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MICROSTRUCTURAL PARAMETERS OF DISPERSION STRENGTHENED Al-Al_4C_3 MATERIAL BY IMAGE ANALYSIS*

M. BESTERCI[1**] O. VELGOSOVÁ[2] AND I. KOHÚTEK[3]

[1]Institute of Materials Research, Slovak Academy of Science, Košice 043 53, Watsonova 47, Slovakia
[2]Dept. of Non-ferrous Materials and Waste Treatment, Faculty of Metallurgy, Technical University in Košice, Letná 9/A, 042 00 Košice, Slovakia, E-mail: velgos@hfnov.hf.tuke.sk
[3]U. S. Steel Košice, Research and Development Centre, 044 54 Košice, Slovakia

Abstract – Al-Al_4C_3 dispersion strengthened material was prepared by a powder metallurgy method of reaction milling. Interparticle distances of Al_4C_3 particles in the Al-Al_4C_3 composite materials were evaluated. In this paper, nearest neighbour distribution function, polygonal methods and the quadrate counts method are used for characterization of particle arrangement.

Keywords– Dispersion strengthened system, image analysis, interparticle distance, quadrant count and polygonal method

1. INTRODUCTION

The matrix-dispersoid system can be prepared in various ways [1]. With the help of mechanical alloying it became possible to incorporate very fine fairly uniform distributed particles into a metal matrix. Mechanical alloying has been developed and used to prepare immiscible alloys and superalloys with nickel matrices, and the method has later spread to other alloys. The process starts with drying, high energy milling of the matrix powder with a dispersoid, and producing a homogeneous composite with a finely controlled microstructure. The intense milling results in a matrix with an even distribution of dispersed particles. Dispersoids can be formed in a solid state reaction by introducing materials that react with the matrix in heat treatment subsequent to mechanical alloying [2].

One kind of mechanical alloying is reaction milling, which was developed for dispersion strengthened aluminium production [3, 4]. To produce aluminium dispersoids, the aluminium powder is intensively dry milled with carbon powder. The transformed dispersed phase Al_4C_3 is then produced by a chemical reaction which begins during milling, and is completed in a subsequent heat treatment process. The resulting powder mixture is then pressed, compacted and isostatic pressing and hot extrusion prepare the final compact.

The mechanism of dispersion strengthening is based on the presence of a low volume fraction of fine secondary particles in the metal matrix. If the particle sizes are negligible, with respect to their mutual distances, they can be considered as *points*. Particles in real material can be visualized in the cross section of the microstructure (by optical microscopy and scanning electron microscopy SEM) or in a projection (by transmission electron microscopy TEM). Quantitative characterization of the particle space distribution is a significant problem. Classification of Al_4C_3 particle arrangement in Al-Al_4C_3 materials with 12% vol. of Al_4C_3 using the stochastic geometry method is the aim of the paper.

2. EXPERIMENTAL MATERIAL AND METHODS

Al-Al_4C_3 experimental material with a nominal volume fraction 12% vol. of Al_4C_3 particles was prepared by reaction milling in the following way: homogenisation milling of Al and C powders in attritor for

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**Corresponding author

90 minutes, compaction, cold pressing, heat treatment and extrusion at 600°C, Fig.1. Randomly selected images of microstructure cross-sections obtained from SEM were digitised by the system for optimisations and digitizations of analog signal TESCAN. Digital images were processed by an image analysis system, DIPS 5.0. Additive noise was removed by linear filters mainly with Gauss characteristics, and analysed objects (particles of Al_4C_3) were defined by so-called "pseudocolors" (all particles were filled by one colour instead of their natural color). Five images with total particle numbers 27088 were prepared from Al-Al_4C_3 experimental material. Centroids of objects were used as point sets, which were analysed below. Voronoi tessellation generated by corresponding point sets (see [5, 6]) were also used for analysis. A series of steps from the original image through defined objects to Voronoi tessellation generated by centroids of particles is shown on Fig.2.

