

CHARACTERIZATION OF POWDER PARTICLE MORPHOLOGY

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Abstract. Technological properties of powders depend on their granulometry and particle morphology. Most of the image analysis methods provide only average size parameters (diameter, area, perimeter, etc.) which are often inadequate for characterizing the morphology of the particles. For particle abrasivity characterization, angularity factors are more sensitive. This paper studies different parameters for describing the hardmetal powder granulometry and morphology. The main aim is to find numerical descriptors for the shape factors that adequately characterize the hardmetal powders produced by the mechanical method – milling in a disintegrator. For the characterization of the powder particle morphology, irregularity parameter *IP* (relation of the diameters of the maximum inscribed and minimum circumscribed circles) suits best, taking into account the ellipticity and irregularity of the particle form. For describing the angularity of ground powders, the so-called “spike parameter – quadratic fit” can be used.

Key words: hardmetal powder, granulometry, morphology, shape factor, angularity

1. INTRODUCTION

Main characteristics of powders are the particle size (granulometry) and particle shape (morphology). Technological properties of powders (bulk density, flowability, surface area, etc.) as well as the potential areas of their application depend on these characteristics. Using powders for thermal spray, the preferable particle shape is spherical in order to have high flowability of powders and

optimal conditions of particle melting and spraying. These are preconditions for producing high density and high wear resistant sprayed or melt coatings. Using hard powder in abrasive tools, particles must have high abrasivity (angularity). The same problem occurs with abrasive wear since the wear caused by abrasive grits depends on their size, shape, hardness, etc. The results of laboratory tests confirm that a direct correlation exists between the abrasive particle shape and the wear rate. Namely, the increase in particle angularity results in a significant increase of abrasive wear.

The granulometry of powders can be determined by different methods (sieve analysis, image analysis, laser analysis, etc.), but the question is how adequately they describe the powder granulometry. In [1] we showed that the evaluation of coarse powder granulometry (particle size more than 50 μm) by means of sieve analysis guarantees sufficiently good results. The granulometry of the powder is adequately described by the modified Rosin–Rammler (Weibull) distribution function, and the method may be used to characterize powders produced by collision. Evaluation of the fine powder granulometry (with particle size less than 50 μm) is more difficult. The results of the sieve analysis do not describe adequately the powder granulometry. As shown in [2], comparison of powder granulometry determined by sieve and laser analyses reveals considerably different results – the laser analysis provides finer granulometry of the powder. It shows that sieve analysis is inadequate and unsuitable for fine powder classification. Due to high speed and accuracy, particle evaluation by means of image analysis increases considerably the quality of the evaluation methods, particularly for fine powders [2].

Size parameters (for example, average diameter, area or perimeter) describe a geometrical object independently of its shape. The morphology of a powder particle is characterized by description (spherical, angular, dendritic, dish-shaped, acircular) or quasi-quantitatively, for example, by means of geometrical shape parameters. The shape parameter characterizes mainly the shape, without considering the size. Qualitative descriptions of particle visual appearance, such as “rounded”, “semi-angular” or “angular”, have been used to classify and differentiate between various groups of abrasive particles. Several attempts have been made to characterize particle shape using various numerical descriptions [3-5]. The aim of this paper is to find new numerical descriptors for characterizing the hardmetal powders.

2. METHODS OF CHARACTERIZING THE PARTICLE SHAPE

To characterize particle shape, different shape factors are calculated. Often ellipticity parameters are used. Ellipticity can be effectively measured and calculated. To characterize ellipticity, aspect AS is used [1,2]

$$AS = a/b, \quad (1)$$

where a and b are the axes of the Legendre ellipse (Fig. 1). The Legendre ellipse is an ellipse with the centre in the object's centroid and with the same geometrical moments up to the second order as the original object area. The Legendre ellipse is often used instead of the original object [1]. The shape factor, elongation EL , is defined as

