Materials Science-Poland, Vol. 23, No. 1, 2005

Application of image analysis for characterization of powders

JAKUB MICHALSKI[1*], TOMASZ WEJRZANOWSKI[1], ROMAN PIELASZEK[2], KATARZYNA KONOPKA[1], WITOLD ŁOJKOWSKI[2], KRZYSZTOF JAN KURZYDŁOWSKI[1]

[1]Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, 02-507 Warsaw, Poland

[2]High Pressure Research Center of the Polish Academy of Science, Sokołowska 29, 01-142 Warsaw, Poland

A number of currently developed and produced modern multi-functional materials are to a large extent based on the use of powders. Powders with precisely characterized size, size distribution, shape and surface are used directly (e.g. catalysis) or to fabricate novel ceramics, metals and composites. Description of morphology of powders, both in micro- and nano-scale, could be obtained by the means of modern stereological methods supported by computerized image analysis. These methods can be used to describe size, shape, surface topography for both the aggregates and agglomerates and single crystallites. Stereological methods supported by computerized image analysis of high-resolution electron microscope images are becoming an important tool of modern powder-related materials science. The present work demonstrates the possibility of using such methods in determining the basic stereological parameters characterizing the morphology of the ceramic powders in micro- and nano-scale.

Key words: *nano-crystals; grain size and shape; image analysis; stereology*

1. Introduction

Progress in development of modern materials, including so-called nanomaterials, is enhanced by advances in powder technologies and improvement in powder characterization methods. Production, processing and thorough characterization of ultra fine-grained powders in connection with novel techniques of their consolidation is an important segment of nanotechnologies. Currently nanopowders are used, for example, as filling materials for different kinds of polymers and ceramics to produce compos-

*Corresponding author, e-mail: jmichalski@inmat.pw.edu.pl.

ites with better strength, hardness, corrosion and electrical resistance. They are also directly applied in medicine (drug delivery) and chemical industry (catalytic processes).

The efficiency of nanopowders is controlled by their chemistry but also by size and shape. The aim of the present paper is to demonstrate how size and shape can be characterized from images of powders particles correlated with methods of indirect size estimates.

2. Size and shape

Powders are usually mixtures of different sized grains, where the fine fraction, decisive on the properties of the particular powder, reveals desirable characteristics of the nanoscale, whilst larger grains might significantly weaken this effect. At the same time, most nanopowders have tendency to form agglomerates as a result of high surface energy related to their high specific surface. This is why nanopowders are usually composed of micrometric agglomerates of nanometrical crystallites.

Figure 1 shows the relation of intensity of a hypothetical size effect (e.g. luminescence) to the average powder grain size $< \overline{d} >$ (horizontal axis, [nm]) and relative grain size distribution (vertical axis, CV(d)/<R>) [2]. An assumption has been made that the effect is strongest for crystals of 5 nm size and weakens with the grain size as 1/R. One contour line represents a decrease in the effect intensity by about 9%. It thus clear that not only the average grain size but also size distribution both influence the effects observed in nanoscale.

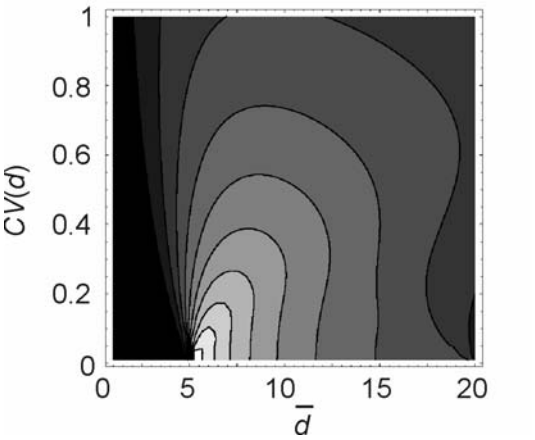

Fig. 1. Relation of intensity of the hypothetical size effect (e.g., luminescence) to the average powder grain size $< \overline{d} >$ (horizontal axis, [nm]) and relative grain size distribution (vertical axis, $CV(d)$)

Therefore, in modern applications it is not sufficient to control only average grain size which can be obtained from standards methods of the specific surface or analysis of peak widths in X-ray diffraction. Thorough characterization is needed which requires description of individual grains and the agglomerates of grain powders. Size

and shape of individual crystallites relate to the first concept, yet the most complete description of the powder as a whole is its grain size distribution.