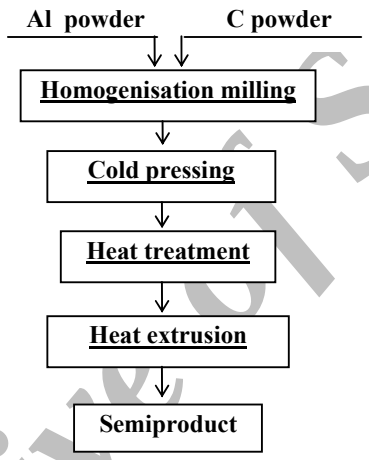
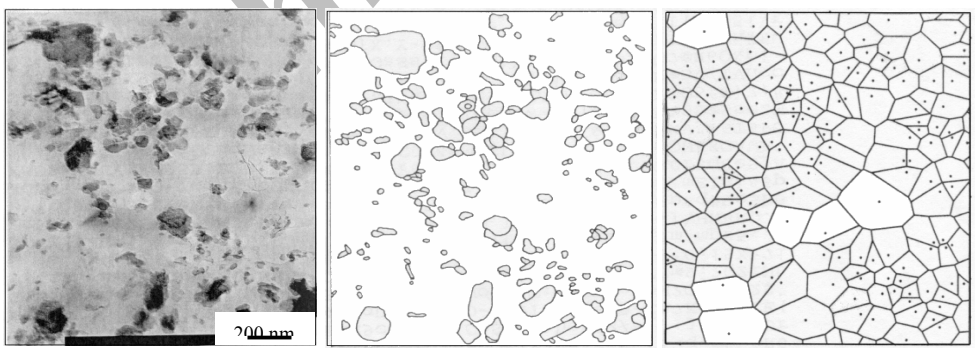


Fig.1. Way of Al-Al_4C_3 material preparing



(a) (b) (c)

Fig. 2. The example of Voronoi tessellation: a) cross section observed by SEM, b) outlines of Al_4C_3 particles, c) Voronoi tessellation with reference point

Point arrangement analysis methods are based on a comparison of the characteristics of an analysed point set with known arrangement models (Fig.3), especially theoretical Poisson point process PPP [5, 7]. A lot of theoretical research and computer simulations were focused into discovering PPP characteristics (see [8]). The following three methods of particles characterization were used:

- nearest neighbour distribution function.
- quadrat counts method.
- polygonal methods.

a) Nearest neighbour distribution function

Distance of the nearest neighbor σ_p (see Fig.4) was chosen from various different definitions of the interparticle distance [9]. Because interparticle distance is a random variable for certain point process, it can be fully described by its probability density function. Probability density function can be estimated from the

set of original (analysed) points. The shape of this function is usually compared with the corresponding function of some known point process (for example PPP).

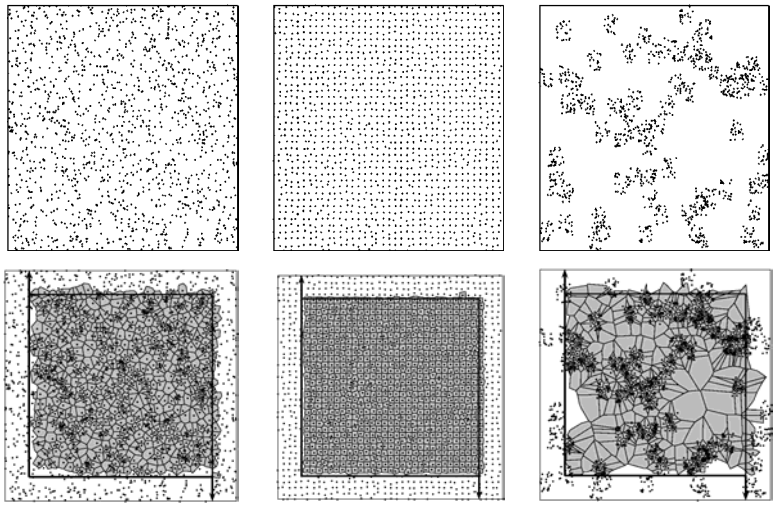


Fig.3. Poisson point process and corresponding Voronoi tessellation (a), regular point process and corresponding Voronoi tessellation (b), cluster process and corresponding Voronoi tessellation (c)