$$EL = \log_2(a/b), \quad (2)$$

and dispersion DP as

$$DP = \log_2(\pi ab). \quad (3)$$

The relationship between EL and AS can be expressed as follows:

$$EL = \log_2(a/b) = \log_2 AS = \log AS / \log 2, \quad (4)$$

$$AS = 2^{EL}. \quad (5)$$

The elongation of a circle is zero and of an ellipse with the ratio of axes 1:2 it is equal to one. Elongation grows for the ellipses with an increase in the ratio of the major and secondary axes (Fig. 2). Dispersion is a measure which allows one to evaluate differences in smoothness. It makes possible to compare smoothness of the evaluated object with the ideal ellipse: dispersion of an ellipse is zero and it grows with the increase of the object's roughness (Fig. 3).

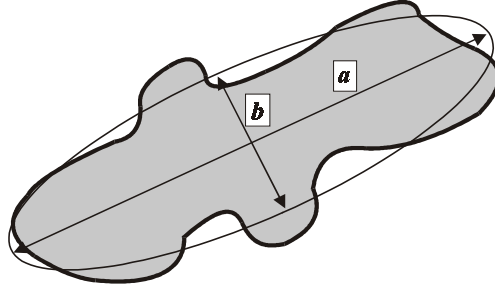


Fig. 1. Morphology study based on the Legendre ellipse.

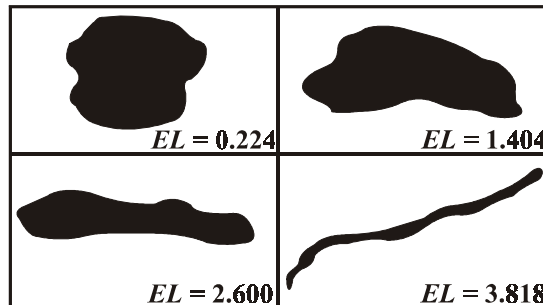


Fig. 2. Samples with different values of the elongation EL .

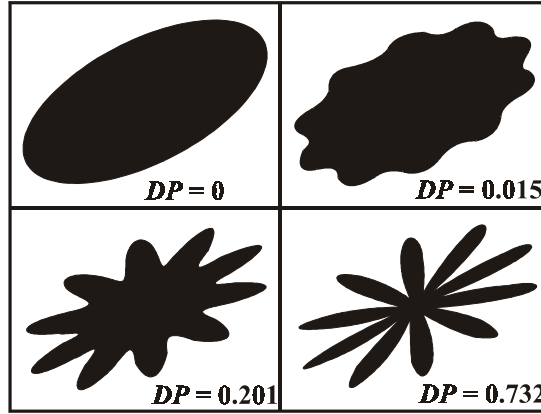


Fig. 3. Samples with different values of the dispersion DP .

Another well-known and often used characteristic for particle irregularity characterization is roundness of the object RN . If P is the perimeter and A is the area of the object, then roundness is defined as [1,3]

$$RN = P^2 / 4\pi A. \quad (6)$$

The roundness of a circle is equal to one. If the object's shape approaches a line segment, it approaches infinity. To characterize the roundness, sometimes shape factor $SF = 1/RN$ is used. The roundness factor proposed in [4], RNF , is defined by the relationship

$$RNF = P/d_A, \quad (7)$$

where d_A is the diameter of the circle with the same area as the particle (Fig. 4).