The methods used to measure the powder grain size, enabling the analysis of the grain size distribution, could be divided into two groups: direct and indirect. The direct methods map the geometry of individual grains via electron scanning and transmission microscopy. Indirect methods are based on physical effects, such as widening of X-ray diffraction reflexes in XRD methods or laser diffraction [1, 2]. The present paper concerns direct measurements of grain/particle size.

Stereological methods could be used for precise description of powders based on their planar images. Useful for powder characterization are parameters measured for individual "objects" such as: equivalent average d_2, d_{max}, d_{min} and shapes factors $\alpha = d_{max}/d_2$, $\beta = p/\pi d_2$, $\gamma = p/p_c$. These parameters are relatively easy to determine with the planimetric methods and computer image analysis. The graphic illustration of the above-listed parameters is shown in Fig. 2.

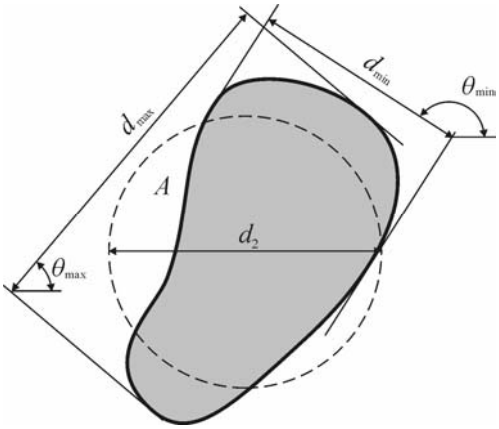

Fig. 2. Graphical interpretation of parameters describing image of the grains: area (A), calculated on planar section, conventional diameter (d_2), diameter of a circle of the same surface as the surface of the analyzed grain, maximal projection (d_{max}), circumference (p), shape coefficient ($\alpha = d_{max}/d_2$, $\beta = p/d_2$ and $\gamma = p/p_C$) [3]

The above parameters are determined for individual grains/particles. Statistical size distribution is described by the frequency function $f(x)$ and usually characterized by means of statistical moments. The basic moment is the average value of the particular parameter $E(x)$. Also, the coefficient of variation, marked as $CV(x)$ is used. It describes the relative extent of dispersion of measured values ($CV(x) = SD(x)/E(x)$, where $SD(x)$ is the standard deviation [3]. Average values: A and d_2, $E(A)$ and $E(d_2)$, allow one to determine the average grain size. However, in order to fully characterize the structure, it is also necessary to determine the size distribution of the measured structure elements. This helps to prevent mistakes in interpretation of the results obtained with the use of parameters such as $E(A)$ or $E(d_2)$ for materials with diversified elements (Fig. 3). In some cases it is possible to establish weighted particle size distribution taking particle volume, as the weight. This enables presenting the real particle volume distribution in the analyzed powder (large particles even though less numerous take up a larger volume than smaller particles which there are usually more of).

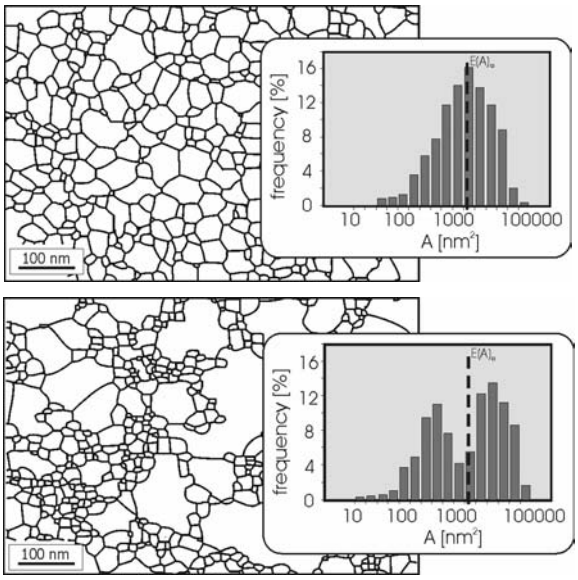

Fig. 3. Sample structures characterized by the same average grain surface value but with different grain size distribution

