CHARACTERIZATION OF DISINTEGRATOR MILLED HARDMETAL POWDER

P. Kulu, V. Mikli, H. Käerdi, M. Besterci

Abstract
Technological properties of powders milled by collision depend on their granularity and powder particle morphology. The grindability of hardmetal powder, the dynamics of milling and changes in particle granularity and morphology were studied. For particle morphology analysis the irregularity parameter as well as particle roundness and aspect ratio were determined. To characterize the particle granularity, the mean particle size was measured and volume distribution function, median of particle size and probability density function of the particles volume were calculated. For describing the angularity of milled powders, the so-called "spike parameter – quadratic fit" (SPQ) was properly described and real experiments for determination of SPQ sensitivity and precision to characterize particle angularity were performed. Using the coefficients of variation for SPQ and particles shape parameters, the changes in dispersions dependent upon milling duration were investigated. Experiments for determination of the influence of hardmetal particle angularity on erosion rate of different metallic materials show the essentiality of this parameter in powder shape characterization.
Keywords: hardmetal powder, recycling, grindability, image analysis, angularity, abrasivity

INTRODUCTION

Recycled metals are becoming increasingly important as industry responds to public demands that resources be conserved and the environment be protected [1]. Hardmetal (WC-based) wastes are a part of metal scrap. With the prices of hard metal rising, their recycling has become an urgent issue. One of the ways for the recycling of old materials is to produce the powdered materials from worn products [2,3]. To produce materials competitive in cost and price with primary producers, secondary producers will have to pursue new technologies and other innovations [1]. Recycling of metal wastes and worn materials in a disintegrator mill is one of the promising technologies for the energy saving production of powdered materials with different properties.

The main properties of powders are: their particle size – granularity and particle shape – morphology. Technological properties of powders (bulk density, flowability, compactibility etc.) as well as the potential areas of their application, depend on their granularity and morphology. In powder used for thermal spray, their preferable particle size is spherical to have a high flowability of powders and optimal conditions of particle spraying and their melting. These are preconditions for producing high density and high wear resistant sprayed or melt coatings. In hard powder used as abrasive material in

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abrasive tools, particles must have high abrasivity (angularity). The same problem occurs with abrasive wear, 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 abrasive particle shape and wear rate - increases in particle angularity result in a significant increase in abrasive wear.

The morphology of powder particle is mostly evaluated by the means of a geometrical shape parameter. The shape parameters characterize mainly the shape, without any influence on the size, and are presented in dimensionless form.

Several attempts have been made to characterize particle shape using various numerical descriptions [4-7].

EXPERIMENTAL MATERIALS, METHODS AND RESULTS

Grindability Studies

The fracture of particles by collision and size reduction of ground product can take place in one of two ways [8]:

- direct fracture as a result of intensive stress waves originated by high velocity collisions;
- low cyclic fatigue fracture as a result of numerous local plastic deformations due to the collisions.

Due to the high price and big amount of used (worn) hardmetal material, its recycling (retreatment and reuse) is significant. The production of hardmetal powder by milling is one of the methods for retreatment of used hardmetal. The grindability [9] and obtained powder characteristics are studied more profoundly.

The technology of producing hardmetal powder was composed of [9,10]

- preliminary thermo-cyclical treatment and mechanical size reduction of worn hardmetal parts in a centrifugal-type mill by collision;
- final milling of pretreated particles by collision in a disintegrator mill.

The preliminary size reduction of hardmetal parts in the disintegrator mill DS-350 was carried out. As it follows from the metallographic studies, the particles were primarily equiaxed. Fine powder as the final product, with a particle size less than 500 μm suitable for thermal spray and fusion, was one object of the study; coarse powder with particles more than 1 and less 2.5 mm was taken as the initial powder for subsequent final milling. To produce powder with particles less than 100 μm, final milling was carried out by the laboratory disintegrator system DSL-160 [7,9]. Particle size was determined by sieving analysis. The grindability curve of a hardmetal powder with an initial particle size 2.5 mm is shown in Fig.1. As it follows from the metallographic studies, the particle shape of multi-milled powder was mainly isometric.

