

## ESTIMATION OF TRUE INTERLAMELLAR SPACING IN PEARLITE

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### ABSTRACT

A new method of analysis, applicable for lamellar microstructures, is presented. The method discussed enables to evaluate the true interlamellar spacing distribution on the basis the known random spacing distribution. The method is applied for the coarse pearlitic microstructure.

*Keywords: distribution function, interlamellar spacing, lamellar microstructure, pearlite.*

### INTRODUCTION

Quantitative parameters describing the lamellar structure, i.e. true ( $l_t$ ), apparent ( $l_a$ ) and random ( $l_r$ ) interlamellar spacings are defined (deHoff and Rhines 1968, Underwood 1970) and basic stereological relationships for structures of this type are elaborated (Czarski and Rys 1987). The aim of the present contribution is to develop a method which enables to evaluate interlamellar spacing distribution in the coarse pearlite.

### STEREOLOGICAL BACKGROUND

Conditional density functions for the random interlamellar spacing  $f(l_r | l_t)$  and for the reciprocal of this spacing  $f(l_r^{-1} | l_t)$  are given by relations (1) and (2) respectively (Czarski and Rys 1987):

$$f(l_r | l_t) = l_t / l_r^2 \quad ; \quad l_t \leq l_r < \infty \quad (1)$$

$$f(l_r^{-1} | l_t) = l_t \quad ; \quad 0 < l_r^{-1} \leq l_t^{-1} \quad (2)$$

Taking into account the function (1) and solving the integral equation (3)

$$f(l_r) = \int_0^{l_r} f(l_r | l_t) f(l_t) dl_t \quad (3)$$

leads to:

$$f(l_t)_{l_t=l_r} = 2 f(l_r) + l_r \frac{df(l_r)}{dl_r} \quad (4)$$

where:  $f(l_r)$ ,  $f(l_t)$  - density functions for random and true interlamellar spacings, respectively.

After simple transformations equation (4) can be presented as (Czarski and Rys 1987):

$$V_v(l_t)_{l_t=l_r} = 4 l_r N_L(l_r) + 2 l_r \frac{dN_L(l_r)}{dl_r} \quad (5)$$

where:  $V_v(l_t)dl_t$  - elementary volume fraction of the lamellar structure with true spacing in the range from  $l_t$  to  $l_t + dl_t$ ,

$N_L(l_r)dl_r$  - relative number of interlamellar spacings in the range from  $l_r$  to  $l_r + dl_r$   $\left[ N_L = \int_0^{\infty} N_L(l_r)dl_r \right]$

There exist the following relation between  $f(l_t)$  and  $V_v(l_t)$  (Czarski and Rys 1987):

$$V_v(l_t) = l_t E^{-1}(l_t) f(l_t) \quad (6)$$

where:  $E(l_t)$  - expected value.

Equation (6) is valid on condition that  $E(l_t)$  exist and  $E(l_t) > 0$ .

Mean values for the random  $\bar{l}_r$  and true  $\bar{l}_t$  interlamellar spacings are defined as (Czarski and Rys 1987):

$$\bar{l}_r = E^{-1}(l_r^{-1}) \quad (7)$$

$$\bar{l}_t = E^{-1}(l_t^{-1}) \quad (8)$$

and it can be shown (DeHoff and Rhines 1968, Underwood 1970, Czarski and Rys) that:

$$\bar{l}_r = 2 \bar{l}_t \quad (9)$$

An equation similar to equation (3) bounds the distribution

functions  $f(l_r^{-1})$  and  $f(l_t)$ . Taking into consideration function (2) it can be shown for any density function  $f(l_r)$ , positively definite in the finite interval  $(l_{tmin}, l_{tmax})$ , that the density function  $f(l_r^{-1})$  in the interval  $(0, l_r^{-1} = l_{tmax}^{-1})$  will take a constant value. In other words, a plateau is observed when this function is plotted and its size can be used to estimate the  $l_{tmax}$  value. Without any considerable error it can be assumed that the minimum true spacing  $l_{tmin}$  is equal to the minimum random interlamellar spacing  $l_{rmin}$ .