Average values of shape parameters $E(d_{max}/d_2)$ $E(p/d_2)$ $E(p/p_C)$ are used for quantitative description of grain elongation, curvature of grain boundary and convexity. Sample shapes of structural elements for different combinations of the shape coefficients are shown in Fig. 4.

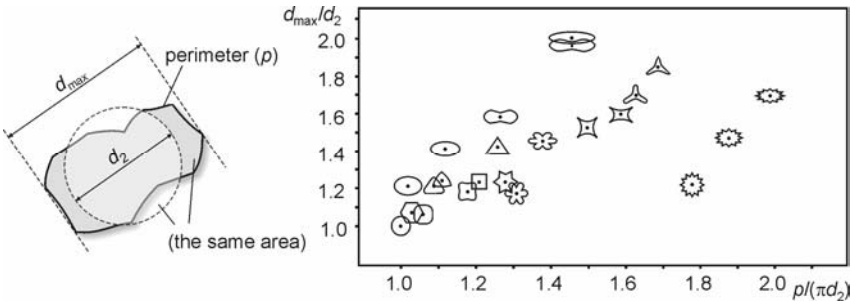

Fig. 4. Shapes of structure elements for different combinations of the shape coefficients

3. Image analysis and stereology

Stereological methods supported by computer image analysis make useful tools for quantitative powder characterization. Software MicroMeter developed at the Faculty of Materials Science and Engineering of the Warsaw University of Technology

was used in the present work [4] for the analysis of the size and shape of the nanopowders.

Procedure of qualitative powder analysis with the use of already mentioned methods could be divided into 3 stages: imaging, image analysis and determination of the stereological parameter.

The choice of imaging technique depends largely on the type of powder and especially its size. In the case of very fine-grained powders, it is necessary to use TEM, nevertheless even in such a case images under smaller magnifications (e.g. SEM) could reveal the degree of agglomeration.

For the purpose of qualitative analysis, microscopic images are transformed into binary ones which determine the powder grains. For the purpose of further analysis those grains are selected, which do not overlap with others (see Fig.5).

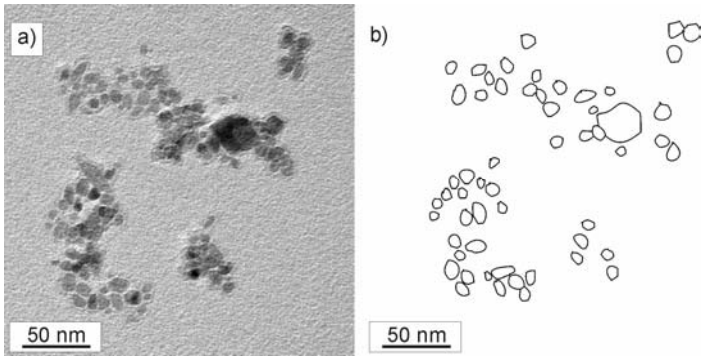

Fig. 5. TEM image of ZrO_2 powder (a) and its binary representation (b)

Parameters obtained as a result of qualitative analysis and their distributions are then transformed into true distribution (3D) through stereological modelling. Such models focus mostly on the grain shape. When powder particles are spherical, their projections on microscopic images are circles, and the diameter of each of these circles could be regarded as the spatial diameter.

4. Examples of applications

AKP-50 Sumimoto sub micron Al_2O_3 powder was subject to laser diffraction (Fig. 6), computer image analysis of SEM (Fig. 7) and TEM (Fig. 8). Quantitative analysis of the ceramic Al_2O_3 Sumimoto powder revealed that this powder is characterized by the average grain size of 120–130 nm. Also, the grain sizes are uniform (coefficient of variation – 30%) and the size distribution is a log-normal.

