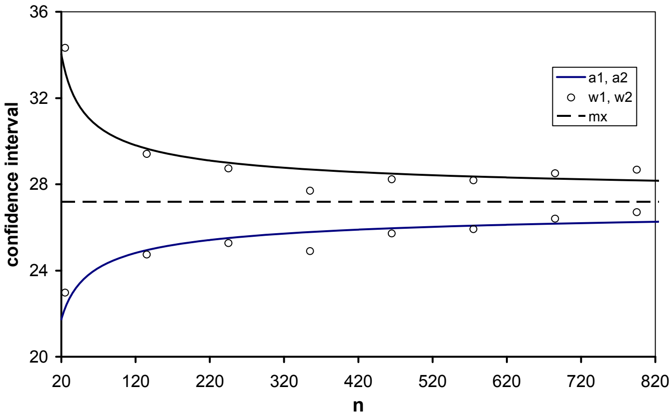

Fig. 6. Austenite grain size confidence interval values (w_1, w_2) and the expected values (a_1, a_2) as function of chord number n (L18, m_X. $\bar{x}_n = 27{,}2$ mm)

3.2.2. Confidence interval for m_X when $\sigma_{\ln X}$ are unknown

From Fig. 4 it results, that the parameter $s_{\ln x}$ for the data-sets L1,...,L18 is approximately constant (the average value is $<s_{\ln x}> = 0{,}50$). With $\sigma_{\ln X} = <s_{\ln x}> = 0{,}50$, the confidence interval in Eq.(6) may be written in the form

$$\left(\overline{X}_n \exp\left(-\frac{u_\alpha}{2\sqrt{n}}\right),\ \overline{X}_n \exp\left(\frac{u_\alpha}{2\sqrt{n}}\right)\right) \tag{9}$$

It is the approximate confidence interval for the analysed medium austenite grain size data sets L1,..,L18. When for a given austenite grain size the parameter $\sigma_{\ln X}$ is unknown and the measured grain size $\bar{x}_n$ belongs to the interval [7; 27 µm], the Eq. (9) may be used for the approximate m_X interval estimation. In order to gain an impression for the approximation using Eq. (9), the interval estimation is made for the data sets with extreme $\sigma_{\ln X}$ values (i.e., the data set L15 with $\sigma_{\ln X} = 0{,}44$ and the data set L11 with $\sigma_{\ln X} = 0{,}56$, Table 1). Fig. 7 shows the end points w_1 and w_2 of the confidence interval values (and the appropriate expected values a_1 and a_2) as a function of n for the data sets L15 and L11 of the extreme $\sigma_{\ln X}$ parameter values, i.e., 0,44 and 0.56 respectively. One can see that the applied approximation is quite satisfactorily.

Finally, it is important to notice, that for the austenite of the analysed structural steels the m_X – estimation (the point one by Eq. (5) and the interval one by Eq. (6)) may be reduced to the simple chord counting measurements made on linear sections.

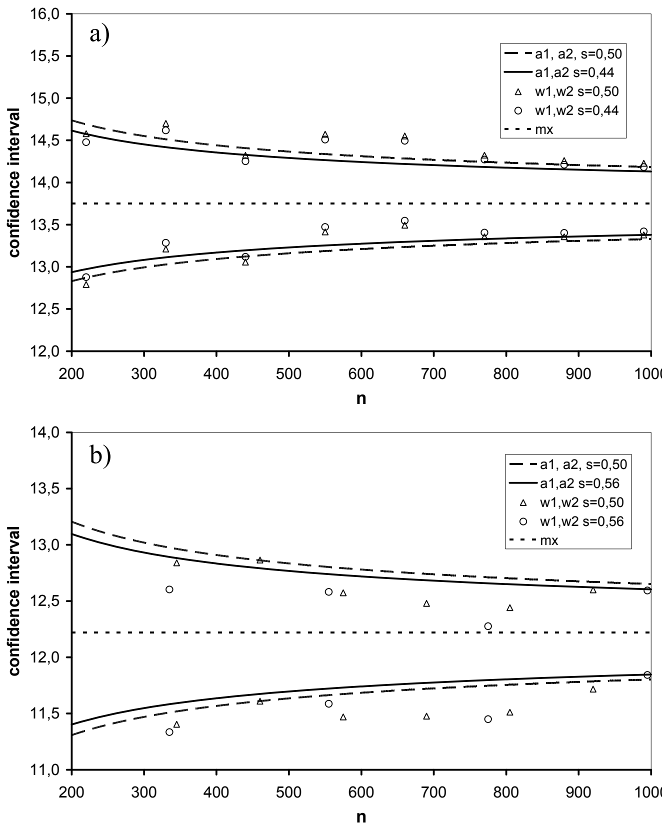

Fig. 7. Austenite grain size confidence interval values (w_1, w_2) and the expected values (a_1, a_2) as function of chord number n for the data-sets: a) L15 ($s_{\ln x} = 0{,}44$ and $s_{\ln x} = 0{,}50$) and b) L11: $s_{\ln x} = 0{,}56$ and $s_{\ln x} = 0{,}50$); $s = s_{\ln x}$

4. Discussion

For the analysed structural steels, the austenite grain chord lengths along a test line are values of a random variable X of logarithmic-normal distribution. The statistical m_X – parameter (the expected value) is the linear austenite grain size. In the first approximation the algebraic $\sigma_{\ln X}$ – parameter is independent of the particular austenite microstructure; from a practical point of view it may be assumed to be constant, i.e., $\sigma_{\ln X}.<s_{\ln x}> = 0{,}5$. (It results from approximately constant variation coefficient v_x of the austenite grain length of chords in the structural steels, also stated in [10]). Next, the sequent chord lengths along a test line are approximately independent values (realizations) of the X. In this case, for a given number n, the arithmetic mean $\bar{x}_n$, given by Eq. (4) may be expressed by Eq. (7) (simple chord counting method). Consequently, the logarithmic-normally distributed random mean $\bar{X}_n$ is an estimator for the grain size m_X. The point estimation is given by Eq. (5). The interval estimation takes into account the $\sigma_{\ln X}$ – parameter of the logarithmic-normal distribution. If the $\sigma_{\ln X}$ – parameter is known, the grain size confidence interval is given by Eq.(6). If the $\sigma_{\ln X}$ – parameter is unknown the approximate confidence interval given by Eq. (9) may be used.