To evaluate the ellipticity and irregularity of the shape of the particle, in [5] the so-called irregularity parameter IP is defined

$$IP = D/d, \quad (8)$$

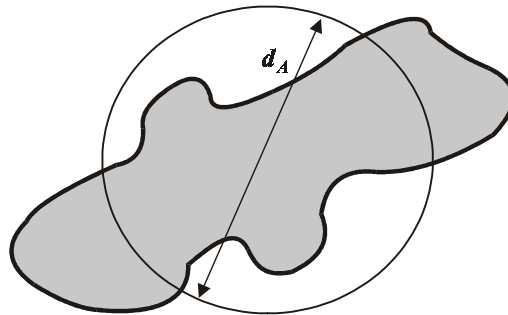


Fig. 4. Morphology study based on the circle with the same area as the particle.

where d and D are the diameters of the maximum inscribed and minimum circumscribed circles, respectively (Fig. 5). As shown in [5], IP can be applied both to elongated and irregular particles, but it does not allow to decide whether the particle is elongated or irregular.

A very important parameter of powder particles is their angularity. Several attempts have been made to characterize abrasive particle angularity using various numerical descriptors and to correlate particle shape with its ability to abrade or erode. For that AS and RN can be used, but as shown in [6], they are not sufficiently sensitive to angularity. Investigating the influence of the shape on the erosion rate, in [7] the ratio of the radius of the tip r to the “mean” radius of the particle R was used as the shape factor. Values of the shape factor r/R are in the range from 1.0 (sphere) up to 2.3–2.7 (cast iron debris).

In [8,9] the particle angularity was described by a numerical parameter called “spike parameter – linear fit” SP . A particle boundary is “walked” around with a fixed step, represented by a set of triangles with the height h and apex angle θ (Fig. 6a). It has been assumed that the sharpness and size of these triangles are directly related to particle abrasivity. Spike value SV is defined as

$$SV = h \cos \frac{\theta}{2}, \quad (9)$$

and SP is calculated as

$$SP = \frac{1}{n} \sum \frac{1}{m} \sum \frac{SV_{\max}}{h_{\max}}, \quad (10)$$

where m is the number of valid SV for a given step size and n is the number of different step sizes used. In this method, many thousands of triangles are to be calculated for each boundary in order to obtain SP .

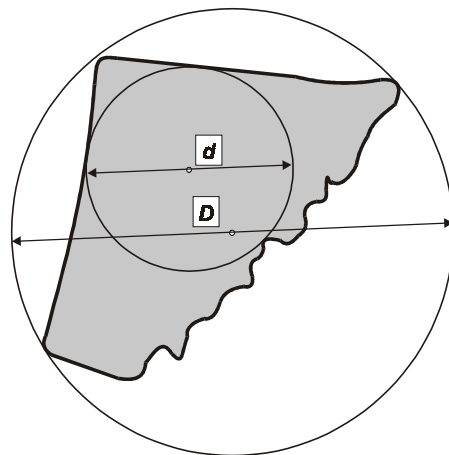


Fig. 5. Morphology study based on the maximum inscribed and minimum circumscribed circles.