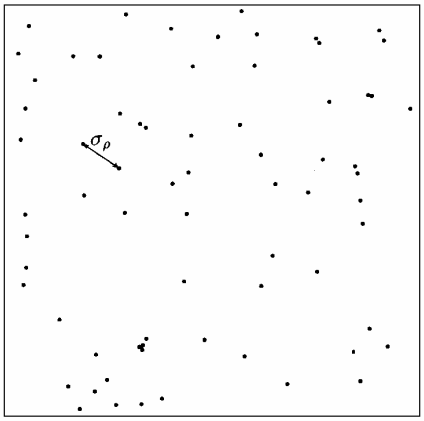


Fig. 4. Examples of nearest neighbour distance

b) Quadrat counts method

This method is based on dividing the investigated area into *m* subregions of equal size, mostly squares. The number of points in individual subregions is counted and these counts are processed by different methods, for example, index of cluster size ICS and index of dispersion ID [10]. In the case of PPP with randomly distributed particles, ICS is approximately zero. The ICS approaches positive value for cluster point processes and negative for regular point processes (lattice), Fig. 3a. The ID of PPP is lower than the ID of point process with the clusters, but higher than the ID of regular points process Fig. 3b. The shape of ICS and ID of the analysed point set is compared with ICS and ID of PPP with respect to the conclusions mentioned above.

c) Polygonal methods

Polygonal methods (also known as dual methods) are based on characteristics of Voronoi tessellation generated by evaluated point set (Fig. 4 and Fig.6). Cells of Voronoi tessellation (usually polygons) are characterised by their measures–for instance areas, perimeters, and angles between adjascent borders. The following statistical characteristics of cell areas were used in the paper: variance $var_{(p)}$, coefficient of

variance $CV_{(p)}$, skewness and kurtosis for definition see [1, 7]. Estimated values of these characteristics are compared with corresponding PPP values.

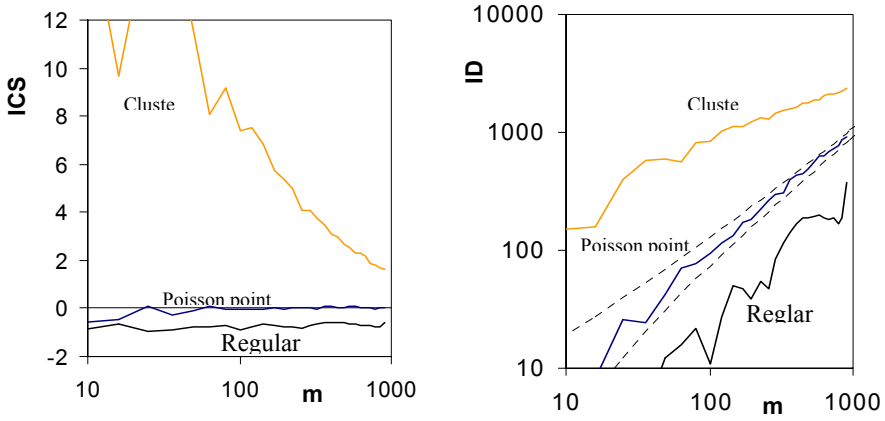


Fig. 5. The index ICS (a), the index ID (b)