Based on the study of grindability and the fracture mechanism of a hardmetal as an example of a brittle composite material, we can state that hardmetal milling takes place as the result of direct fracture. To study the size and shape characteristics of powder particles dependent on milling cycles in DSL-160, the 1X, 2X, 4X, 8X, 16X, and 32X milled powder was taken under observation.

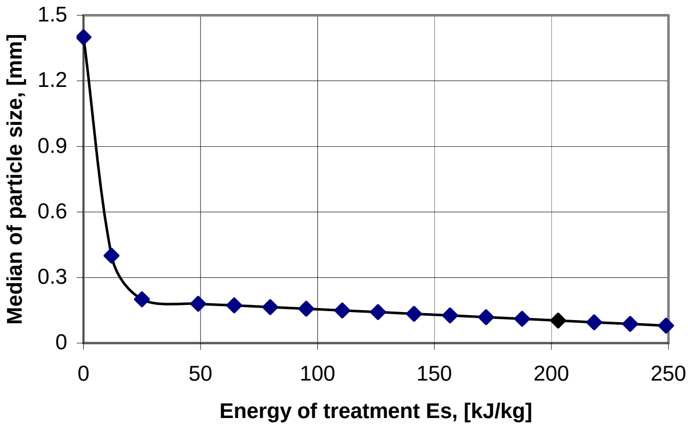


Fig.1. Dependence of the final product – hardmetal powder particle size on the specific energy of milling by sieving analysis.

Investigated powder particle size and shape parameters

To describe the particle size and shape, the image analysis method was used. Image analysis was carried out by an image processing system, which consisted of scanning electron microscope (Jeol JSM-840A), video transferring system, and Image-Pro 3.0 (with Materials-Pro) image analysing programs [11]. For characterization of particle size and shape, the following parameters are investigated:

for particle size characterization

- mean diameter d_m is the average from distances between particle centroid and particle boundary, measured by 5 degree intervals around the centroid;
- cumulative volume distribution function F_V of the particles volume;
- median diameter d_{50}, which corresponds to the half of the total content of powder obtained from cumulative distribution function;
- volume density function f_V, calculated by formula (1):

$$f_V = \frac{F_V^{i+1} - F_V^i}{\log d_{i+1} - \log d_i} \quad (1)$$

for particle shape characterization

- aspect (particle ellipticity) *AS* reports the ratio between the major axis and the minor axis of the Legendre ellipse equivalent to the object (i.e., an ellipse with the same area, first and second geometrical moments);
- roundness (roughness) *RN;* obtained by the formula (2)

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

where *P* is perimeter; *A* is area of the particle

- irregularity parameter *IP* proposed in [6], which is the ratio between minimum circumscribed and the maximum inscribed circles.

In its character, the *IP* is similar to the other used shape factor- Short/Long ratio [12], which is defined as the ratio of minimum diameter to maximum diameter of the projected image of the particles.

Changes in particle size and shape during milling process

Particle shape depends on the duration of milling: with an increase in time, the larger sized particle shape approaches spherical with a smooth surface (Fig.2).

Table 1 shows the results of granularity and morphology studies of the disintegrator milled WC-Co hardmetal powders.

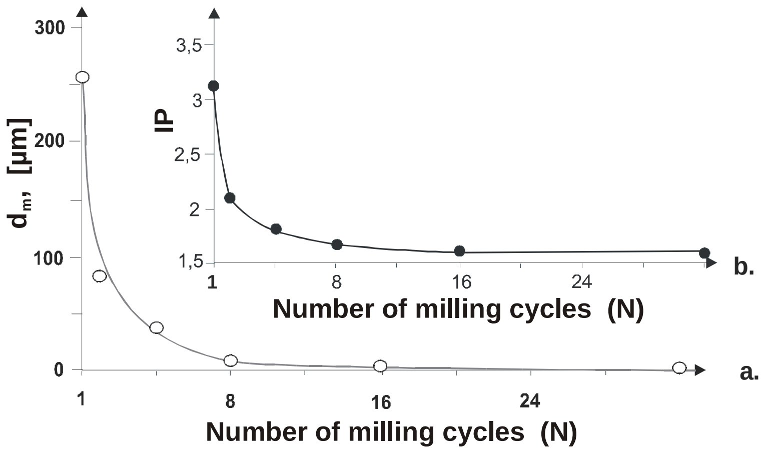


Fig.2. Dependence of (a) particle mean diameter dm and (b) particle shape parameter IP on multiciplicity of milling by image analysis.