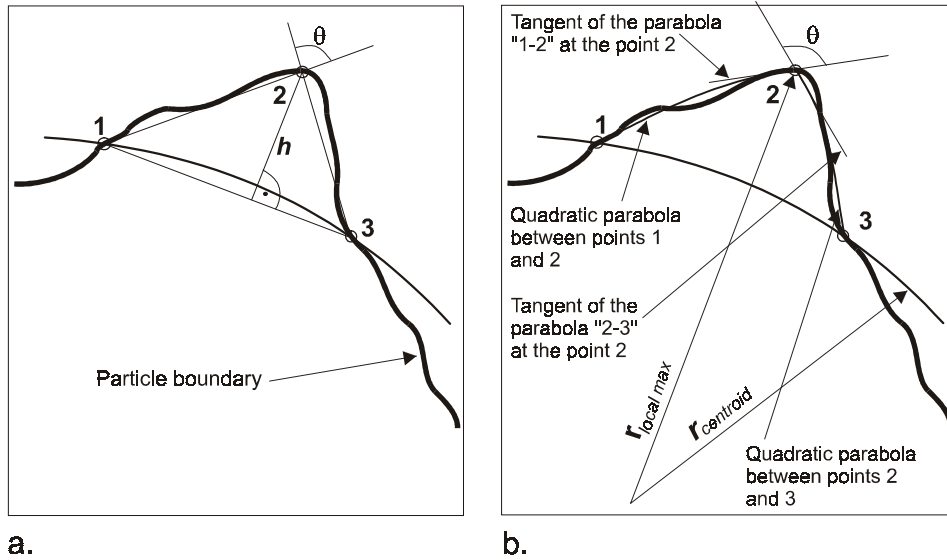


Fig. 6. Schematic illustration of calculation methods of SP by constructing triangles round the boundary (a) and of SPQ based on fitting quadratic segments to protruding sections of the particle boundary (b).

A modified particle angularity parameter, called “spike parameter – quadratic fit” SPQ , was developed by the same authors to eliminate the deficiencies of the SP method [9]. The parameter SPQ takes into account only those spikes that are outside the circle with equal particle area centred over the particle centroid. The areas outside the circle, spikes, are regarded as areas of interest, while those inside the circle are omitted. For each protrusion outside the circle, i.e., spike, the local maximum radius is found and this point is treated as the spike’s apex, as shown in Fig. 6b. The sides of the spike, which are between the points 1–2 and 2–3 are represented by fitting quadratic polynomial functions. Differentiating the polynomials at the point 2 yields the apex angle θ and the spike value SV , i.e., $SV = \cos(\theta/2)$. SPQ is calculated as

$$SPQ = SV_{av}. \quad (11)$$

From [10] it follows that SPQ is very sensitive; for different abrasives of +0.25–0.3 mm (glass, silica sand, garnet, quartz, alumina, SiC) the value of SPQ is in the range of 0.0231–0.6008. The effects of the particle angularity on two-body abrasive rates are explained in [11].

3. APPLICATION OF PARTICLE SHAPE PARAMETERS FOR CHARACTERIZING DISINTEGRATOR MILLED HARDMETAL POWDERS

The technology of producing hardmetal powders from the used (recycled) WC-Co hardmetal consists of preliminary cyclic thermal treatment and mechanical size reduction – milling of pretreated particles by collision [12,13]. To obtain hardmetal powder with the predetermined granulometry, multistage milling (up to 16 times) was used (specific energies of milling are given in Table 1).

Table 1. Milling parameters (disintegrator DSL 160, velocities of rotors 8000/8000 rpm)

| Multiplicity of milling | Specific milling energy, kJ/kg | Multiplicity of milling | Specific milling energy, kJ/kg |
|-------------------------|--------------------------------|-------------------------|--------------------------------|
| 1 | 12.4 | 9 | 111.6 |
| 2 | 24.8 | 10 | 124.0 |
| 3 | 37.2 | 11 | 136.4 |
| 4 | 49.6 | 12 | 148.8 |
| 5 | 62.0 | 13 | 161.2 |
| 6 | 74.4 | 14 | 173.6 |
| 7 | 86.8 | 15 | 186.0 |
| 8 | 99.2 | 16 | 198.4 |

Granulometry and morphology studies of the powders were carried out using two methods:

- a) optical microscope transmission method, to produce the projections of powder particles,
- b) optical microscope reflection method, to obtain the images of the cross-section polish.

Particle shape depends on the duration of milling: with an increase of time, milling particle obtains smooth spherical shape. In Table 2, the results of milling WC-Co hardmetal in a disintegrator are given [12,13]. The shape factors EL , DP , and RN were determined by two different image analysis systems: DIPS [14] and Image-Pro Plus 3.0 [15]. Their dependence on the number of disintegration cycles is shown in Fig. 7.

The elongation measured with DIPS drops monotonically while with Image-Pro it has a maximum 0.81 after 3 cycles. Generally, the values of the elongation vary only in a small range – maximum 0.82 corresponds to the ellipse with the axes ratio 7:4 and the minimum 0.51 to the axes ratio 7:5. The dependence of DP on the multiplicity of milling is monotonous, i.e., particles become smoother after every disintegration cycle.