### EXPERIMENTAL PROCEDURE

A model alloy Fe-Fe<sub>3</sub>C (see Table 1) was used for experiments. In order to obtain a coarse pearlite the material was austenized at the temperature 1173 K for 0.5 h and isothermally annealed in lead bath at the temperature 973±2 K for 3.5 h. The annealing time necessary for pearlitic transformation was established experimentally. The microstructure was investigated by means of the optical as well as scanning electron microscopes (see Figs 1 and 2).

Table 1. Chemical composition of Fe-Fe<sub>3</sub>C alloy; (wt-%)

C	Mn	P	S	Cr	Ni	Al	N	Fe
0.77	0.06	0.003	0.01	0.04	0.03	0.01	0.006	rest

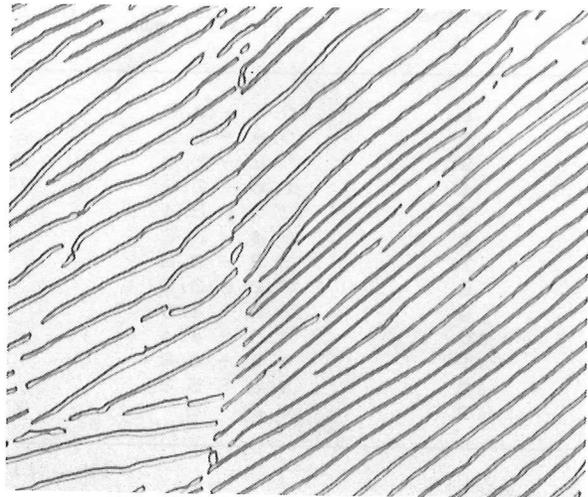
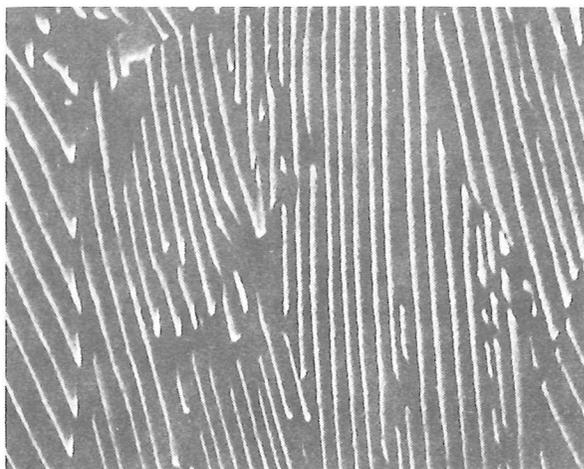


Fig.1. Coarse pearlite.  
Optical microscope,  
x2500,  
etched by 2% picral.

Fig. 2. Coarse pearlite.  
SEM, x2500,  
etched by picral.



## RESULTS AND DISCUSSION

### Evaluation of the distribution functions for random spacing and its reciprocal

The measurements were performed on the optical microphotographs. A single measurement was shown schematically in Fig. 3. A total number of  $N=11548$   $l_r$  measurements was taken. The accuracy of measurements was  $0.5 \mu\text{m}$  which means that  $l_r$  was evaluated with accuracy  $0.2 \cdot 10^{-3} \text{ mm}$  (linear magnification of microphotographs was x2500). The results were put in Table 2 and shown in Fig. 4.

