Particle Size of Coke Filler Fines

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Introduction

Particle size is critical wherever there is a particle packing problem. In graphite, maximum density is obtained by using a spectrum of particle distributions, because graphite may shrink during processing but not deform. Surface area is important because it may affect the amount of pitch needed to obtain good wetting and maximum strength. Grain anisotropy ratios are critical because they affect such properties as thermal expansion and other parameters like thermal conductivity and ablation rates. The method described below may also be used for correlating dimensional changes with particle size changes that occur during calcining.

Experimental Methods

Optical stereology was used for analyzing particle size in the range 0.5 μ m to 50 mm. For this purpose a powder mount was produced using epoxy as the dispersion medium. Photomicrographs were made with magnifications at 200X, 400X, or 4000X. These were utilized to determine (a) the diameter of a circle encompassing the maximum particle dimension d_{max} = ℓ , the diameter of a circle equivalent in area to the projected area d_a = w; and (b) the diameter of a circle equivalent to the minimum particle diameter d_{min} = t.

Surface area and surface volume could be obtained from the laws of geometry. Volume and surface coefficients could be calculated by the method described by Heywood¹, Stockham and Fochtman², and Herndan³. Sphericity and roundness have been described by Wadell⁴. For analytical purposes, particle sizes were separated into 48 intervals. With the methods described by Stockham², this information could be used to calculate the arithmetic mean d_{av} , the surface mean d_{s} , the volume mean d_v , the volume surface mean d_{vs} , and the weight mean diameter d_w .

If the data represent a true Gaussian distribution, a linear plot on log-probability paper as illustrated in Fig. 1 can be obtained. The geometric mean $d_{\rm g}$ is read directly from the graph.

Maxima and minima were determined. A maximum was equivalent to the length of the particle. A minimum was taken as the smallest breadth observed. When more than one minimum mode occurred, they were taken as representing a thickness and a breadth. The geometric standard deviation of any population $\sigma_{\rm d}$

is the value obtained at 84.1 percent (Fig. 1, divided by the value obtained with 50 percent of the particles, i.e., 35/16 = 2.19 for the mean standard deviation. For -1σ , the value is calculated from the value obtained at 50 percent divided by the value obtained at 16 percent i.e., 16/11 = 1.45. Anisotropy = 29.8/9.5 = 2.84 at 50 percent.



Fig. 1 Combined Evaluation of Coke Particles

Shape factors were calculated from $d_{max} = l$, $d_{area} = w$, and $d_{min} = t$; k = the volume coefficient $= \pi_v^3$; f = surface coefficient $= \pi d_s^2$; m = breadth/thickness = w/t; the flatness ratio n = l/w = length /breadth, i.e., the elongation ratio. A shape coefficient C can be calculated by the method described by Stockham and Fochtman² and Herndan³. Sphericity ψ is given by $(d_v/d_s)^2$ or may be estimated from the projected area diameter and the maximum dimension by a method described by Wadell⁴.

<u>Results</u>

Analysis of Coke

Properties calculated for coke are shown in Fig. 1. Two batches were sampled per month. The average coke particle has the dimensions 31.6 x 17 x 9.5 $(\mu m)^3$, where σ_1 = 2.5, σ_2 = 2.0, and σ_3 = 1.45. The average diameter is 19.4 μm , where d_s = 16 μ m, and the range of average particle diameters is from 7.5 to 55 μm (Fig. 1). K, the volume coefficient = 0.359 \pm 0.027, which value is characteristic of flue dust. F, the surface coefficient = 2.347 ± 0.078, which value is characteristic of coal. S_W, the specific surface per unit weight, is equal to 0.25 x $10^8 \ \mu m^2/g$. S_V, the specific surface per unit volume, is equal to (0.1113 ± 0.012) x $10^{-4} \ \mu m^2/\mu m^3$.

Superfines

Superfines are similar to coke, but smaller (Fig. 1). The average particles have the dimensions $(7.1 \times 3.69 \times 1.62) \mu m^3$. For the particular sample illustrated $\sigma_1 = 1.90$; for all of the lots it is 1.27 μ m. The average particle diameter is (4.08 ± 0.68) μ m, where d_g = 3.69 μ m and the range of particle diameters is 1.5 to 15 μ m. The average volume is 33.3 μ m³, and the average surface area is 58.2 μ m². C = 5.08 (i.e., the particles are tetrahedral), f = 2.21 (1.e., the surface coefficient is characteristic of ace). Cient is characteristic of coal), and the volume coefficient k is 0.3167 (i.e., characteristic of tetrahedral particles of coal or flue dust). Roundness ø is 0.524, f/k is 6.98, and the anisotropy factor is 4.38.

Discussion of Results

The method is comparable with other methods such as the Elzone method (Fig. 2) which uses electrical conductivity of a suspension of liquid passing through a known orifice, and with other methods such as a laser light technique, and sedimentation analysis⁵,

Optical microscopy can be used to analyze size distribution in particles ranging in size from 1 to 50 um. The method is reproducible from batch to batch and there is no significant deviation from lot to lot. For smaller particles, scanning or trans-mission electron microscopy may be used. Both coke particles and superfines are similar in that they are irregularly shaped, highly anisotropic, and resemble coke particles or flue dust.





Conclusions

Optical stereology can be used to analyze particle size distribution in coke particles and superfines. It is reproducible and comparable to other techniques. In addition to size factors, shape factors and effective surface area may be determined from the same analytical data. The method has been utilized for other particles including BeO and silica spheres.

References

- 1. H. Heywood, "Particle Shape Coefficients," J.
- Imp. College, Chem. Eng. Soc., 2, 1944.
 2. J. D. Stockham and E. A. Fochtman, <u>Particle</u> Size Analyses, Ann Arbor Science, Ann Arbor, MI, 1977, pp. 1-21,111-117.
- G. Herndan, <u>Small Particle Statistics</u>, Academic Press, Inc., <u>Butterworths</u>, London, 1960.
 H. Wadell, "Sphericity and Roundness of Rock Particles," J. Geology, Vol. 41, 1933, pp. 310-331.
- 5. W. G. Bradshaw, 15th Biennial Conference on Carbons, Extended Abstracts and Program, Philadelphia, PA, June 1981, pp. 488-489.