The value of RN depends weakly on the multiplicity of milling and also on the method of image processing (Table 2, Fig. 7).

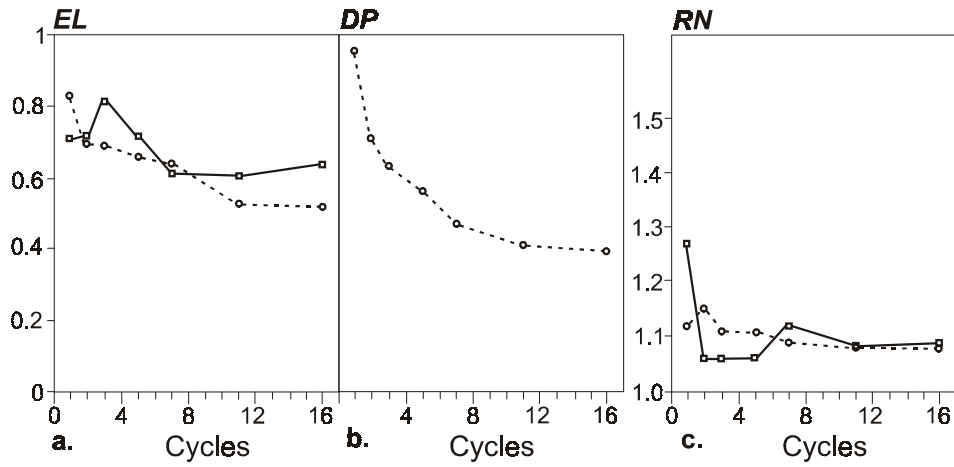







Fig. 7. Dependence of the average values of the elongation *EL* (a), dispersion *DP* (b) and roundness *RN* (c) on the number of desintegration cycles (---- DIPS, — Image-Pro).

Table 2. Shape factors of milled WC-Co hardmetal powder particles

| Shape factor | Method of investigation | Multiplicity of milling | | | | | | |
|--------------|-------------------------|-------------------------|-------|-------|-------|-------|-------|-------|
| | | 1 | 2 | 3 | 5 | 7 | 11 | 16 |
| <i>AS</i> | Image-Pro | 0.71 | 0.72 | 0.81 | 0.72 | 0.61 | 0.60 | 0.64 |
| <i>EL</i> | DIPS | 0.82 | 0.69 | 0.68 | 0.65 | 0.63 | 0.52 | 0.51 |
| <i>DP</i> | DIPS | 0.095 | 0.071 | 0.064 | 0.056 | 0.047 | 0.041 | 0.039 |
| <i>RN</i> | Image-Pro | 1.27 | 1.06 | 1.06 | 1.06 | 1.12 | 1.08 | 1.09 |
| <i>RN</i> | DIPS | 1.12 | 1.15 | 1.11 | 1.11 | 1.09 | 1.08 | 1.08 |

In Table 3, the shape factors of regular configurations are given in the order of increasing sharpness: circular, orthogonal, hexagonal, quadrangular, and triangular. As it follows from Table 3, *IP* is the most sensitive parameter and *RNF* is the most insensitive one. Whereas *IP* characterizes integrally both angularity and elongation, it does not allow to find separately angularity and elongation. In our case (mechanically milled hardmetal powder), where the powder particles are not elongated in shape (Fig. 8), *IP* is suitable for particle angularity characterization.