Table 2. Empirical distribution of random interlamellar spacing in pearlite

Class Interval Number $i$	Class Intervals $l_r \cdot 10^3, \text{ mm}$	Number of spacings $n_i$	Frequency $n_i/N$
1	0.4 - 0.8	935	0.0810
2	0.8 - 1.2	2415	0.2091
3	1.2 - 1.6	2031	0.1759
4	1.6 - 2.0	1267	0.1097
5	2.0 - 2.4	875	0.0758
6	2.4 - 2.8	596	0.0516
7	2.8 - 3.2	441	0.0382
8	3.2 - 3.6	353	0.0306
9	3.6 - 4.0	282	0.0244
10	4.0 - 4.4	231	0.0200
11	4.4 - 4.8	202	0.0175
12	4.8 - 5.2	172	0.0149
13	5.2 - $\infty$	1748	0.1513

$N = 11548$

Fig.3. Evaluation of random interlamellar spacing, schematically.

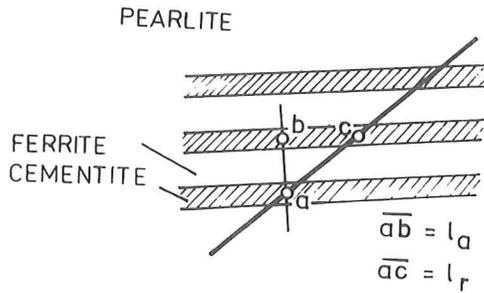
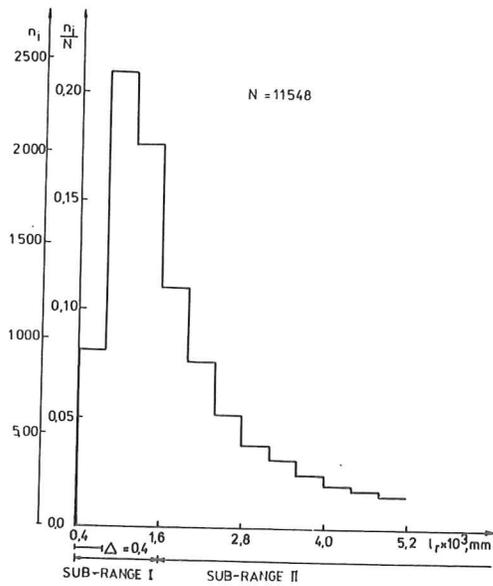


Fig.4. Empirical distribution of random interlamellar spacing (  $\Delta$  - class interval width ).



The empirical distribution of the random distribution reciprocal was presented in Table 3 and Fig.5. In the range from  $1.25 \cdot 10^3$  to  $2.5 \cdot 10^3 \text{ mm}^{-1}$  the distribution function in Fig.5 is drawn with the broken line - accuracy of single  $l_r$  measurements disabled of more precise estimation of the distribution in this range.

The mean  $\overline{l_r^{-1}}$  value was equal to  $0.674 \cdot 10^3 \text{ mm}^{-1}$ . Thus,

according to (7)  $\bar{l}_r$  is equal to  $1.545 \cdot 10^{-3} \text{ mm}^{-1}$  and (see eqn. (9))  $\bar{l}_t$  is equal to  $0.773 \cdot 10^{-3} \text{ mm}$ .

Table 3. Empirical distribution of random spacing reciprocal

Class Interval Number $i$	Class Intervals $l_r^{-1} \cdot 10^{-3}, \text{ mm}$	Number of spacings $n_i$	Frequency $n_i/N$
1	0 - 0.2083	1920	0.1662
2	0.2083 - 0.4166	2105	0.1823
3	0.4166 - 0.6250	2142	0.1855
4	0.6250 - 0.8333	2031	0.1759
5	0.8333 - 1.0417	1303	0.1128
6	1.0417 - 1.2500	1112	0.0963
7	1.2500 - 1.4583	679	0.0588
8	1.4583 - 1.6665		
9	1.6665 - 1.8750		
10	1.8750 - 2.0833	256	0.0222
11	2.0833 - 2.2917		
12	2.2917 - 2.5000		

