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GB/T 3520-2024

Replacing GB/T 3520-2008

Test method for fineness of graphite

石墨细度试验方法

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1 Scope

The document describes test methods for the fineness of graphite.

It applies to the determination of fineness on graphite products.

3 Terms and definitions

3.1 the end of filter - the English term is printed in the document in this form and is reproduced here as it stands: after the test sample has been sieved for the specified time, sieving continues for a further period and is stopped when the ratio of the change in mass of the undersize, or of the residue, to the amount of sample is less than 0.3 %.

3.2 residue: the part of the charge that has not passed through the apertures of the specified sieve.

3.3 undersize: the part of the charge that has passed through the apertures of the specified sieve.

3.4 cumulative diameter Dn: geometric mean particle size corresponding to the n-th percentile of the cumulative size distribution taken from small to large. The note adds that n is usually an integer and that D10, D50 and D90 are the ones commonly used, the choice depending on the sector and the application. The definition is taken from GB/T 41949-2022, 3.3.

4 Taking and preparation of the test sample

4.1 A 1 kg sample is taken as laid down in GB/T 6679 and reduced by quartering to 200 g.

4.2 The reduced sample is dried in an electric drying oven at 110 °C +/- 5 °C until its moisture is below 1 %, then taken out, cooled to room temperature in a desiccator and held for testing.

5.1 Negative pressure sieving method

The measuring range is 38 micrometres and coarser.

Apparatus: a rotary air-jet sieve working at a negative pressure of 2 kPa to 6 kPa, shown schematically in Figure 1, whose parts are keyed as air jet nozzle, motor, control panel opening, negative pressure gauge connection, connection to the vacuum source and dust collector, and sieve seat; test sieves conforming to GB/T 6003.1, whose apertures are to be cleaned often and checked for size at intervals because the sample blocks and wears the mesh; a balance of accuracy not lower than 0.01 g; a stopwatch; a brush.

Procedure: the chosen test sieve is weighed to 0.01 g; 10 g of sample is weighed to 0.01 g, placed in the test sieve with its pan and spread evenly over the mesh; the loaded sieve is fitted on the seat of the rotary air-jet sieve, the lid is put on, the sieve is started with the working negative pressure held at 2 kPa to 6 kPa and the time is taken, while the lid and the frame are tapped lightly with a small wooden mallet so that sample sticking to them falls back onto the mesh. The sieving time is set by aperture: 2 min for sieves coarser than 75 micrometres, 3 min for sieves from 45 micrometres to 75 micrometres, and 5 min for sieves finer than 45 micrometres. The sieve is then taken out with its lid, the sample on the lid is brushed into the sieve, and the sieve together with the residue is weighed. The weighed sieve with its residue is refitted and check-sieved for 30 s to judge whether the end of sieving has been reached; if it has not, sieving continues in further 30 s steps until it has, and the sieve with the residue is weighed to 0.01 g.

Calculation: the residue content and the undersize content, as mass fractions expressed in percent, are calculated as X1 and X2 by formulas (1) and (2) and kept to two significant figures. The formulas come out of the extracted text with the fraction bar lost and are not reconstructed here. Their symbols are: X1, residue content as a mass fraction, in percent; X2, undersize content as a mass fraction, in percent; m0, mass of the empty test sieve, in grams; m, mass of the test sample, in grams; m1, total mass of the test sieve and the residue at the end of sieving, in grams.

When the absolute difference between two parallel determinations is not greater than 2.0 %, the arithmetic mean is taken as the result; otherwise the test is repeated on a fresh sample.

5.2 Sieve shaker method

The measuring range is 38 micrometres and coarser.

Apparatus: a sieve shaker conforming to DZ/T 0118; test sieves conforming to GB/T 6003.1, with the same warning about blocked and worn apertures; a balance of accuracy not lower than 0.01 g; a stopwatch; a brush.