Table 3. Shape factors of particles of different form

| Shape factor | Configuration | | | | |
|--------------|---|---|---|---|---|
| |  |  |  |  |  |
| <i>RN</i> | 1.0 | 1.055 | 1.103 | 1.273 | 1.654 |
| <i>RNF</i> | 1.0 | 1.027 | 1.050 | 1.128 | 1.286 |
| <i>IP</i> | 1.0 | 1.082 | 1.155 | 1.414 | 2.0 |
| <i>SPQ</i> | 0 | 0.383 | 0.5 | 0.707 | 0.866 |

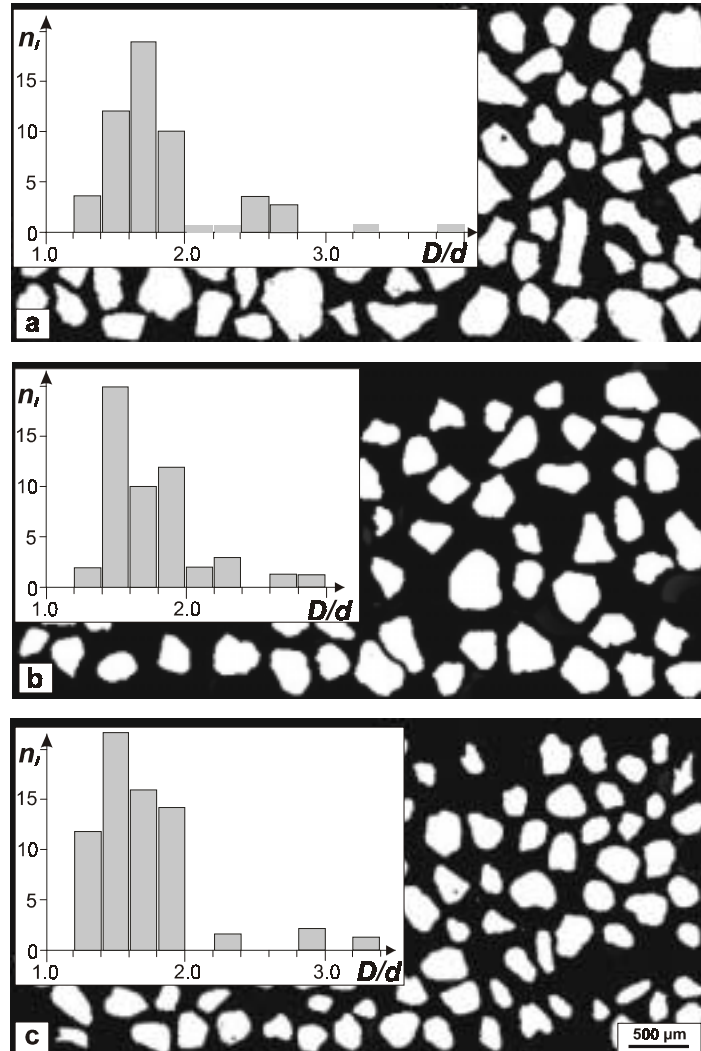


Fig. 8. Powder particle cross-sections and shape factor IP histograms; multiplicity of milling: a – 2, b – 5, c – 15.

Using finely polished metallographic samples and photographs of cross-sections of powder particles (Fig. 8), diameters D and d were measured and IP was calculated. Results of calculation are given in Table 4 and on histograms in Fig. 8. Table 4 shows the results of multiple fine milling of hardmetal powder, preliminarily ground in a coarse grinder to the granulometry less than 2.5 mm (number of milling cycles up to 30–40). From Table 4 follows that the shape factor D/d (both average and median) decreases during multiple milling only a little because of the preliminary multistage milling. Mode is not expressed

particularly clearly – the maximums of the histograms (Fig. 8) are smoothed and remain more or less unchanged during the multistage milling. At the same time, an increase in the share of finer fraction ($D/d \leq 1.6$) is observed in the multistage milling process. Figure 8 shows also that the two left columns of the histograms increase when the number of treatments increases.

