

Quantifying Mineral Liberation – A Conventional and New Automatic Sophisticated Techniques Approach

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Abstract

The characterization of textural properties of minerals is closely related to the process of their respective liberation. Measurements of mineral liberation, related to grinded ore, can be performed using optical ore microscope, by conventional, classical methods – point counting, linear intercepts method or planimetric measurements method (2D). Modern automatic devices and sophisticated measurement techniques (QEMSCAN/MLA) imply recording free surfaces area of mineral grains on polished sections samples in order to determine mineral degree of liberation. Value of mineral liberation obtained over free surfaces area can be of interest to flotation concentration, although not for gravity separation or, for example, magnetic separation. The prediction accuracy for behavior of one feed ore during the concentration process depends on the method of measuring/recording mineral liberation. Considering raw materials with complex textural characteristics it is crucial which method will be applied for determination of mineral liberation respecting whether for concentration process is crucial physical or chemical method.

Keywords: mineral liberation, free surface area, texture characterization, prediction, ore microscopy

Introduction

The calculation of the mineral liberation degree of the given mineral is made on representative samples of a classified grinded ore, on polished surfaces of mineral grain cross-sections (2D) applying the ore microscopy. Measurements are performed using point counting (Glagolev-Chayes method), linear intercepts method (The Rosiwal-Schand method) or planimetric measurements method (De Lesse method). After measurements have been performed, so called Gaudin correction coefficient- the stereological error correction is added. Based on the measurments of the liberation degree of the mineral of interest, it is possible to predict the behaviour of the raw material during the concentration and predict the quality of the future concentrate, i.e. the loss causes of the useful mineral in the tailings. Nowadays, due to current devices and sophisticated techniques of automatic characterization of minerals (QEMSCAN/MLA) some reseachers (Al Cropp, 2013.) determine the calculation of mineral liberation on the basis of the size of free surfaces area of the mineral o interest. Namely, the total mineral surface area whereby a flotation reagent can make a direct contact is determined. This "calculation of liberation" (obtained over the size of free surface area of the mineral of interest particles) can point to possible (high) recovery, but at the same time to the very low quality of the concentrate. Figure 1 shows the concept according to which the categories of calculation of mineral liberation, measured over the size of free surface areas, can be compared with conventional understanding of the mineral liberation (modified Cropp, 2013).

According to conventional, widely accepted definitions of liberation of the given mineral in the ground ore, the raw material grains/particles cannot be liberated partially, i.e. more or less, in a very wide range (from a few percents to almost completely free). Multiphase grain/particle is always non-liberated, middling particle, which can be classified as intermediate product and sent to regrinding - additional liberation. Therefore, the intergrown grains cannot be further classified into classes according to the liberation degree of the mineral of interest. They can be categorized into groups according to the size of the free surface areas, regarding the external contacts over which the mineral grains can interact with the reagents for concentration. For the category of intergrown grains, only the percentage (volume, mass) average-middle share of the measured mineral in the intergrowths can be expressed, for all the measured intergrown grains, or these data can be classified into certain group intervals (interval of 10%). The figure 1, showing example of simulated particles containing ore mineral grains classified by both - degree of liberation and the free surface area.

Methods of measurement

Mineral liberation determination. The distribution of linear intercept lengths on polished sections of the feed ore samples gives a very useful characterization of the mineralogical texture such as grain and crystal aggregate sizes and shape, specific surface areas, mineral-mineral association, surface coatings, proximity index, contiguity index, degree of liberation etc.

Calculation of the contiguity index. In order to enable the successful concentration of useful minerals from the raw material, it is necessary, before crushing, to determine a number of characteristics of associated minerals related to non grinded, raw ore, such as crystal size, shape, type of intergrowth, frequency and the complexity of contact surfaces of some mineral pairs and other structural characteristics like their distribution, etc.