Tab.1. Particle size and shape parameters of disintegrated WC-Co powder (for *IP* calculation only a coarser fraction was used).

Particle size and shape parameters	Multiciplicity of treatment					
	1X	2X	4X	8X	16X	32X
Mean diameter d_m, μm	257.5	83.4	36.6	9.1	2.5	2.0
Median diameter d_{50}, μm	561	209	144	88	57	30
Irregularity parameter *IP*	3.67	2.28	1.63	1.86	1.53	1.6

To describe the hardmetal powder particle size, the cumulative volume distribution function F_V and volume density function f_V (by formula (1)) were calculated as well. Fig.3 presents the cumulative distribution function F_V obtained by image analysis. An increase in the number of milling cycles causes the F_V curve shift to left (to the direction of smaller particles). At the same time, the left side of the curve shifts upward. It means that the currently used system didn't distinguish smaller particles, and F_V could be used as an indicator for accuracy of the analysis. For instance: in the case of 32x milled powder, 20% of particles are smaller than d = 10 μm (log d = 1, Fig.3). Median diameter d_{50} is calculated from the experimental cumulative distribution functions (Fig.3) and presented in Tab.1. As seen from the image analysis data in Tab.1, the d_m is 2-3 times smaller than d_{50} due to a different nature of descriptors (d_m- linear descriptor; d_{50}- volume descriptor). According to the methodics described in [13], the probability density function f_V of particle volume is calculated by formula (1) using the data obtained from the image analysis. The results are given in Fig.4. The f_V curve of 2X milled powder has 2 maxima (at approx. 150 and 350 μm).

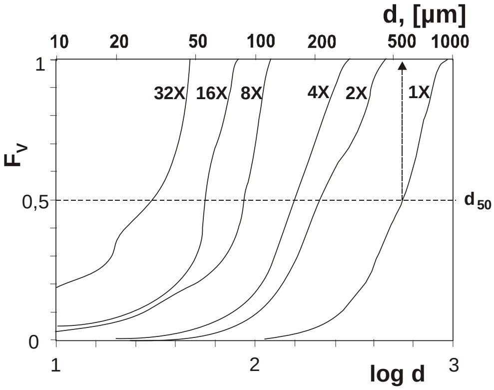


Fig.3. The cumulative distribution functions of particle volume F_V of the image analysis at different multiplicity of milling.

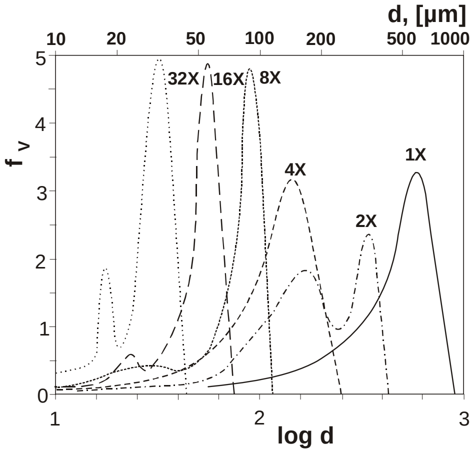


Fig.4. The probability density function of particle volume f_V of image analysis at different multiplicity of milling.

ANGULARITY STUDIES

To characterize, special angularity studies on 1X and 1X+8X milled WC-9Co hardmetal powders were carried out. In addition to angularity, the main parameters for the particle size and shape characterization were measured. The preliminary milling (1X) was performed in the DS-350. To observe the milling dynamics and changes in the particle shape dependent on E_s, the preliminary milled hardmetal particles were treated to 8 cycles in DSL-160. Figures 5 and 6 show the investigated 1X and 1X+8X milled WC-Co particles respectively. The main characteristics of the particle size and shape are presented in Tab.2. Particle shape descriptors, as well as angularity parameters, were obtained using images made at cross-section polishes of the investigated WC-Co powder particles (Fig.6).

