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Size distribution measurement and the Humphries' micrometer eyepiece

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(1 Fig, 1 Photo)

Meranie zrnitostnej distribúcie a aplikácie Humphriesovho mikrometrického okulára

Úvodom práca charakterizuje problematiku zrnitostnej analýzy a niektoré možné definície pojmu „veľkosť zrna" spolu s opisom niektorých semimanuálnych optických prístrojov na zrnitostnú analýzu.

Opis a charakteristika Humphriesovho mikrometrického okulára tvorí hlavnú časť článku. Je to prístroj na semiautomatickú mikroskopickú zrnitostnú analýzu na výbrusoch, nábrusoch alebo voľných zŕn. Skladá sa (obr. 1) z vlastného mikrometrického okulára (1), kde sú uložené 2 pohyblivé vertikálne rysky na meranie priemeru zrna (najčastejšie Feretovho) a jedna horizontálna referenčná ryska; nastavovacieho kotúča (2), ktorý je spriahnutý s ryskami a ktorý kóduje veľkosť zŕn po nastavení veľkosti zrna (jeho točením); spínacieho tlačidla (3), po stlačení ktorého registračná jednotka (6) zaradí veľkosť zrna do jedného zo 16 zrnitostných tried usporiadaných do radu s koeficientom $\sqrt[4]{2}$. Skutočná veľkosť častíc je závislá od zväčšenia objektívu a dĺžky tubusu mikroskopu. Nastavením pohyblivej časti tubusu možno koincidovať opísanú veľkostnú škálu s Krumbeinovou ‛fí' škálou, ktorej vlastnosti sú v článku opísané. Prístroj možno kalibrovať pre každý objektív pomocou mikrometrického stolčeka. Za 1 hodinu možno analyzovať až 1000 zŕn. Veľkou výhodou prístroja je jednoduchosť a cenová prístupnosť.

Prístroj je pripojiteľný na všetky druhy mikroskopov (monokulárne alebo binokulárne) a meranie možno robiť za každého druhu osvetlenia. Záverom sú podané príklady aplikačných možností zariadenia.

Introduction

The measurement of the distribution of particle sizes is a familiar and important aspect of the analysis of a wide variety of natural and manufactured products.

Sieving, sedimentation and elutriation are well known methods for particle size analysis of disaggregated or ‛loose' material, but clearly they are not applicable to continuous ‛networks' of grains or cells as in a rock, a metal, or plant and animal tissue, with or without gas or fluid inclusions, pore spaces or matrix. For such materials it is necessary to measure each grain individually. The acquisition of data with a microscope fitted with a graticule is extremely slow and it is not surprising that such measurements are avoided whenever possible. Several attempts have been made to increase the rate of microscope data collection by partial automation and a review of some of these instruments has been given elsewhere (D. W. HUMPHRIES, 1969).

In recent years, fully automatic image analysis devices have been developed which will carry out particle sizing operations at high speed. Unfortunately, the very high cost of these instruments has tended to restrict their wide application.

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Initial Design Considerations

In determining particle size distribution, it is common practice to group the individual size measurements into classes to permit the subsequent use of statistical measures. Therefore there appears to be little point in measuring each grain to a high degree of accuracy provided it can be placed into its correct size class.

In modern studies of sediments it is usual to define these classes in terms of a geometric scale rather than a linear one and to use the ‘phi (φ) scale‘ of W. C. KRUMBEIN (1934, 1938). In this scale the limits of each class increase or decrease by a factor of $\sqrt{2}$. For greater precision each class can be sub-divided into 1/4 φ units with a factor of $\sqrt[4]{2}$. The φ-scale itself is a simple logarithmic conversion of the standard scale of sizes used for sieves. Thus $\varphi = -\log_2 \zeta$, where ζ is the size in millimeters. Part of the φ-scale, together with the 1/4 φ-scale for sieves of the ASA and BSS range as follows:

Sieve size (mesh)	8	10	12	14	16	18	22
Size in mm	2.00	1.68	1.41	1.19	1.00	0.840	0.707
φ-scale	—1.00				0.00		
$\frac{1}{4}$ φ-scale	—1.00	—0.75	—0.50	—0.25	0.00	0.25	0.50

An advantage of this scale is that class size limits are a simple cycle of numbers, each differing from the next by a value of 0.25 units. The negative sign in the definition makes the φ values of the smaller particles (that is, the ones most commonly measured by sieves or microscope) positive.

