Size reduction by milling

Oldřich Holeček,
English translation by Michal Kordač

I Basic definitions

I.1 Introduction

The rate of many chemical and physical processes is determined by the size of interfacial area. If the solid phase is in place, an increase of the interfacial area is done by particle size reduction. By reducing the size of the solid particles the total interfacial area increases and the particle size distribution changes as well.

In chemical and metallurgical industry is the size reduction one of the basic operations, which consumes high amounts of electrical energy. Thus, correct choice of size-reduction equipment is an important task. As most of the knowledge of the process is based on empirical experience, the choice is often based on size reduction experiments. During these experiments, influence of different factors on the size reduction can be followed and in the end can improve the efficiency of the final process.

Size reduction experiments in the students laboratory is made in a ball mill. These mills are suitable for intermediate and fine reduction of abrasive materials. A typical ball mill has a cylindrical shell slowly turning about a horizontal axis. The shell is filled to about half its volume with a solid grinding medium. The grinding medium can be metal rods, chains or balls. In a ball mill, most of the size reduction is done by impact as the balls drop from near the top of the shell. When the mill is rotated, the balls are picked up by the mill wall and carried nearly to the top, where they break contact with the wall and fall to the bottom to be picked up again. Centrifugal force keeps the balls in contact with the wall and with one another during the upward movement. While in contact with the wall, the balls do some grinding by slipping and rolling over one another, but most of the grinding occurs at the zone of impact, where free-falling balls strike the bottom of the mill.

The faster the mill is rotated, the farther the balls are carried up inside the mill and the greater power consumption and the capacity of the mill. If the speed is too high, however, the balls are carried over and the mill is said to be centrifuging. The speed at which the centrifuging occurs is called the critical speed. An approximate estimate of the critical speed of the mill can be obtained by a balance of centrifugal and gravitational forces, as the centrifuging will occur when these two forces are in equilibrium:

\[ m_k g = m_k \omega^2 D / 2 \]  

(1)

where \( D \) is the shell diameter, \( m_k \) is ball weight and \( \omega \) is the angular velocity of the mill. The mill rotation frequency \( f \) can be calculated from the angular velocity:

\[ \omega = 2 \pi f \]  

(2)

Finally, combining relations (1) and (2) we obtain for the critical speed:

\[ \omega = \sqrt{2 g D / m_k} \]

(3)

By describing the movements of the balls we can conclude, that in the lower part of the mill the reduction is done by impacts and in the space between the balls attrition takes place.
So far, there is no method to determine the efficiency of the reduction. Very little is known of how much energy is needed for increasing the surface. Roughly we can estimate the energy is just a few per cent of the total power input of the mill. Operation of mills is more frequently characterized by amount of surface increase obtain per unit of energy input. While the energy is measured easily, the determination of the surface increase is more complicated. At some assumptions, it can be calculated from measurements of size distribution.

I.2 Determination of specific surface

Determination of size distribution is done by sieve analysis. A sample of material is sieved on a set of sieves and the amounts of material (fractions), which remained above the sieves, are weighted. If the particle size is well determined by the size of the neighbouring sieves, all particles have identical density and all of them approximately identical shape, following procedure can be made:

As the particle sizes in each fraction are not very different, we can idealize the fraction as being made of particles of identical shapes and of an average size only. In such fraction we can determine the weight and the surface:

\[ A_i = K_A l_i^2 n_i \]
\[ m_i = K_V l_i^3 n_i \rho \]

where \( l_i \) is characteristic size of the particles in fraction \( i \), \( n_i \) is number of particles and \( \rho \) is density of the particles. Index \( i \) denotes fraction, which remained on \( i \)-th sieve (numbered from the bottom up). \( K_A \) and \( K_V \) are shape factors (e.g. for spheres, the characteristic size is particle diameter, is \( K_A = \pi \) and \( K_V = \pi/6 \)). Dividing equations (4) and (5), specific surface of a fraction is obtained as:

\[ \frac{A_i}{m_i} = \frac{K_A}{K_V \rho} \]

The value of the ratio \( K_A / (K_V \rho) \) is unknown, but can be assumed, it is a constant for all fractions. Total area of the analyzed sample can be calculated by summing areas of all fractions:

\[ A = \sum A_i \]

and quantity proportional to the total specific surface will be:

\[ S = \sum \frac{A_i}{m_i} \]

where \( x_i \) is the weight fraction of \( i \)-th fraction.