Finally, it is important to notice, that the found properties of the prior austenite grain chords are adequate to particular metallurgical conditions (chemical composition, heat treatment, etc. [1]) only. For other metallurgical conditions the austenite chord length distribution follow the gamma distribution [10]. It seems, the decisive structural process which determines the austenite grain structure is a special interaction between the disperse phase (carbide, nitride, carbonitride) and the austenite grain boundaries at elevated temperature (during the austenitizing heat treatment) expressed by the Smith-Zener Eq. (1), [1, 11]. An interesting feature of the structural steels austenite chord lengths distribution (independent from the approximation by gamma or logarithmic-normal distribution) is the approximately constant value of the variation coefficient, v_x (in the range of 0,5-0,6).

5. Concluding remarks and conclusions

1. For a random variable X of logarithmic-normal distribution an approximate confidence interval for m_X is given by Eq. (6).
2. In the analysed low alloy structural steels, the sequent austenite grain chord lengths $x_1,x_2,...,x_N$ along a test line are independent values of random variable X of logarithmic-normal distribution; so, when the parameter $\sigma_{\ln x}$ is known, Eq. (6) may be used for the m_X interval estimation.
3. For the data-sets L1,...,L18 the parameter v_x and consequently the parameter $s_{\ln x}$ are approximately constant, so $\sigma_{\ln X}.<s_{\ln x}> = 0{,}50$.
4. If the parameter $\sigma_{\ln X}$ is unknown, in a first approximation the grain size m_X interval estimation may be performed using Eq. (9).
5. For the structural steels, the austenite grain size m_X estimation may be reduced to the simple chord counting measurements made on linear sections.

Acknowledgements

The main parts of this paper were presented at X Stermat Conference,1-4 June 2016 in Białka Tatrzańska (Poland). The financial support work within the AGH project 11.11.110.299 is gratefully acknowledged.

REFERENCES

[1] T. Gladman, The Physical Metallurgy of Microalloyed Steels, The Institute of Materials, 1998.
[2] C.S. Smith, Grains, Phases and Interfaces: An Interpretation of Microstructure, Trans. AIME **175**, 15-31 (1948).
[3] J.G Speer, J.R Michael, S.S. Hansen, Carbonitride Precipitation in Niobium/Vanadium Microalloyed Steels, Met. Trans. A **18A**, 211-222 (1987).
[4] H. Adrian, Thermodynamic model for precipitation of carbonitrides in high strength low alloy steels containing up to three microalloying elements with or without additions of aluminium, Materials Science and Technology **8**, 406-420 (1992).
[5] M. Perez, M. Dumont, D. Acevedo-Reyes, Implementation of classical nucleation and growth theories for precipitation; Acta Materialia **56**, 2119-2132 (2008).
[6] P. Maugis, M. Gouné, Kinetics of vanadium carbonitride precipitation in steel: A computer model, Acta Materialia **53**, 3359-3367 (2005).
[7] H. Adrian, J. Augustyn-Pieniążek, P. Marynowski, P. Matusiewicz, Model of carbonitride precipitation kinetics in HSLA steels, Metallurgist, **81/4**, 208-214, 2014. (in Polish)
[8] J. Ryś, Stereology of materials. Fotobit, Kraków 1995 (in Polish).
[9] H. Schumann, H. Oettel, Metallographie, 14-th ED. Willey – VCH, Weinheim, 2005.
[10] H. Adrian, K. Wiencek, Austenite grain size estimation in structural steels by linear section method. Archives of Metallurgy and Materials **60**, 2463-2469 (2015).
[11] R. Staśko, PhD Thesis, AGH, Kraków 2007.
[12] R. Staśko, H. Adrian, A. Adrian, Effect of nitrogen and vanadium on austenite grain growth kinetics of a low alloy steel. Mater. Char. **56**, 340-347 (2006).
[13] H. Adrian, E. Głowacz, The effect of nitrogen and microalloying elements (V and V+Al) on austenite grain growth of 40Cr8 steel, Archives of Metallurgy and Materials **55**, 107-116 (2010).
[14] J. Krawczyk, H. Adrian, The kinetics of austenite grain growth in steel for wind power plant shafts, Archives of Metallurgy and Materials **55**, 91-100 (2010).
[15] H. Adrian, M. Pelczar, A. Adrian, J. Augustyn-Pieniążek, The effect of B and microalloying elements (V, Ti, Nb) additions on the austenite grain growth of low alloy steel, Solid State Phenomena **197**, 25-32 (2013).
[16] E. Głowacz, H. Adrian, W. Osuch, Nitrogen content effect on carbonitride coagulation in 40Cr8 steel with micro-additions V and V+Al, Archives of Metallurgy and Materials, **59**, 609-613 (2013).
[17] PN-84/H-04507: Standards for determination of the grain size.