$N = 11548$

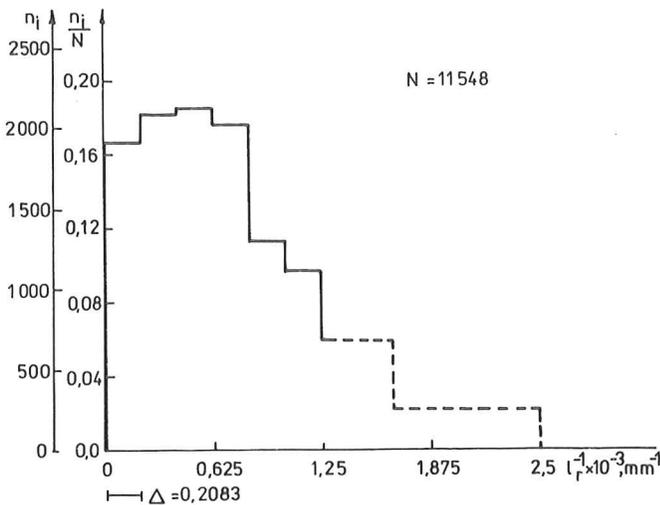


Fig. 5. Empirical distribution of random distance reciprocal.

In the empirical  $l_r^{-1}$  distribution (Fig. 5 and Table 3) the relative numbers  $n_i/N$  in the first four class intervals varied

within the range of 0.01932. So, it implies that the plateau exists in the range  $l_r^{-1} \in (0, 0.833 \cdot 10^3 \text{ mm}^{-1})$ . From the previous analysis (Stereological Background)  $l_{t\max}$  can be estimated as  $1.2 \cdot 10^{-3}$  mm and the  $l_{t\min}$  value can be evaluated as  $0.4 \cdot 10^{-3}$  mm (see Fig. 4 and Table 2).

For practical use of equation (4) the empirical random interlamellar spacing distribution should be interpolated or approximated by a continuous function or by family of functions. It enables to evaluate a function  $f(l_r)$  or at least its value and derivative at given points.

Let us divide the range of  $l_r$  values into two sub-ranges: the first (sub-range I) will cover  $l_r$  from  $l_{r\min}$  to  $l_r = l_{t\max}$  while the second one (sub-range II) will cover  $l_r$  from  $l_r = l_{t\max}$  up to infinity. We are interested in approximation in the first sub-range as only in this sub-range the distribution function  $f(l_r)_{l_r=l_r} > 0$ .

It was stated earlier that the  $l_{t\max} = 1.2 \cdot 10^{-3}$  mm. In order to avoid an error to choose underestimated value as an upper limit for sub-range I (i.e. value less than real  $l_{t\max}$  in the microstructure analysed) the following values were chosen:

- sub-range I from  $0.4 \cdot 10^{-3}$  to  $1.6 \cdot 10^{-3}$  mm,
- sub-range II from  $1.6 \cdot 10^{-3}$  mm to infinity

In the sub-range considered (sub-range I) there existed three class intervals (see Fig. 4), so every approximation would be inaccurate. Thus, sub-range I should be divided into a larger number of class intervals which required new, additional and more accurate  $l_r$  measurements. To improve the resolving power and accuracy of measurements a scanning electron microscope was applied. The specimen surface was perpendicular to the incident beam direction to avoid any distortion effects. Preparation technique (polishing and etching) was the same as for the optical micrographs. However, the same accuracy of measurements (0.5 mm) gave, thanks to the magnification  $\times 12500$ , a real accuracy of  $4 \cdot 10^{-5}$  mm.

In sub-range I  $N_I = 1575$  of individual  $l_r$  values, grouped into 15 class intervals were analysed (see Table 4). Relative numbers  $n_{Ii}/N_I$  given in Table 4 could not be considered as empirical values for the  $l_r$  distribution as only measurements from one of the two sub-ranges were taken into analysis. These values referred only to the sub-range I and should be multiplied by a factor of 0.466 (a sum of relative numbers in sub-range I from the first series of measurements) in order to achieve correct values. A corrected  $l_r$  empirical distribution was shown in Fig. 6. Note that the class intervals in sub-range II were the same in distribution presented in Fig. 4.