	Free and intergrowth simulated particles classified by free surface area (external free contacts) (u %)	Locking characteristics of particles with ore and gangue Degree mineral liberation of particles classified at 100%, 75%, 50% and 25%			
The mineral liberation of particles classified by free surface area , %		100%	75%	50%	25%
	100% Free surface area of the mineralized particles (free external surface area)				
	75% Free surface area of the mineralized particles (free external surface area)				
	50% Free surface area of the mineralized particles (free external surface area)				
	25% Free surface area of the mineralized particles (free external surface area)				
	0% Free surface area of the mineralized particles (free external surface area)				

Fig. 1. The degree of mineral liberation and mineralized particles liberation by free surface area (free external surface area) – textural classifications, are a key driver in all mineral separation processis (modified after Al Cropp, 2013). Legend: marked particles, all 11, are intergrowth, not free. Conventional degree of liberation is 14.81%. At the same time the liberation of minerals expressed as a free surface area is 85.18%

Rys. 1. Stopień uwolnienia minerałów oraz ziaren zmineralizowanych za pomocą oceny zewnętrznej powierzchni wolnej – klasyfikacje tekstur są kluczowe we wszystkich procesach przeróbki mineralnej (poprawione wg AlCropp, 2013). Legenda: zaznaczone ziarna, których jest 11, są zrostami. Tradycyjny stopień uwolnienia wynosi 14,81%. Uwolnienie minerałów wyrażone jako pole powierzchni wolnej wynosi 85,18%

Textural characterization of ore-contained minerals can be performed and represented by descriptive (Amstutz, 1960; Craig and Vaughan, 1994; Amstutz and Giger, 1972), and accurate numerical data which are commonly more convenient for operating engineers. For a mineral liberation prediction based on microscopy of the textural-structural properties of the ore, one must determine: first, the mode and degree of mineral intergrowth; secondly, the minerals which intergrow with mineral of interest in the technological process, when the ore contains deleterious components.

Associations of the selected pairs of minerals may be expressed by the "contiguity index" or, as termed by some authors (Gurland, 1958), "intergrowth, locking or proximity index" witch is approximately the same as the connectivity (Amstutz and Giger; 1972). Where ground ore is concerned, this index is closely related to the free surface area of the selected mineral and its relationship with other associated minerals. The contiguity index relates the given grain surface area of the selected mineral and its total surface. This parameter is useful for parent ore, as the input processing material, to predict the mineral liberation, and even more for the analysis of liberation from comminuted ore to characterize the intergrown grains.

The coordination number or coordination index (Jeulin, 1981), has been extensively used in textural characterizations of various rocks (Amstutz and Giger, 1972) and their classifications. Thus the coordination number between Ai and Ak phases is given by the relation:

$$\mathbf{K}_{\left(\mathbf{A}_{i},\mathbf{A}_{k}\right)}=\frac{\mathbf{N}_{\left(\mathbf{A}_{i},\mathbf{A}_{k}\right)}\cdot\mathbf{N}}{\mathbf{N}_{\left(\mathbf{A}_{i}\right)}\cdot\mathbf{N}_{\left(\mathbf{A}_{i}\right)}}$$

where:

$$\begin{split} N_{(Ai,Ak)} &- number \ of \ contacts \ between \ A_i \ and \ A_{k,} \\ N &- total \ number \ of \ investigated \ grains, \\ N_{(Ai)} &- number \ of \ grains \ A_{k,} \\ N_{(Ak)} &- number \ of \ grains \ A_{k}. \end{split}$$

Contiguity index of mineral A to mineral B can be written (Gurland, 1958; Jones and Barbery, 1975) as:

$$\mathbf{V}_{\mathbf{A}\!/\!\mathbf{B}} = \mathbf{S}_{\mathbf{A},\mathbf{B}} \big/ \mathbf{S}_{\mathbf{A}} = \mathbf{S}_{\mathbf{V}_{\!(\mathbf{A},\mathbf{B})}} \big/ \mathbf{S}_{\mathbf{V}_{\!(\mathbf{A})}}$$

or

$$\mathbf{P}_{\mathbf{A}/\mathbf{B}} = \mathbf{S}_{\mathbf{A}/\mathbf{B}} \cdot 100 / \mathbf{S}_{\mathbf{A}}$$

where

 $V_{A/B}$ – is the proximity index,

 $S_{(A,B)}$ – is the surface area of A in contact with B,

 S_A – is the total surface area of mineral A;

 $S_{V(A,B)}$ – is the specific surface area of A in contact with B (i.e. the contact area per unit volume of A),

 $S_{V(A)}$ – is the specific surface area of A, and

 $P_{A/B}$ – proximity index of minerals A and B (%).