The characteristic dimension is obtained as mean sieve diameter:

\[ l_i = \bar{D}_i, \text{ where } \bar{D}_i = \frac{(D_{i+1} + D_i)}{2} \]

I.3 Calculation of mill characteristics

A mill characteristic is a quantity \( \Delta A/\Delta E \). Due to practical reasons, instead of the increase of total area \( \Delta A \) the increase of the specific surface quantity calculated from eq. (8) is used. The characteristics can be

a) integral
\[ \varepsilon_j' = \left( a_j' - a_0' \right) / \sum_{i=0}^{j} \Delta E_i \]  

(10)

b) differential

\[ \text{where index } j \text{ denotes value after } j\text{-th of milling experiment and index 0 is the material before milling. Quantity } \Delta E \text{ in the definitions is obtained as the amount of energy consumed by the mill.} \]

**I.4 Graphical representation of the size distribution**

Apart from the mill characteristic, the size distribution of the product is also of an interest, as it may be a criteria of suitability of the milling process used. Usually it is represented in a graphical from as a cumulative diagram, where weight fraction of the material above i-th sieve is plotted against sieve diameter:

\[ \sum_{i=1}^{k} x_i \text{ as a function of } D_{i+1} \text{ where } i = -1, 0, \ldots, k \]  

(12)

where \( k \) is number of sieves (including the bottom of the sieve column, where the smallest particles are), \( x_1 = 0 \) a  \( D_0 = 0 \). \( D_{k+1} \) is the maximum particle diameter in the material, which cannot be determined by the sieve analysis and has to be known independently. From the cumulative diameter it is easy to find out what is the fraction of particles smaller (or equal) then chosen size.

**II. Aims**

1. Determine dependence of the differential and integral mill characteristics on the milling time.
2. Graphically show the size distribution of the material before and after each milling.

**III. Apparatus description**

The mill (fig.1) is a stainless cylinder 1 with diameter and length 250 mm, which is closed by a cap 2 kept in place by a pair of screws. To reduce noise and dust immisions, the mill is inside a box 3, which can be opened from the top. A safety switch is below the lid, which will switch off the mill if the lid is opened. At the bottom a discharge chute 4 is located, to which a box with a mesh insert can be added. This is used to remove the material and balls from the mill. The mesh insert is used to facilitate separation of the material from the balls. The mill is rotated by a electrical drive motor 6 using V-belt transmissions 5.
The mill is operated using a panel where is a speedometer (gives the speed of the mill in min\(^{-1}\)), wattmeter (where immediate power input of the motor can be read). The speed of the mill is pre-set to a constant value.

In a mill, balls are used, which type and size is given by the task.

Sieve analysis is made using a vibrating pad with a set of sieves. A sample is taken using a quartering sampler – a circular pad with four discharges and a metal splitting cross.

**IV Work description**

**IV.1 Apparatus preparation**

Switch off the mill drive and open the lid of the mill. Manually rotate the mill to a position, where the mill cover is accessible. Loose the screws holding the cover and remove the cover. Carefully clean the mill and the discharge chute from old samples using a brush. Fill the mill with weighted amount of milling balls and weighted amount of sample material, which size distribution you have determined before by a sieve analysis (paragraph IV.3). Re-place the cover back on the mill fix it well in its position using the two screws. Switch on the mill while starting to measure the milling time.

**IV.2 Milling experiment**

Milling experiment is splitted into a sequence of milling periods of prescribed length, inbetween which the mill characteristics and particle size distribution are determined. During each milling it is necessary to repeatedly read (approx. 5 times) the power input of the mill drive.

At the end of each milling period switch off the mill drive and put the box including a metal mesh below the discharge chute. After the mill had stopped open the lid of protection box and remove the mill cover. Then pour the milled material including milling balls into the discharge. Carefully collect rests of the material all remains of the material from all surfaces which were in contact with the material. Separate the balls from the sample by lifting (and possibly shaking) the metal mesh in the box. Take a sample of the material and determine its particle size distribution. The rest of material can be kept either in the mill or the collecting box.
After completion of the experiment, put all sample into a box for milled material – do not mix it with the non-milled material.