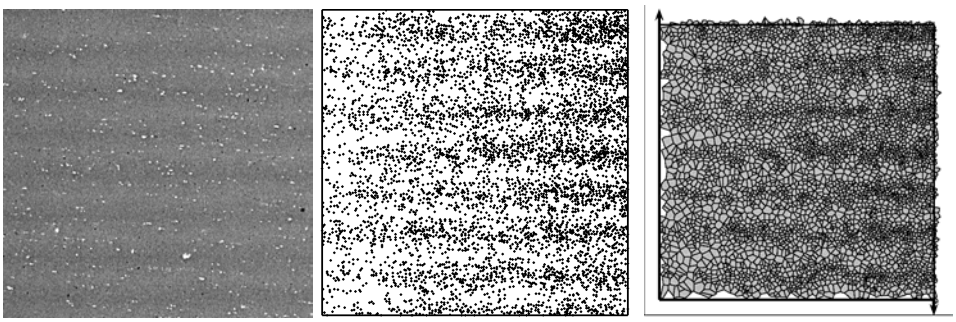


Fig. 6. The process of generation of Voronoi tessellation: (a) initial picture – metallographic specimens with Al_4C_3 particles, (b) picture processed by image analyse - centroids of particles, (c) Voronoi tessellation

3. RESULTS AND DISCUSSION

The dependence of the ID and ICS on the number of subregions can be seen in Fig. 7. Index ID of the evaluated point set lies above the PPP line, which indicates the tendency of microstructure to clustering. Index ICS of the evaluated point set is positive, which indicates the presence of particles clustering. Both ICS and ID indices indicate the tendency of microstructure particles to form clusters.

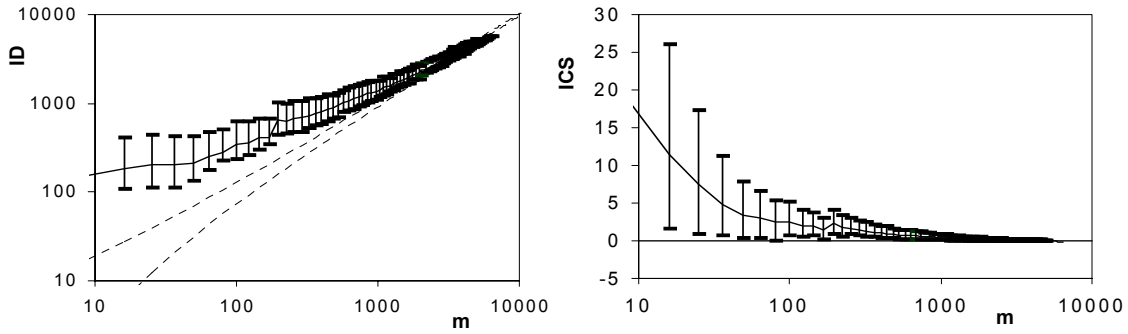


Fig. 7. The ID and ICS indices of Al-Al_4C_3 experimental material

The comparison of probability density function of nearest neighbour distance $f(\sigma_\rho)$ of experimental materials and PPP is presented in Fig. 8. From this analysis it can be stated that the minimal distances between particles in Al-Al_4C_3 material are 0.24 (in non-dimensional unit). This behaviour can by caused by

the presence of empty surrounding around every point, which can be explained by real non-zero size of all particles in samples. This idea can also be confirmed also by the shape of corresponding $f(\sigma_\rho)$ function of PPP, which is slightly shifted towards the left. Shapes of both compared functions are very close in intervals from the common local maximum (mode). From the above, it is possible to state that particles are randomly distributed in the microstructure with a very slow tendency to regular arrangement caused by their non-zero size.