The specific surface area of mineral is calculated from the relation (Jones and Barbery, 1975):

$$S_{V(A)} = S_{(A)}/V_{(A)} = 4/\overline{L}_{(A)}$$

where

 $S_{V(A)}$ – specific surface area of mineral A,

 $S_{(A)}$ – total surface of mineral A,

$$V_{(A)}$$
 – volume of A, and

 $\overline{L}_{(A)}$ – mean intercept length on mineral A.



Fig. 2. Illustration of contiguity index measurement on a simulated surface area of a polished section of an raw ore (Amstutz and Giger; 1972). Legend: Polished sections of on multiphasic feed ore samples; Linear Rosiwal-Schand method of measurement; The target mineral is red phase A; The set of parallel test lines across each grain on the surface of polished section I – V; Polimineral raw ore with 5 tipe of minerals, from A to F phase; Phase A – the target, mineral of interest Rys. 2. Ilustracja pomiaru wskaźnika przyległości wykonanego na symulowanym polu powierzchni wypolerowanego przekroju rudy (Amstutz i Giger, 1972). Legenda: wypolerowane przekroje próbek wielofazowej nadawy rudy; liniowa metoda pomiaru Rosiwala-Schanda; minerały celowe są czerwoną fazą A; zbiór równoległych linii testowych wzdłuż każdego ziarna na powierzchni wypolerowanego przekroju I – V; wielomineralna ruda z pięcioma typami minerałów, od fazy A do F; Faza A – cel, czyli minerał będący przedmiotem zainteresowania

Along a set of parallel lines across the surface of an ore polished section, volume percentages of minerals were measured and their textures characterized, and the number of contact points, both internal and external, on the set lines was registered. The number of transitions from one phase to another, or one mineral into another, was registered with the purpose of defining mineral association across the contact areas of mineral grains in the analyzed material (Fig. 2).

Sizes of contact areas of this association mineral pairs, as an important textural characteristic, are expressed by the contiguity index. A statistical processing of results (density of contact points) was used to calculate areas of direct contacts between mineral pairs in the analyzed ore. The calculated contiguity indices suggest the behaviour of the ore in crushing and grinding, the behaviour of each mineral during its liberation, and the effects of processing on the concentrate.

When the contiguity index among certain minerals is very low, due to the absence of the direct contact (genetic relationship) of the two given minerals, then the intergrown grains of these two minerals cannot be expected in the concentrate of the basic mineral as undesirable.

Mineral liberation - prediction

The known value of the contiguity index for the given mineral can be used to deduce other ore characteristics. The empirical expression relating the degree of liberation for the given (selected) mineral phase (α) and the specific surface area (Steiner, 1975) is the following:

$$\mathbf{L}_{\pmb{\alpha}}(\mathbf{D}) = 1 - \mathbf{S}_{\mathbf{V}_{\pmb{\alpha}}}^{(i)}(\mathbf{D}) \Big/ \mathbf{S}_{\mathbf{V}_{\pmb{\alpha}}}^{(e)}(\mathbf{D}) \qquad \quad \mathbf{S}_{\mathbf{V}_{\pmb{\alpha}}}^{(e)}(\mathbf{D}) > \mathbf{S}_{\mathbf{V}_{\pmb{\alpha}}}^{(i)}(\mathbf{D})$$

where:

 $L_{\alpha}(D)$ – is the proportion of α that is liberated at particle size (D),

 $s_{\mathbf{v}}^{\emptyset}(\mathbf{D})$ – is the interfacial α/β area per unit volume of α , for particles of size (D),

 $\mathbf{S}_{v_{a}}^{(r)}(\mathbf{D})$ – is the external surface area of a per unit volume of a, for particles of size (D).