Table 4. Random interlamellar spacing measured by using SEM (sub-range I)

Class Interval Number $i$	Class Intervals $l_r \cdot 10^{-3}, \text{ mm}$	Number of spacings $n_{Ii}$	Frequency $n_{Ii}/N_I$
1	0.40 - 0.48	16	0.0102
2	0.48 - 0.56	28	0.0178
3	0.56 - 0.64	52	0.0330
4	0.64 - 0.72	79	0.0502
5	0.72 - 0.80	98	0.0622
6	0.80 - 0.88	122	0.0775
7	0.88 - 0.96	129	0.0819
8	0.96 - 1.04	152	0.0965
9	1.04 - 1.12	135	0.0857
10	1.12 - 1.20	139	0.0883
11	1.20 - 1.28	138	0.0876
12	1.28 - 1.36	133	0.0844
13	1.36 - 1.44	127	0.0806
14	1.44 - 1.52	121	0.0768
15	1.52 - 1.60	106	0.0673

$N_I = 1575$

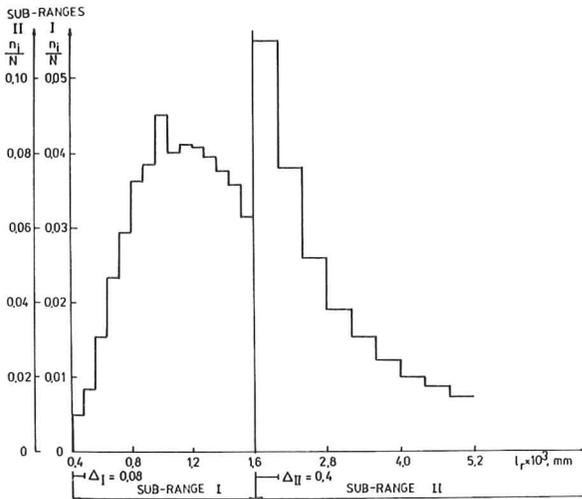


Fig. 6. Empirical distribution of random spacing. Sub-range I - SEM, sub-range II - optical microscope.

$\Delta_I$ ,  $\Delta_{II}$  - class interval width in sub-range I and II, respectively.

Evaluation of the distribution function for true interlamellar spacing

To simplify notation the parameter  $x = l_r \cdot 10^3$  was used in this paragraph instead of the  $l_r$  variable. Empirical distribution of the variable  $x$  in the sub-range I was described by the following function (Gersternkorn et al. 1979):

$$f(x) = \frac{|\beta| \lambda^{\alpha/\beta}}{\Gamma(\alpha/\beta)} (x-x_0)^{\alpha-1} e^{-\lambda(x-x_0)^{\beta}} \quad ; \quad x \geq x_0 \quad (10)$$

where:  $\alpha, \beta, \lambda$  - constant values ( $\alpha \beta > 0, \lambda > 0$ )  
 $x_0$  - translation parameter ( $x_0 > 0$ )

$\Gamma$  - Euler gamma function

Parameters of the approximation function (10) were established numerically using the last square method as:

$\alpha = 3.85, \beta = 0.5, \lambda = 6.45, x_0 = 0.36$ .

Function (10), with respect to the parameters evaluated, was shown in Fig. 7.

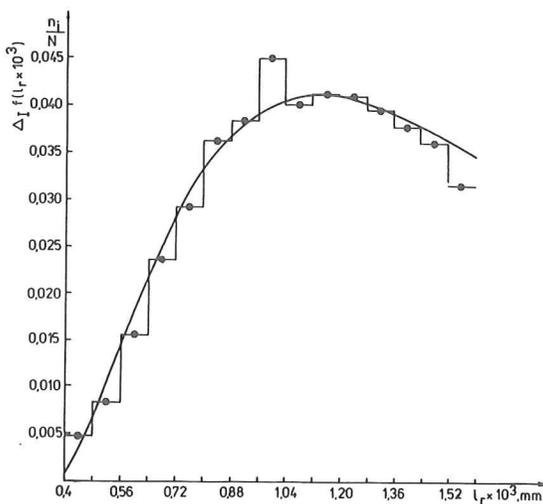


Fig.7. Empirical distribution of random spacing in the sub-range I.