Table 4. Characteristics of the disintegrated WC-Co hardmetal powder

| D/d | Multiplicity of milling and number of particles | | | | | |
|-------------------------------|---|------|------|------|------|------|
| | Initial | 1 | 2 | 5 | 10 | 15 |
| 1.00–1.20 | 0 | 0 | 0 | 0 | 0 | 0 |
| 1.21–1.40 | 4 | 4 | 3 | 2 | 10 | 11 |
| 1.41–1.60 | 12 | 22 | 23 | 20 | 15 | 22 |
| 1.61–1.80 | 18 | 24 | 20 | 9 | 19 | 16 |
| 1.81–2.00 | 10 | 12 | 13 | 12 | 2 | 14 |
| 2.01–2.20 | 1 | 3 | 2 | 2 | 4 | 0 |
| 2.21–2.40 | 1 | 4 | 4 | 3 | 2 | 2 |
| 2.41–2.60 | 4 | 1 | 2 | 0 | 1 | 0 |
| 2.61–2.80 | 3 | 3 | 1 | 1 | 1 | 0 |
| 2.81–3.00 | 0 | 0 | 2 | 1 | 0 | 3 |
| 3.01–3.20 | 0 | 0 | 0 | 0 | 1 | 0 |
| 3.21–3.40 | 1 | 1 | 1 | 0 | 0 | 2 |
| 3.41–3.60 | 0 | 0 | 0 | 0 | 0 | 0 |
| 3.61–3.80 | 0 | 0 | 0 | 0 | 0 | 0 |
| 3.81–4.00 | 1 | 0 | 0 | 0 | 0 | 0 |
| Total number of particles n | 55 | 74 | 71 | 50 | 55 | 70 |
| Average $\frac{D}{d}$ | 1.89 | 1.79 | 1.81 | 1.74 | 1.71 | 1.76 |
| $\frac{D}{d} \leq 2.0$, % | 80 | 84 | 83 | 86 | 84 | 90 |
| $\frac{D}{d} \leq 1.6$, % | 29 | 35 | 37 | 44 | 45 | 47 |
| Standard deviation of D/d | 0.51 | 0.36 | 0.40 | 0.34 | 0.38 | 0.44 |
| Median of $\frac{D}{d}$ | 1.75 | 1.67 | 1.70 | 1.64 | 1.64 | 1.64 |
| Mode of $\frac{D}{d}$ | 1.6 | 1.6 | 1.6 | 1.5 | 1.6 | 1.5 |

Figure 9 shows the photographs of cross-sections of the analysed powder particles after 2, 5, and 15 times of milling. Table 5 presents changes in the parameter SPQ , characterizing the dependence of powder particle angularity on the number of milling cycles. It shows that the circularity of coarse fraction increases (parameter SPQ decreases) with an increase of the number of milling

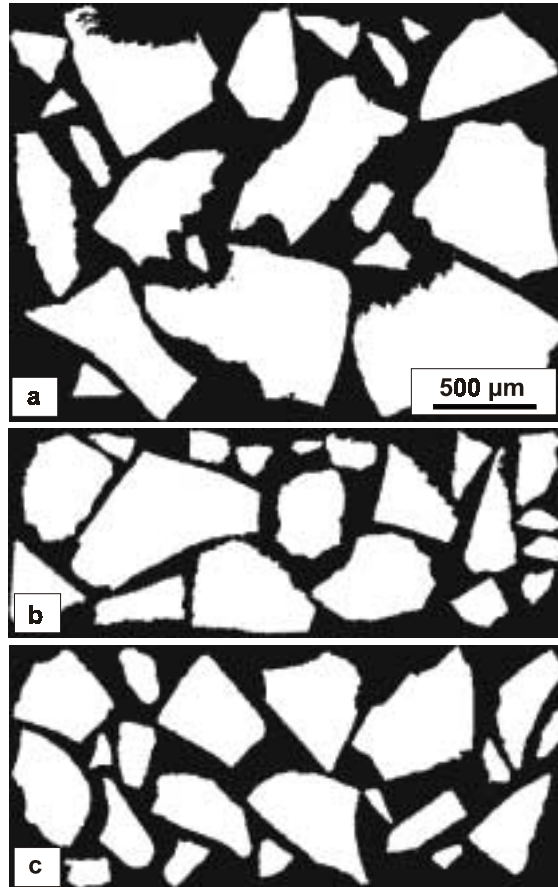


Fig. 9. Powder particle cross-sections; multiplicity of milling: a – 2, b – 5, c – 15.