Procedure: the chosen test sieve is weighed to 0.01 g; 50 g of sample is weighed to 0.01 g and poured into the test sieve with its pan; the lid is fitted and the sieve is shaken on the machine, the time being set by aperture: 10 min for sieves coarser than 75 micrometres, 20 min for sieves from 45 micrometres to 75 micrometres, and 30 min for sieves finer than 45 micrometres. When the set time is up the machine is stopped, the sieve is taken out and the sieve with the residue is weighed. The weighed sieve with its residue is refitted, check-sieved for 1 min and weighed again to judge whether the end of sieving has been reached; if it has not, sieving continues in further 1 min steps until it has, and the sieve with the residue is weighed to 0.01 g.

Calculation: the residue content and the undersize content, as mass fractions expressed in percent, are calculated as X3 and X4 by formulas (3) and (4) and kept to two significant figures. The formulas come out of the extracted text with the fraction bar lost and are not reconstructed here. Their symbols are: X3, residue content as a mass fraction, in percent; X4, undersize content as a mass fraction, in percent; m2, mass of the test sample, in grams; m3, mass of the empty test sieve, in grams; m4, total mass of the test sieve and the residue at the end of sieving, in grams.

When the absolute difference between two parallel determinations is not greater than 2.0 %, the arithmetic mean is taken as the result; otherwise the test is repeated on a fresh sample.

5.3 Laser diffraction method

The measuring range is 38 micrometres and finer.

Reagents and materials: dispersant, ethyl phenyl polyethylene glycol of purity not less than 98 %; dispersion medium, anhydrous ethanol of analytical grade or purified water meeting at least the grade three water of GB/T 6682, with anhydrous ethanol recommended for testing done for quality supervision and trade settlement and either medium for production control; dispersion, 3 g/L, prepared by weighing 1.5 g of the dispersant into a 250 mL beaker, adding 100 mL of the dispersion medium, stirring until dissolved, making up to the mark in a 500 mL volumetric flask with the dispersion medium and mixing; certified particle size, granularity or fine particle reference material; a glass rod; beakers.

Apparatus: a laser diffraction particle size analyser conforming to GB/T 41949; an ultrasonic disperser of 15 kHz to 30 kHz with adjustable power from 100 W to 800 W; a balance of accuracy not lower than 0.001 g; a stopwatch.

Preparation of the test portion: about 0.5 g of thoroughly mixed sample, weighed to 0.001 g, is placed in a beaker; 0.5 mL of the dispersion is added and stirred in gently with the glass

rod until the sample is completely wetted; 30 mL of the corresponding dispersion medium is added and stirred for about 5 min; the mixture is then poured into the ultrasonic disperser, the recommended settings being 15 kHz to 30 kHz, 100 W to 400 W and 120 s to 300 s.

Procedure: the analyser is switched on; before testing it is warmed up and its pipework and optics cleaned as its manual requires; dispersion medium is added to the sample cell; with the pump running between 1 500 r/min and 2 500 r/min the refractive indices of sample and medium and the other test parameters are set, and the background is measured until it meets the requirement; the prepared test portion is then added to the cell in the amount the instrument needs, and the measurement is started once the obscuration is within its limits. To keep the sample from re-agglomerating during the test, the total time of repeated measurements is not to exceed 10 min.

Results: the cumulative diameters D10, D50, D90 and Dmax are read from the size distribution table and the distribution curve and kept to two significant figures.

Repeatability: at least three measurements are made on a reference material within a measuring cycle in which enough analytical signal is obtained; the coefficient of variation is not to exceed 3 % for D50 and 5 % for D10, D90 and Dmax, and these maxima may be doubled where the cumulative diameter is below 10 micrometres.

Test report: it is recommended to give the sample name, the document applied, the production batch number, the date, time and place of testing, the instrument model used and the operator; to record the results as a size distribution table, a distribution curve and the cumulative diameters D10, D50, D90 and Dmax; to note any abnormal phenomena observed during testing; and to note any operation not laid down in the document and any freely chosen test condition.

6 Relationship to previous standards

The document was first issued in 1983 as GB 3520-1983, revised for the first time in 1995 as GB/T 3520-1995 and for the second time in 2008; the 2024 text is the third revision and replaces GB/T 3520-2008.

Against the 2008 edition the foreword lists these main technical changes: the scope of application has been changed; the terms and definitions have been changed; a clause on the taking and preparation of the test sample has been added; the negative pressure sieving method and the sieve shaker method have been changed; and the laser diffraction method has been added.