Powder grain shape analysis showed that the particular powder grains are spherical-like shaped (α = 1.26–1.33). The low value of the variation coefficient obtained for the shape factor α (Tables 1 and 2) proves a strong shape homogeneity of powder particles.

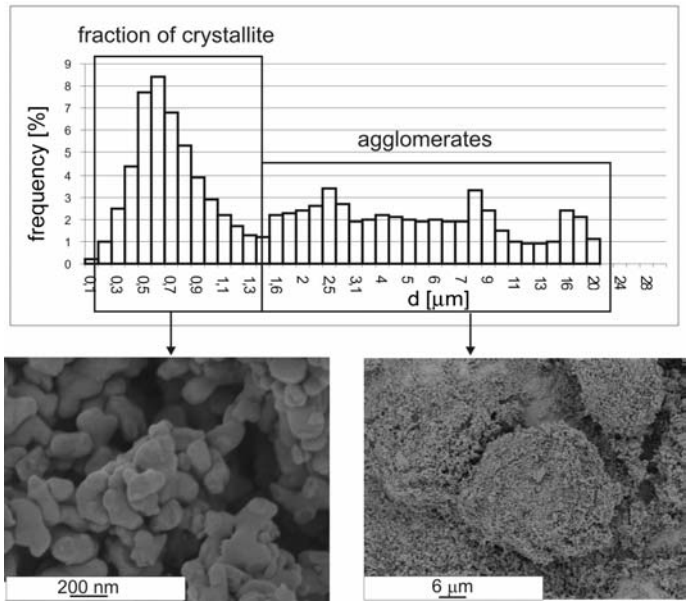

Fig. 6. Grain size distribution of sub micron powder AKP-50 measured by the laser diffraction

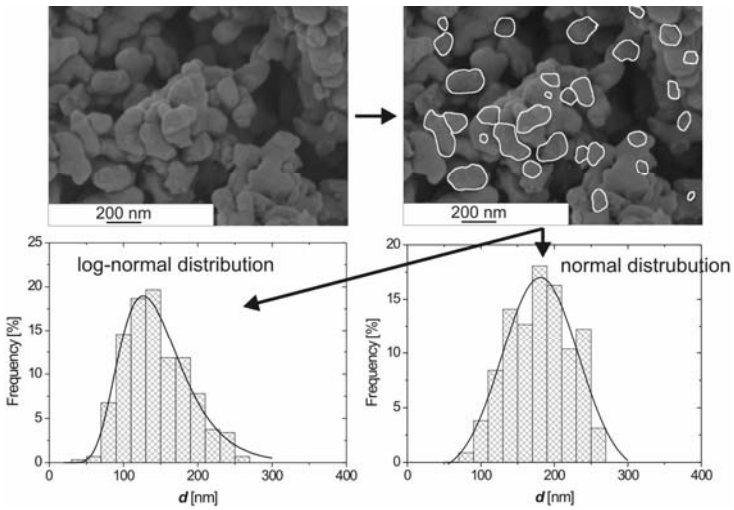

Fig. 7. Grain size distribution of the sub micron powder AKP-50 by computer analysis of SEM image, log-normal distribution (a), weight distribution (b)

When comparing the results obtained by laser diffraction with the results from quantitative analysis of images it is easy to notice the deviation caused in laser diffraction by powder agglomeration. The average grain size of 3.16 μm is a combination of sizes of individual crystallites and agglomerations, whilst the results of quantitative analysis performed on SEM and TEM images result in true values of 120 to 130 nm.