The micrographs of WC-9Co hardmetal powder particles after preliminary milling in DS-350 (Fig.6a) and followed with 8 times treatment in DSL-160 (Fig.6b), respectively show the observable difference in the particle shape. To avoid errors in the angularity parameter SPQ, and wear calculations caused at variations in particle size the disintegrated product was preliminarily classified using sieving methodics (see Tab.2, d_m). The particle size was between 180 and 500 μm. Compared with 1X milled material, 1X+8X milled powder particle shape is more spherical (Tab.2, AS decreases) and regular (IP decreases), and particle surfaces are smoother (RN decreases).

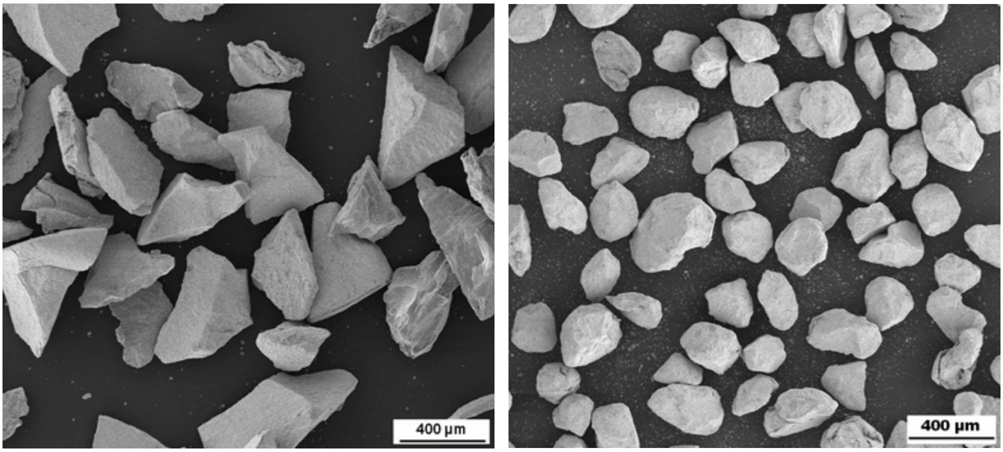


Fig.5. SEM picture of investigated WC-Co powders: a - 1X milled; b - 1X + 8X milled.

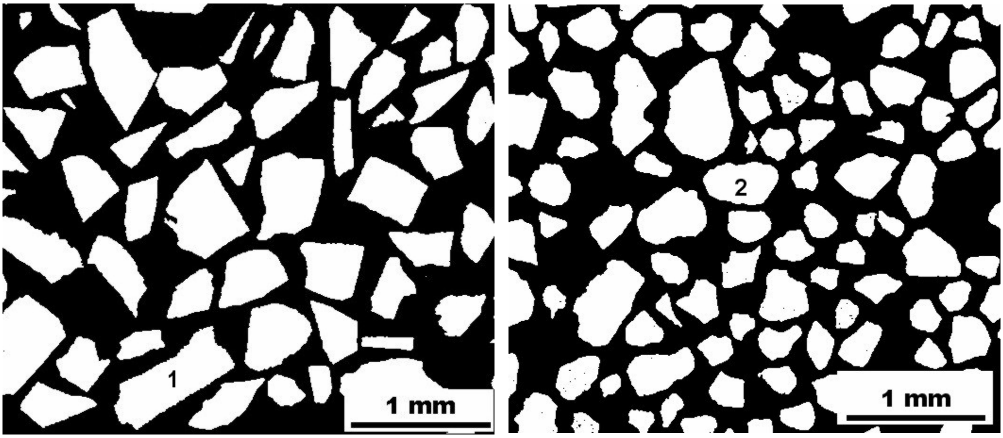


Fig.6. Binary images at cross-sections of investigated WC-Co powders: a – 1X milled; b – 1X + 8X milled.

Tab.2. Main characteristics of the powder particles.