Table 5. Angularity parameter SPQ of disintegrated WC-Co hardmetal powder

| Characterization of powder particles | Multiplicity of milling | | |
|--|-------------------------|-------|-------|
| | 2 | 5 | 15 |
| Coarse fraction (particle size 0.3–1 mm, 10 particles) | 0.652 | 0.585 | 0.522 |
| Fine fraction (particle size 0.1–0.3 mm, 10 particles) | 0.645 | 0.648 | 0.630 |
| Average | 0.649 | 0.617 | 0.576 |

cycles. At the same time, SPQ of particles of fine fraction does not decrease essentially. It means that fine fraction is the product of direct fracture of milling by collision. The circularity increases (angularity decreases) considerably by increasing number of milling cycles (Figs. 9, 10).

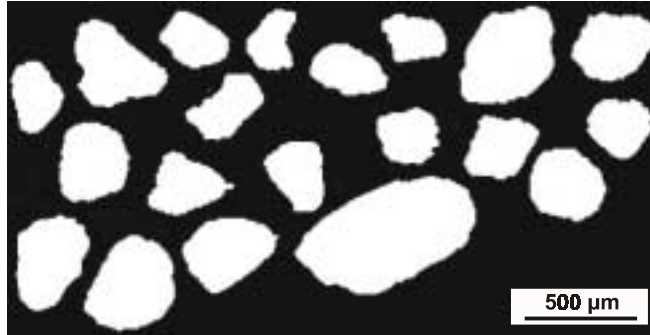


Fig. 10. Powder particle cross-sections milled about 45 to 50 times, $SPQ = 0.258$.

4. CONCLUSIONS

1. The irregularity parameter IP and spike parameter SPQ , the numerical descriptors of the shape factor that adequately characterize the particle morphology, may be used to characterize powders produced by mechanical methods. With an increase in the angularity of regular configuration (from circular to triangular), the values of IP and SPQ are in the range of 1.0–2.0 and 0–0.866, respectively.

2. The parameter SPQ , studied for characterizing WC-Co hardmetal powders produced by multiple disintegrator milling, is a more sensitive characteristic of angularity than IP . For disintegrator ground powders, the values of SPQ are in the range of 0.652–0.258. At the same time, the average values of IP that takes into account the ellipticity and irregularity of particles are in the range of 1.89–1.76.

3. The parameters IP and SPQ may be used to classify various groups of WC-Co hardmetal powders: powders for abrasives and powders for wear resistant composite coatings.

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PULBRIOSAKESTE MORFOLOOGIA KIRJELDAMINE

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Pulbrite tehnoloogilised omadused sõltuvad eelkõige nende granulomeetriast ja pulbriosakeste morfoloogiast. Osakeste suurust ja kuju iseloomustavad näitajad (diameeter, pindala, perimeeter jt.), mis on saadud kujutisanalüüsi tulemusel, ei kajasta sageli piisava täpsusega pulbriosakeste morfoloogiat, näiteks osakeste nurgelisust. Töös on vaadeldud pulbriosakeste morfoloogiat puudutavaid parameetreid, mis võimalikult adekvaatselt kirjeldavad desintegraatorjahvatusega saadud kõvasulampulbreid. Sobivaimad parameetrid kõvasulampulbri osakeste morfoloogia puhul on irregulaarsusnäitaja *IP*, mis integreeritult arvestab osakeste elliptilisust ja ebakorrapärasust, ning nurgelisuse näitaja *SPQ*.