Normalization of function (10) with stable parameters in the sub-range I leads to:

$$f_1(x) = \left[ \int_{x_{min}}^{x_{max}} f(x) dx \right]^{-1} f(x) \quad ; \quad x_{min} < x < x_{max} \quad (11)$$

where:  $x_{\min}$ ,  $x_{\max}$  - lower and upper limits of the sub-range I, respectively (  $x_{\min} = 0.4$ ,  $x_{\max} = 1.6$  )

The definite integral in equation (11) was evaluated numerically by using the double-point Gaussian quadrature (Fortuna et al. 1982) giving the value of 0.4721.

Function  $f_1(x)$ , described by equation (11), could be interpreted as probability density function in the sub-range I. Application of the chi-square test (Fisz 1958) enabled to verify the hypothesis that the distribution function (11) described the empirical distribution of  $x$ . Using equation (4) and function (10) a new function  $f(z)$  was evaluated, where  $z = l_t \cdot 10^3$ . Minimum values of  $z$  and  $x$  were identical. The maximum  $z$  value was calculated from the condition:

$$\int_{z_{\min}}^{z_{\max}} f(z) dz = 1$$

Finally, the true interlamellar spacing distribution function took the following form:

$$z = l_t \cdot 10^3, \text{ mm}$$

$$f(z) = 309.12 e^{-6.45(z-0.36)^{0.5}} (z-0.36)^{2.85} \cdot \left[ 2 + z(z-0.36)^{-1} [ 2.85 - 3.225(z-0.36)^{0.5} ] \right]$$

$$0.4 < z < 1.328 \quad (12)$$

Taking the expected value  $E(z) = 0.877$  from function (12) and using (6) the function  $V_V(z)$  was calculated as:

$$z = l_t \cdot 10^3, \text{ mm}$$

$$V_V(z) = 352.47 e^{-6.45(z-0.36)^{0.5}} z(z-0.36)^{2.85} \cdot \left[ 2 + z(z-0.36)^{-1} [ 2.85 - 3.225(z-0.36)^{0.5} ] \right]$$

$$0.4 < z < 1.328 \quad (13)$$

Function (12) and (13) were shown in Fig.8

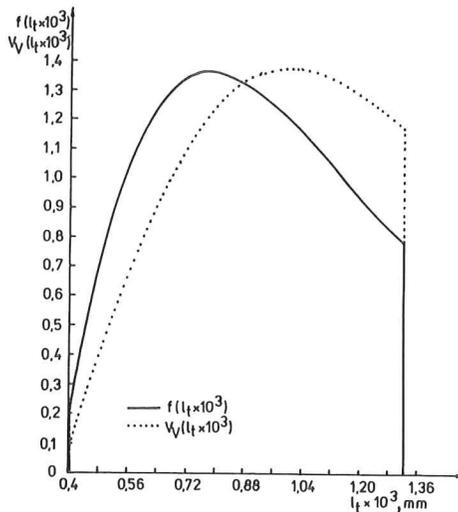


Fig.8. Distribution  $f(l_t \cdot 10^3)$  (12) and  $V_V(l_t \cdot 10^3)$  (13) for analysed coarse pearlite.

## CONCLUSIONS

The method presented is very time- and workconsuming. Therefore it is not interested in metallographical practise. From this point of view approximate solution for equation (4) should be elaborated.

Special consideration should be paid to the distribution of random spacing reciprocal. It seems that thanks to existing definitions of the mean values (eqs.(7) and (8)) and the observed plateau which length describes the range of true interlamellar spacing, the distribution discussed gives more interesting information concerning the lamellar microstructure than the random spacing distribution.

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