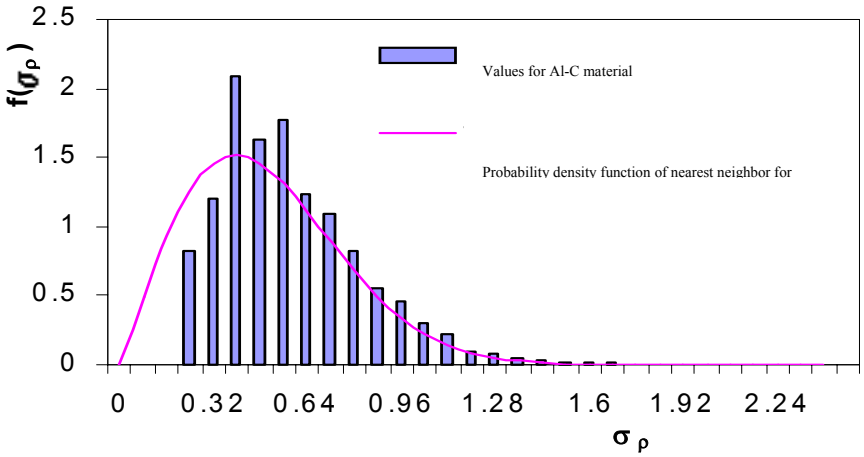


Fig. 8. Comparison of probability density functions of nearest neighbor for experimental materials and for PPP

Estimations of statistical characteristics of Voronoi tessellation generated by both analysed point sets and PPP are presented in Table1. From the computer simulations of various types of point arrangements it is known that values of presented parameters above corresponding PPP values indicate that particles are arranged in clusters. On the other hand, values below the PPP values could be produced by regular (ordered) point sets.

Table. 1. Statistical characteristics of cell area of Voronoi tessellation

Samples	Number of particles	$var_{(p)}$	$CV_{(p)}$	skewness	kurtosis
a	6360	0.299	0.539	1.324	2.622
b	5250	0.357	0.593	1.561	4.034
c	4916	0.306	0.555	1.417	3.106
d	4817	0.513	0.720	2.166	7.72
e	5745	0.405	0.634	1.74	4.682
PPP	-	0.281	0.529	1.033	4.600

As all images were randomly chosen from one sample, it is possible to state that particles of the second phase exhibit a tendency to cluster with the presence of ordering probably caused by the non-zero size of original particles.

4. CONCLUSION

Tests did not provide clear information about the type of particle distribution. Two of them indicated the tendency to cluster, and one, the tendency towards random arrangement with regular features. This visible contradiction can help us to understand that the conception of uniform classification of microstructures into one from predefined types of point arrangement is not suitable. Real microstructure is a more complicated system and it can usually be assigned only as a possible mixture of several basic arrangement types.

Regarding the real distribution of particles in microstructure, it is very important to use various tests which are based on different principles and which are sensitive to different features in point arrangement.

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REFERENCES

1. Besterci, M. (1999). *Dispersion strengthened Al prepared by mechanical alloying*. Cambridge Int. Sci. Publ., ISBN 189832655.
2. Weiβgäerber, T. & Kieback, B. (2000). Disperse strengthened materials obtained by mechanical alloying – An overview. *Materials Science Forum*, *8*, 275.
3. Korb, G., Jangg, G. & Kutner, F. (1975). Eigenschaften von dispersionsverfestigten Al-Al_4C_3 werkstoffen. *Draht*, *30*, 318.
4. Jangg, G., Kutner, F. & Korb, G. (1975). Dispersionsverfestigten werkstoffen af Al basis. *Aluminium*, *51*, p. 641.
5. Okabe, A., Boots, B. & Sagihara, K. (1992). Spatial tessellation. J. Wiley, Chichester.
6. Besterci, M., Kohútek, I., Saxl, I. & Sülleiová, K. (1999). Analysis of spatial arrangement of particles in thin foil of Al-Al_4C_3 material. *J. of Materials Science*, *34*, 1055.
7. Stoyan, D., Kendall, W. S. & Mecke, J. (1987). *Stochastic geometry and its applications*. Academy-Verlag, Berlin.
8. Hinde, A. L., Milles, R. E. (1980). Stochastic geometry. *J. Statis. Comput. Simul*., *10*, 205.
9. Besterci, M., Kohútek, I., Sülleiová, K., Saxl, I. & Zrník, J. (1996). Možnosti popis obrazov heterogénnych štruktúr využitím stereologických postupov. *Acta Metallurgica Slovaca*, *2*(4), 304.
10. Besterci, M. (1996). Disperzne spevnený Al pripravený mechanickým legovaním. *Pokroky práškove metalurgie*, *2*, 5.