Size and shape factors	Number of milling cycles	
	1X	1X + 8X
Mean diameter; d_m μm	316	235
Aspect ratio; AS	2.00	1.51
Roundness; RN	1.52	1.21
Irregularity parameter; IP	2.43	1.75

Particle angularity and SPQ calculation

In spite of the difference in particle angularity, it is clearly observable (Figs.5,6) that the numerical description of this difference needs a calculation of the particle angularity parameter called "spike parameter – quadratic fit" (SPQ), developed by Stachowiak [14]. The SPQ parameter takes into account only those spikes (Fig.7a) that are outside the circle with equal particle area centered over the particle centroid. Fitting quadratic polynomial functions represents the sides of the "spike". Differentiating the polynomials at the point "2", (Fig.7a), yields the apex angle Θ and the spike value SV=cosΘ/2. The SPQ is calculated as an arithmetical mean of spike values:

$$SPQ = SV_{average} \quad (3)$$

Figure 7b demonstrates the measuring of the apex angles with the case of real particles. In the upper side the 1X milled powder particle is presented (particle 1 in Fig.7b), on the lower side the 1X + 8X respectively (particle 2 in Fig.7b) [15].

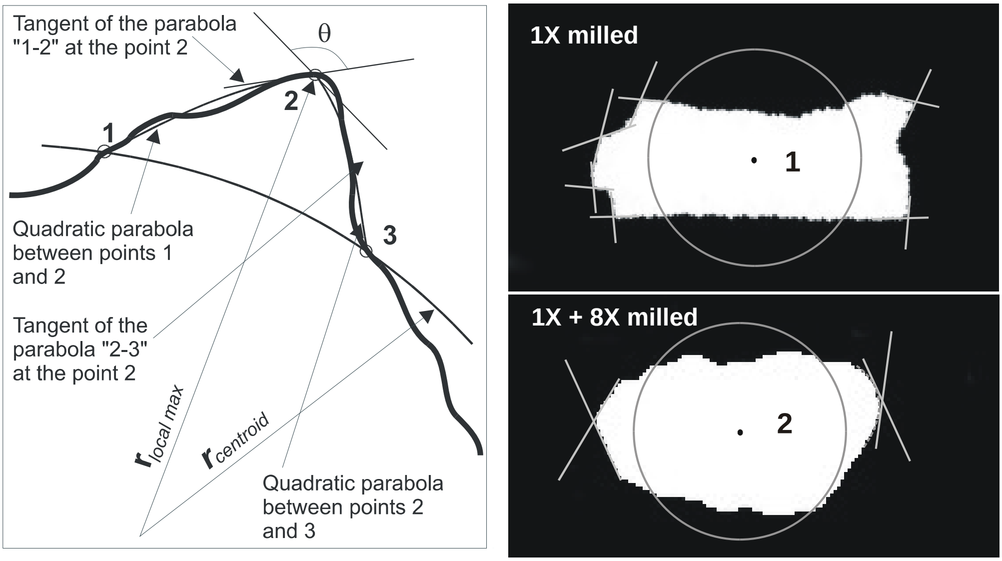


Fig.7. Determination of the apex angle Θ: a - basic principles; b - for real particles 1 and 2 from the Fig.6a and 4b respectively.

Tab.3. Particle angularity and coefficients of variation.

Particle angularity and coefficients of variation	Number of milling cycles	
	1X	1X + 8X
SPQ	0.688	0.492
S_{SPQ}	0.132	0.162
S_{IP}	1.017	0.316
S_{AS}	0.821	0.424
S_{RN}	0.342	0.155
V_{SPQ}	19.2%	32.9%
V_{IP}	41.9%	18.1%
V_{AS}	41.4%	28.1%
V_{RN}	22.5%	12.8%

Table 3 presents the changes in the parameter SPQ, characterizing the dependence of powder particle angularity on the number of milling cycles. As it follows from Tab.3, the circularity of powder particles increases (parameter SPQ decreases) with an increase in the number of milling cycles. Obtained SPQ values are in the good accordance with earlier measured results [7]. Consequently, the above-described SPQ calculation methodics gives reproducible results and could be effectively used in particle angularity characterization. The obtained SPQ values for particles presented in Fig.7b were: SPQ_{1X} = 0.604; $SPQ1X_{+8X}$ = 0.362. For instance the SPQ values for a square are 0.707, for a hexagon 0.5 and for an octagon 0.383 [7].

Calculation of the coefficients of variation

Coefficient of variation is the ratio of the standard deviation (S) to the arithmetic mean, expressed as a percent. This relative measurement is used to compare the dispersions of two or more distributions of investigated parameters.