Most of these (free) areas in the above relation can be estimated from either linear or planimetric measurements as mentioned earlier. It may be used to deduce the liberation by stereologic method following the expression (Steiner, 1975):

$$\mathbf{L}_{\alpha}(\mathbf{D}) = 1 - \mathbf{B}_{\alpha}^{(i)}(\mathbf{D}) / \mathbf{B}_{\alpha}^{(e)}(\mathbf{D}) = 1 - \mathbf{I}_{\alpha}^{(i)}(\mathbf{D}) / \mathbf{I}_{\alpha}^{(e)}(\mathbf{D})$$

where:

 $B_{\alpha}^{(i)}(D)$ – is the boundary length of α/β intefaces measured on sections through particles of size (D),

 $B_{\alpha}^{(e)}(D)$ – is the boundary length of α /matrix interfaces measured on sections through particles of size (D),

 $I_{\alpha}^{(i)}(D)$ – is the number of intersections of a test line with α/β interfaces, for particles of size (D),

 $I_{\alpha}^{(e)}(D)$ - is the number of intersections of a test line with α / matrix interfaces, for particles of size (D).

Classical mineral liberation can bee expressed by two different parameters: weight ratio and exposure ratio. In single particles, the former indicates the weight proportion of one mineral with regard to the total particle weight, while the latter quantifies the proportion of exposed perimeter occupied by this mineral (Perez-Barnuevo et al, 2012). The degree of mineral liberation and mineralized particles liberation by free surface area (free external surface area) – textural classifications, are a key driver in all mineral separation processis (Reyes et al, 2018).

A prediction of mineral liberation in ore grinding is possible on the basis of the identified distribution law (distribution of linear intercepts, and use of Gauss-Laplace probability function) and modeling the mineral texture (Tomanec and Milovanović, 1994).

Frequency distribution, classified intercept lengths and sample means provided, based on the identified lognormal distribution and the use of Gauss-Laplace integral probability function can be used the following:

$$F(d) = \left[exp\left\{ -(logd - log\overline{d})^2 / 2(log\sigma)^2 \right\} \right] / \left\{ log\sigma \cdot (2\pi)^{(1/2)} \right\}$$

for the prediction of particle size to which ore should be ground for the desired mineral liberation (Tomanec and Milovanović 1994a).

Where is:

F(d) – normal distribution (probability) function,

d – grain size diameter and \overline{d} – geometric mean intercept,(d = l); σ – standard deviation.

Results and discussion

In the past ten and more years, the mineral liberation has been expressed in two ways. The first, based on the volumetric/mass distribution of one mineral in free grains in relation to its total presence, in free and intergrown grains in the ore. The second way, adjusted to contemporary devices and sophisticated automatic measurement methods, based on the size of the free surface area, namely the external boundary zone of mineral particle contacts which can result in the contact of concentration reagents and the given mineral. In the 2D space, that is the grain perimeter, the size of the free peripheral line on the product surface. In real 3D conditions, all such grains are intergrowths, and they are by no means really free grains. Certainly, both methods of measurement contribute to the general characterization of mineral grains and indicate, i.e. provide a better evaluation of the mineral grains behaviour during their concentration, especially flotation.

The mass distribution of the measured free grain mineral in the sample is obtained by multiplying the results with the density of the given mineral, while the size of the measured perimeter (free edges) on the intergrown grains is obtained by putting in relation to the total free surface of the given mineral measured in the 2D level.Both perimeters can be calculated using Barbier's formula (Perez-Barnuevo et al, 2012).

Conclusions

We should bear in mind that once obtained results of calculation of liberation of the given mineral must be interpreted adequately depending on the device or the recording method. The final values, as well as the integral mineral liberation, will not be comparable if the measurement is carried out by different methods.

At the same time, the prediction of the grinding fineness, the concentrate quality, the recovery, possible losses of useful minerals in the tailings will differ significantly depending on the method of liberation recording.

This paper, among other things, should serve to clarify how important the mineral textural characteristics of the preparation processes involved are, and how important it is to understand the relation of the mineral liberation and the size of the free surface areas of the mineral in the ground raw material.

The liberation and free surface areas of minerals are crucial for concentration processes and must be a high priority for engineers engaged in dressing processes.