This scale was adopted in designing the micrometer eyepiece and is preferred, but it will be shown later that althoug the size classes will be related by a factor of $\sqrt[4]{2}$ on the millimeter scale (or 1/4 φ units of the Krumbein scale), their absolute values need not be those of the standard sieve sizes. It will also be shown that the factors controlling the absolute sizes of the classes depend on the exact magnification of the microscope objective and on the tube length of the microscope.

The second basic design factor concerns the particular parameter of a grain that it is desired to measure. It is common practice to express the size of a particle in terns of a single linear measurement. If the particle is sectioned as in a thin section or a polished surface, the dimensions of the section measured will not necessarily have any direct relationship to the dimensions of the grain from which the section was cut. For this reason, a number of ‘statistical parameters‘ of grain size have been introduced, but their precise relationship to the ‘true‘ size is still not fully defined.

The micrometer eyepiece has been designed to permit the measurement of several (linear) parameters. The various measures are summarised below.

Grain size parameters

(a) *Direct linear measures* for use with ‘loose‘ grains

1. *Long axis*: the maximum overall length of a grain independent of its orientation.
2. *Intermediate axis*: the maximum dimension of a grain in a direction perpendicular to the long axis.
3. *Short axis*: the maximum dimension of the grain in a direction perpendicular to the plane containing the long and intermediate axes.

It is usually not possible to measure all three axial dimensions simultaneously. Suitable techniques for handling small particles have been described by C. W. H. HULBE (1955) and D. W. HUMPHRIES (1966)

(b) *Statistical linear measures* for use with sectioned grains (Fig. 1)

1. *Martin's statistical diameter* (G. MARTIN, et al. 1928).
2. *Feret's statistical diameter* (I. R. FERET 1931).
3. The *maximum horizontal intercept* (W. C. KRUMBEIN, 1935).
4. The *horizontal intercept*
5. The *apparent long axis* (e. g. M. A. ROSENFELD 1953).

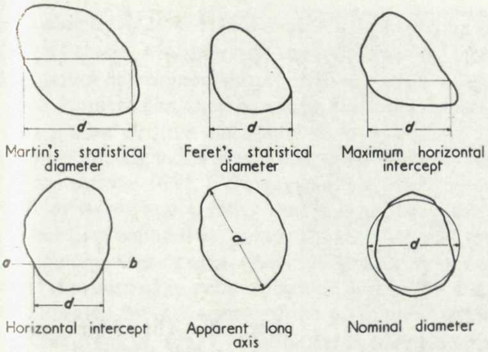

Fig. 1. Statistical measures for grain size description. d — grain diameter, $\overline{ab}$ is the reference line on which the horizontal intercept is measured.

Obr. 1. Štatistické deskriptory zrnitosti. *d* — priemer zrna, $\overline{ab}$ je referenčná priamka pozdĺž ktorej sa robí meranie horizontálneho priesečníka.

Of these measures, Feret's statistical diameter is probably the easiest to determine and is very commonly used. It is said by H. HEYWOOD (1946) to deviate from the nominal section diameter (see below) by about 12 %.

(c) *Measure based or the projected area of the grain.*

1. The *nominal sectional diameter* is the diameter of a circle of the same area as the grain projection. It has been used in the Zeiss- Endter particle size analyser (F. ENDTER and H. GEBAUER,, 1956) and by A. HÖRSTEN (1960).

It has been noted already that the relationship between a size distribution of sectioned grains and the 'true' size distribution of the grains is not yet a completely solved problem. Some progress has, however, been made with the size distribution of sections of spheres and ellipsoids (see E. E. UNDERWOOD, 1970, for discussion).

The Semi-Automatic Micrometer Eyepiece

The instrument is housed in a small light-weight aluminium box which can be attached to the eyepice tube of the microscope by means of a short tube and clamp (Fig. 2). The box, which is fitted with its own × 10 Kellner eyepiece, can be placed to the left or the right of the microscope according to which eye the observer chooses to use. It can also be attached to a binocular microscope when a short length of tube to carry the second eyepiece can be placed in the eyepiece tube not occupied by the micrometer.