For the spike parameter SPQ, the coefficient of variation can be calculated by the equation (4):

$$V_{SPQ} = (S_{SPQ} / \ SPQ) \cdot 100\% \qquad (4)$$

Coefficients of variation (V) in Tab.3 shows the differences in the investigated parameter values at the same milling times. In the case of SPQ calculation, coefficient of variation increases from 19.2% to 32.9%. It means that differences in particle angularity increase noticeably. It is caused by the milling process – at first the disintegrator produces continuously off the small (1-10 µm) size particles from the top of the powder fraction (preferably from the tops of sharp edges); and secondly the particles were shattered into smaller ones, which causes the appearance of sharp angles.

Inversely to SPQ, the coefficient of variation for aspect, roundness and irregularity decreases considerably, which demonstrates the unification of the particle shape in disintegrator milling.

Abrasivity studies of hardmetal powder

Investigated WC-Co powders were used as an abrading material to study erosion wear at the different impact angles. Normalized steel C45 (0.45% C) and pure aluminium Al 99.5 (99.5 % Al) were used as reference materials. Erosion rate v_v was calculated by the formulae (5):

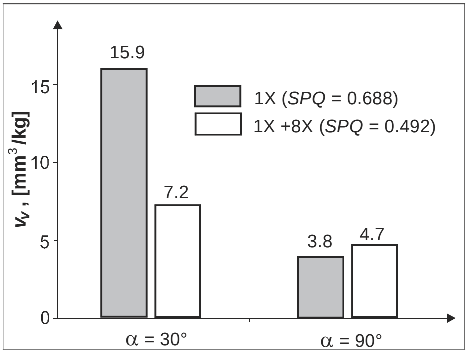
$$v_v = \frac{Material \quad loss \quad [g]}{Weight\ of\ abrasive\ [kg] \cdot density}; \quad \left[\frac{mm^3}{kg}\right] \qquad (5)$$

density of steel C45= 7.85 g/mm^3 and Al 99.5= 2.7 g/mm^3.

Differently, the methodics described in [16] the abrasive material had the same composition (hardmetal) and particle size. Only the particle shape and angularity was changed.

Based on the study of wear by abrasive particles with different angularity and the same hardness, direct correlation between the shape of abrasive particles and the wear rate at oblique impact was observed and presented in Fig.8.

Figure 8a shows the influence of angularity (angular - 1X and round - 1X+ 8X milled) of the abrasive to the wear rate of steel (0.45% C) at different impact angles. At low an operating angle (30°) the erosion rate is more than twice as big in the case of angular particles. At 90°, the wear rate didn't depend significantly on particle angularity and is slightly bigger in round particles. Figure 8b shows the same study, using pure aluminium as a target material. The difference in wear rate with angular and round abrasive particles is practically the same as by steel. Due to low hardness and high plasticity, the wear rate is at the small (30°) impact angle more than six times higher than in steel. At the normal impact (90°) the penetration of hardmetal particles to the Al target took place, and as a result, by the angular abrasive particles the weight of specimen increased (the wear rate is negative).



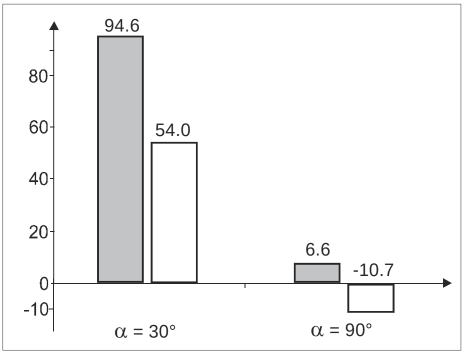


Fig.8. Influence of abrasive particle angularity on wear rate of metallic materials at different impact angles: a - steel C45; b – Al 99.5.

CONCLUSIONS

On the basis of the present study, the followed conclusions can be proposed:

- The disintegration process of WC-Co was studied and dependences of particle shape parameters on milling time were determined.
- According to the results, the major changes in particle size and shape took place during low grinding energies (1-4 cycles).
- Experiments for determination the influence of hardmetal powder particle angularity to erosion rate of different metallic materials, show the essentiality of this parameter in powder shape characterization.
- Substance of particle angularity was profoundly described, and real calculation methodics were proposed.

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