IV.3 Sieve analysis

To achieve reliable particle size distribution, sampling of the material is crucial step. For solids, this is done using quartering: All material is putted on the quartering pad into a cone, which is spread flat reducing its thickness. Using a metal cross, split the layer into four equal parts. Remove two opposite quarters and mix the other two, creating a cone in the middle of the pad. The cone is spread again, split on quarters etc. until we obtain amount suitable for the sieve analysis.

Because the material is not mixed very well in the mill, it is necessary to use previous procedure for both the material before and after milling.

Place the sieves into the column, pay attention to place sieve with respect to the sieve size, which has to decrease constantly and check each sieve if its clean and without holes. About 100g of sample put on the topmost sieve, which has the bigger hole size. Place to sieve column on a shaking pad and fix it well. Switch on the shaking pad and let it running for given time. After this, remove the sieves and put the sample remaining on top of each sieve into cans and weight them with 0.1g precision. The minimum diameter of the particles at the bottom of the sieve column is assumed to be 0, the maximum particle size is given.

Analyzed sample can be returned into the mill as well as the rest of the sample and milling balls to continue with milling experiment.

V Safety instructions

1. The mill can be switched on only when the protection lid is closed, to protect surrounding from noise and dust and to prevent accidents due to rotating parts of the mill.
2. The drive should be switched on using the minimum frequency. Fast changing of the frequency damages the equipment, so adjusting the frequency should take about 10 seconds.
3. Before switching on the mill, make sure the cover is well fixed. It tends to get loose during milling spreading the content of the mill across whole lab.
4. When lifting the lid, it is absolutely necessary switch off the power first.
5. The sieves should be cleaned carefully using a brush to prevent damage of the sieve structure.

VI Evaluation of the experiment

1) Calculating the power consumed by the mill

The average power input $\bar{P}_j$ is calculated as arithmetical average of the measured values of the power input during whole milling period. The energy consumed by the mill during the period is calculated from relation:

$$\text{(13)}$$

where $\tau_j$ is the length of $j$-th milling period. Energy consumed since the beginning of milling is calculated as:
2) Calculation of milling characteristics

From the determined weights of the sieve fractions $m_i$ calculate their weight fractions using relation:

$$x_i = \frac{m_i}{m}$$

(15)

where $m_i$ is mass of the i-th fraction and $m$ is total mass of the analyzed sample. For each fraction determine ratio which is then used to calculate the specific surface:

$$\sum_{i=1}^{j} \Delta E_i$$

(14)

where $x_{ij}$ is weight fraction of i-th sieve fraction in j-th period of milling.

For each period of milling determine:

1. Integral characteristic, for total milling time since the start of the milling to taking the j-th sample (comparing a product of j-th milling with the feed) using equation (10).
2. Differential characteristic - $\varepsilon_j$ for given period of milling (comparing milling product with it’s state before the milling period) using equation (11).
3. Using method given in paragraph 1.4 plot a cumulative diagram of the size distribution (see example on fig.2). The graph has to start at point of origin, otherwise negative sizes would have to exist in the sample.

![Figure 2. Cumulative particle size distribution](image-url)
VII Symbols

\( a \) specific surface of material, given in eq. (6) \( \text{m}^{-1} \)
\( a' \) quantity proportional to the specific surface of material given by eq. (8) \( \text{m}^{-1} \)
\( D \) sieve hole diameter \( \text{m} \)
\( d \) inner diameter of the mill shell \( \text{m} \)
\( K_\lambda \) shape factor of the particle surface \( 1 \)
\( K_V \) shape factor of the particle volume \( 1 \)
\( l_i \) characteristic dimension of the particles \( \text{m} \)
\( \varepsilon \) differential milling characteristic \( \text{m}^1 \text{ W}^{-1} \)
\( \varepsilon' \) integral milling characteristic \( \text{m}^1 \text{ W}^{-1} \)
\( \omega \) angular velocity of the mill \( \text{s}^{-1} \)

Indexes lower
\( i \) sieve fraction (sieve)
\( j \) milling period
\( k \) milling ball