Two parallel vertical reference lines in the eyepiece micrometer are used to measure the grain image. A horizontal line is also present. The spacing of the vertical lines can be varied by rotation of the handwheel on the top of the instrument case. The linkage between the reference lines and the handwheel consists of light-weight frames to which are attached followers running on the edges of two specially designed cams.

The cam is so designed that 20° of rotation of the handwheel brings about a shift in the reference lines equivalent to 1/4 φ units in the image plane of the microscope. The total movement is limitede to 320° by stops. The range of 320° is divided into 16 classes. A rod with a push-button passes through the centre of the handwheel and carries at its lower end an arm to which is attached a knife edge and an electrical contact. Pressure on the spring-loaded push-button guides the contact on the arm onto one of sixteen contacts arranged outside the crown wheel. Each of these contacts is connected with an electromechanical counter in the counter bank. A seventeenth counter sums the total counts over the whole bank.

Should the size of a grain being measured coincide exactly with a 1/4 φ unit, the knife edge on the contact arm throws the arm at random to one side or the other of the knife-edged teeth of the crown wheel.

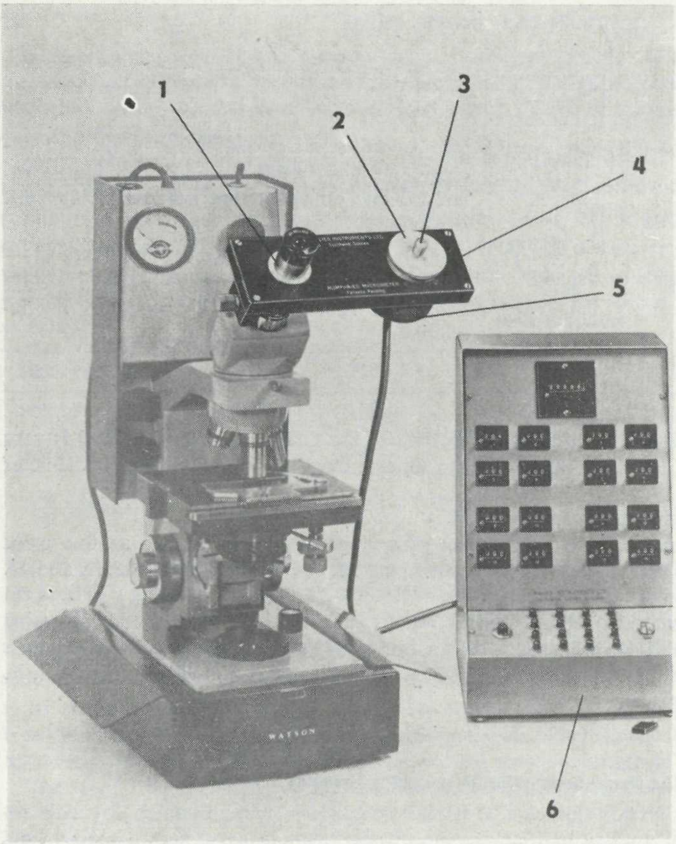

Fig. 2. The Humphries' Micrometer Eyepiece.
1 — The eyepiece and the hairlines (not visible).
2 — The sizing hand wheel.
3 — The contact (start) button.
4 — Instrument housing.
5 — The sizing cam (housed).
6 — The counter bank.

Obr. 2. Humphriesov mikrometrický okulár
1 — Okulár s pohyblivými ryskami
2 — Kotúč na nastavovanie rysiek, tj. veľkosti zrna
3 — Registračné tlačítko
4—Teleso mikrometrického okuláru.
5 — Triediaca jednotka
6 — Počítacia jednotka