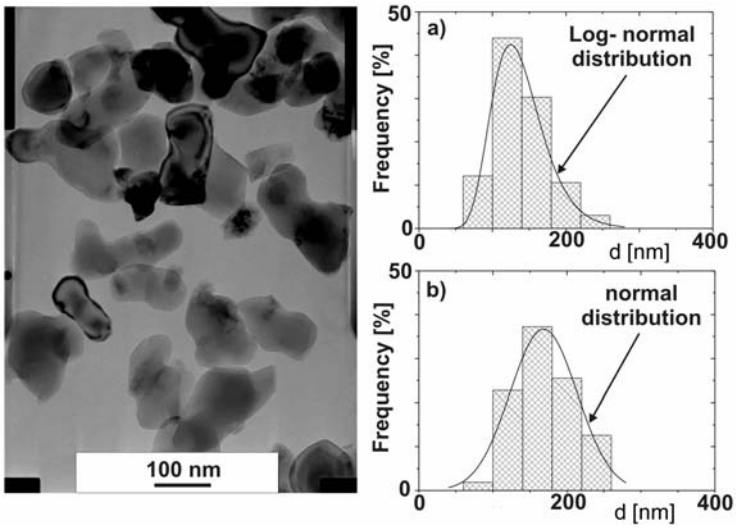

Fig. 8. The grain size distribution of sub micron powder AKP-50 by computer analysis of TEM image, log-normal distribution (a), weight distribution (b)

Table 1. The results of measurements shown in Fig. 7 for SEM

Value	*d*	Shape factor α
Average Coefficient of variation	130 nm 30%	1.26 9%

Table 2. The results of measurements shown in Fig. 8 for TEM

Value	*d*	Shape factor α
Average Coefficient of variation	120 nm 30%	1.33 9%

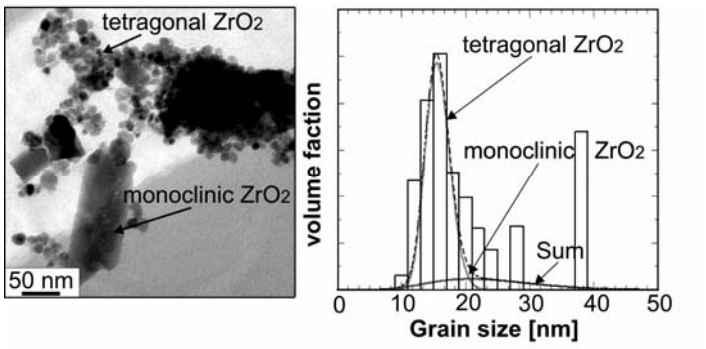

Fig. 9. The grain size distribution of ZrO_2 nano-powder obtained from TEM image analyses and from XRD method

ZrO_2 powder, obtained by the hydrothermal method, was also subject to computer analysis of TEM images and a unique method of XRD experimental data analysis (Fig. 9).

The results obtained through two different methods (Fig. 9) show high degree of conformity in the crystallite size distribution. The qualitative TEM image analysis revealed the presence of large crystallites of about 40 nm in the tested powder. The XRD analysis has shown that the tested powder contains two phases, tetragonal ZrO_2 and monoclinic ZrO_2, with larger crystallites of the latter.

4. Summary

Computer quantitative SEM and TEM image analysis could be a very useful tool providing on a fast and precise analysis of powders both in micro and nano scales. It enables not only an accurate estimation of average grain size of the analyzed powder but also a characterization of its grain size distribution. This is important in order to process technologically the samples and also to forecast the properties of powders and materials obtained from the powder.

Acknowledgement

This research has been financially supported by the State Committee for Scientific Research (KBN) under contract No. 3 T08A 029 27.

References

[1] PIELASZEK R., *Analytical expression for diffraction line profile for polydispersive powders*, Appl. Crystallography, Proceedings of the XIX Conference, Kraków, Poland, September 2003, pp. 43–50.
[2] PIELASZEK R., J. Alloys Comp. 382 (2004), 128.
[3] KURZYDŁOWSKI K.J., RALPH B., *Quantitave description of the microstructure of materials*, CRC Press, Baton Rouge, USA, 1995.
[4] WEJRZANOWSKI T., *Computer assisted analysis of gradient materials microstructure*, Master Thesis, Warsaw University of Technology, 2000.

Received 6 June 2004
Revised 11 June 2004