Literatura - References

- 1. AMSTUTZ, Gerhardt Christian. A geometric classification of basic intergrowth patterns of minerals. Geotimes, vol. 5, 1960. p. 24.
- 2. AMSTUTZ, Gerhardt Christian, GIGER, Hans. Stereological methods applied to mineralogy, petrology, mineral deposits and ceramics. Journal of Microscopy, vol. 95, 1972, p. 145-164.
- 3. CRAIG James; VAUGHAN David. Ore Microscopy and Ore petrography. 2nd edition. New York : John Wiley & Sons Inc, 1994. p. 434, ISBN 0-471-55175-9.
- 4. CROPP, Al. Liberation And Free Surface Area In The Float Feed. Minassist, [online]. Accessed 11.9.2018. Available at: http://www.minassist.com.au/blog/liberation-and-free-surface-area-in-the-float-feed/ .
- 5. GURLAND, John: The measurement of grain contiguity in two-phase alloys. Trans. Metallurgical Society of AIME, Vol. 212, 1958, p.452-455.
- 6. JEULIN Dominique. Mathematical morphology and multiphase materials. 3rd European Symposium on Stereology, Ljubljana, 1981. p. 265–86.
- JONES, M.P., BARBERY, Gilles. The size distribution and shapes of mineral in multiphase materials: particle determination and use in mineral process design and control. XIth International Mineral Processing Congress, Cagliari, 1975, paper 36.
- 8. PEREZ-BARNUEVO Laura, PIRARD Eric, CASTROVIEJO Ricardo. Textural Descriptors for Multiphasic ore Particles. – Image Anal Stereol 31, 2012, p.175-184.
- REYES Francisco, CILLIERS Jan., NEETHLING Steven. Quantifying mineral liberation by grade and surface exposure using X-ray micro-tomography for flotation processes, 29th International Mineral Processing Congress, 2018, Moscow – Russia, 2019, p. 3985-3994.
- 10. SPENCER, Stiven and SUTHERLAND, David. Stereological correction of mineral liberation grade distributions estimated by single sectioning of particles. Image Anal Stereol, 19, 2000, p. 175-182.
- 11. STEINER H J. Liberation, kinetics in grinding operations. XIth International mineral processing congress, Caligari, Ente Minerario Sardo, 1975, p. 35-58.
- 12. TOMANEC, Rudolf. Degree of Mineral Liberation Depending on Useful Component Concentration and Fineness of Grind, Faculty of Mining and Geology, University of Belgrade. Doctoral thesis, 1989.
- TOMANEC Rudolf, MILOVANOVIĆ Jelica. Mineral liberation and energy saving strategies in mineral processing. Physicochemical Problems of Mineral Processing Journal, vol 28, and XXXI Polish Mineral Processing Symposium, Wroclav - Poland, 1994, p. 195-205.
- 14. TOMANEC Rudolf, MILOVANOVIĆ Jelica. 1994a: Mineral liberation prediction based on texture characterization, 5th International Mineral Processing Symposium, Cappadocia Turkey, 1994a, p 3-9.

Ocena uwolnienia minerałów – podejście konwencjonalne i nowe techniki automatyczne

Charakterystyka właściwości tekstury minerałów jest blisko związana z procesem ich uwolnienia. Pomiary uwolnienia minerałów powiązane są z mieleniem rudy i mogą być wykonane za pomocą mikroskopu optycznego przy zastosowaniu konwencjonalnych metod – liczenia punktów, metody linii przecięcia albo metody pomiarów planimetrycznych (2D). Nowoczesne urządzenia automatyczne, jak również wyrafinowane techniki pomiarowe (QEMSCAN/MLA) stosują pomiar pól powierzchni wolnych ziaren minerału na próbkach wypolerowanych przekrojów w celu określenia stopnia uwolnienia minerałów. Wartość tego uwolnienia otrzymana na podstawie pola powierzchni wolnej może być przedmiotem zainteresowania w kontekście prowadzenia procesu flotacji, aczkolwiek nie w przypadku wzbogacania grawitacyjnego, czy magnetycznego. Prawidłowość prognozy odnośnie zachowania rudy podczas procesu zależy od metody oceny uwolnienia minerałów. Biorąc pod uwagę surowce o skomplikowanej teksturze bardzo ważnym jest, którą metodę zastosuje się w celu określenia stopnia uwolnienia minerałów pamiętając także o tym, czy dany proces jest oparty o metody fizyczne, czy też chemiczne.

Słowa kluczowe: uwolnienie minerałów, pole powierzchni wolnej, charakterystyka tekstury, prognoza, mikroskopia rudy