Setting up and Range fo Size Measurement

The micrometer eyepiece is inserted into the eyepiece tube of the microscope and lightly clamped into position. The microscope is then focussed on a stage micrometer with the reference lines at their maximum spacing. With many objectives it is found that the reference line spacing is close to an exact $^{1}/_{4}$ φ unit and can be made precisely so by slackening the clamping screw and raising or lowering the eyepiece micrometer in the eyetube of the microscope. It will be necessary to refocus the microscope throughout this operation. When coicidence is obtained the micrometer is reclamped and this adjustment is not disturbed while the particular objective is in use. It will be noted that the adjustment is thus made by varying the tubelength of the microscope. With microscopes fitted with a rack and pinion to adjust the tube length great care must ge taken that this adjustent is not disturbed once the micrometer is set up. In use, the side of grains or the section is traversed across the field of view using the mechanical stage and as each grain comes to the centre of the field the reference lines are adjusted until they just touch the image of the grain at te position for the particular parameter being measured (Feret's diameter is the easiest to use). The contact button is pressed and the grain is automatically counted in the appropriate size class. It has been found that objectives of nominally the same magnification by the same maker vary considerably in true magnification. Thus it may be possible to set the scale to exact $1/4$ φ units in cases of difficulty by changing the objective. However, in the event of this not being possible, a new scale can be calculated by setting up as before, noting the stage micrometer dimension at the maximum opening of the reference iines and taking this value as the initial value of the scale. The complete scale is calculated by dividing this value by $\sqrt[4]{2}$(= 1.189) to give the lower limit of the first (coarsest) class and repeating this over sixteen classes. Alternatively, the phi value can be calculated from the identity given earlier and the class limits determined simply by adding the value of 0.25 φ units successively.

The eyepiece can thus be set up for any objective of any magnification either on the exact scale of $^1/_4$ φ units or on a calculated scale. With a × 95 objective the lower limit of the scale of sizes is approximately 0.004 mm. The overall range of size that can be measured with any single objective is in the ratio of 16:1. If the range of particles present in any preparation exceeds this limit, then the particles larger and smaller than the limits of the micrometer can be counted manually if few in number (i. e. counted as 'greater than' or 'smaller than') or included in the limiting classes which then must be regarded as open ended. At present is it not possible to change objective during an analysis and if it is desired to count larger or smaller particles by changing the objective, this objective must be calibrated by movement of the micrometer and a new analysis commenced. It has been found, however, that in many routine analyses on particular materials, once a suitable objective has been chosen, it is rarely necessary to change it.

Accuracy of Measurement

The accuracy of the instrument depends essentially on the profile of the cam and the position of the crown wheel teeth. The maximum error for any division between classes at the coarser end of the scale is less than 1 % of the true size at that position and less than 2 % at the fine end.

The Counter Bank

The seventeen connters are set up in a 4 × 4 bank with the totalising counter above them (Fig. 2). All counters can be reset to zero after each analysis. Each of the counters has its own push-button by which it can be operated independently of the eyepiece for simple counting of phases or attributes such as color or shape. The 4 × 4 arrangement allows simultaneous counting of two attributes such as roundness and sphericity over a limited number of classes.

Counting Rate and Grain Count

The measurement of the long axis involves the rotation of the microscope stage and it should be noted here that the eyepiece micrometer should not be rotated in the eyepiece tube since this will upset the setting of instrument as well as cause wear to the tube. About *400 grains per hour* can be measured without undue fatigue. It is however, much quicker and simpler to count grains using the statistical measures and about *1000 grains per hour* can be counted using Feret's diameter. The counting rate is measurably reduced if large areas of empty slide have to be traversed. Not surprisingly, counting grains in continuous networks is quicker than counting loose grains. The counting rate using an ordinary eyepiece scale, pen and paper and allowing time for classification is rarely more than 100 grains per hour, and often falls to 30 grains or less per hour after a prolonged spell of measuring.

The number of particles to be classified is determined by the purpose of the analysis and the accuracy required. M. A. ROSENFELD (1953) quoting W. J. DIXON and F. J. MASSEY (1951) suggested that the minimum number of grains would be that which satisfies specified confidence limits of the mean and this topics is discussed by E. E. UNDERWOOD (1970) However, it appears that most authors regard a count of 400-500 grains per analysis to be adequate.

Concluding Remarks

The micrometer eyepiece greatly increases the rate of counting particles for grain-size distribution determinations compared to fixed graticule methods. It enables the operator to retain full control over the analysis, a feature of considerable importance where it is necessary to size a single component in a multi-component system where differentiation of components may require the full resources of the polarising microscope. It also enables a check to be made on the performance of fully automatic image devices and to select materials suited to their high-speed capabilities. Furthermore, the comparatively low cost of the instrument puts an efficient means of particle size analysis within reach of many laboratories which hitherto have rejected microscopic methods as too slow and too tedious yet were prevented by financial considerations from entering the field of fully automatic image analysis or considered that these instruments were incapable of solving their particular problem.

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