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Determination of the crucible swelling number of bituminous coal - Electrical heating method

烟煤坩埚膨胀序数的测定 电加热法

(ISO 501:2003, Hard coal - Determination of the crucible swelling number, MOD)

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Determination of the crucible swelling number of bituminous coal - Electrical heating method

1 Scope

This standard specifies the method summary, instruments and equipment, test preparation, test steps, result expression, method precision, test report, for determining the crucible swelling number (CSN).

This standard applies to bituminous coal.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB 474 Method for preparation of coal sample

GB/T 483 General rules for analytical and testing methods of coal

3 Method summary

Place the coal sample in a special crucible, to heat it to (820 ± 5) °C according to the specified procedure. The obtained coke block is compared with the side shape diagram of a set of standard coke blocks with serial numbers. Use the closest coke shape number as the crucible swelling number.

4 Instruments and equipment

4.1 Electric heating furnace (Figure 1)

On a grooved refractory plate, which has a diameter of 100 mm and a thickness of 13 mm, wind a nickel-chromium wire coil, which has a power of 1000 W. The refractory plate is placed on a board with the same specifications. Cover a quartz dish, which has a wall thickness of 1 mm, a height of 10 mm, an outer diameter of 85 mm, to accommodate the crucible.

The above-mentioned heating part is placed in a refractory brick, which has a diameter

of 140 mm and a groove with a depth of 60 mm and a diameter of 105 mm. The top is covered by a 20 mm thick refractory plate. There is a 50 mm diameter hole in the center of the plate, to accommodate the crucible. The entire refractory brick is placed on a 3 mm ~ 5 mm thick asbestos board. Fill insulation material between the surrounding bricks and the furnace shell. There is a refractory cover on the top of the furnace and a hole in the bottom. Insert the temperature-measuring thermocouple through the hole, until its hot junction is in contact with the inner surface of the quartz vessel. The electric heating furnace is equipped with suitable temperature measurement and temperature control devices.

4.2 Crucible and cover (Fig. 2)

It is made of porcelain or quartz, that is resistant to high temperatures (greater than 1000 °C). Crucible total height: (26 ± 0.5) mm; top outer diameter: (41 ± 0.75) mm; bottom inner diameter: 11 mm ~ 14 mm; mass: 11 g ~ 12.75 g; volume: 16 mL ~ 17.5 mL; inner diameter of crucible cover (no hole) 44 mm, height 5 mm.

4.3 Crucible cover with hole (Figure 3)

It is made of high temperature resistant (>1000 °C) porcelain or quartz. The size is the same as the 4.2 Crucible cover without hole; it has a 6 mm diameter round hole, for insertion of thermocouple.

4.4 Thermocouple

Armored nickel-chromium galvanic couple, 2 pieces.

4.5 Coke block observation tube (Figure 4)

The coke block observation tube shall comply with the requirements in Figure 4.

4.6 Heavy objects

(500 ± 10) g flat bottom weight.

4.7 Timer

Accurate to seconds (s).

4.8 Balance

The minimum graduation value is 0.01 g.

to GB 474. During specimen preparation, coal specimens shall be prevented from being ground too finely. The specimen shall be tested as soon as possible after preparation; otherwise, it shall be sealed and refrigerated. The test period shall not exceed 3 days. The coal specimens shall be thoroughly mixed for at least 1 minute before weighing.

6 Instrument debugging

Turn on the electric heating furnace. Heat it to about 850 °C and maintain a constant temperature. Open the furnace cover. Place a cold empty crucible into the center of the quartz dish in the furnace (start the stopwatch at the same time). Quickly apply the crucible cover with a hole. Then insert the thermocouple into the crucible through the cover hole. Press it to make the hot contact be close to the inner surface of the bottom of the crucible. Observe the temperature rise, without covering the electric furnace cover. If the bottom temperature in the crucible reaches (800 ± 10) °C within 1.5 minutes and reaches (820 ± 5) °C within 2.5 minutes after the cold crucible is put in, record the furnace temperature and current & voltage adjustment method. Follow this method when conducting the test control. If the above requirements cannot be met, adjust the voltage, current, furnace temperature, until the above requirements are met.

7 Test steps

7.1 Weigh (1.00 ± 0.01) g of air-dried coal specimen. Put it into the crucible and shake it flat. Then place it on a rubber board, which has a thickness of not less than 5 mm. Grasp the crucible containing the coal specimen downwards with the five fingers of your hand, to lift it about 15 mm height. Let go and let it fall freely. Fall like this for a total of 12 times (rotate the crucible by an angle each time it falls).

7.2 Open the furnace cover. Place the crucible containing the coal specimen into the center of the quartz dish in the furnace, that has been heated to the predetermined temperature. Immediately cover it with a crucible cover without holes. Start the stopwatch at the same time, until all volatile matter escapes. The escape time shall not be less than 2.5 min. Then take out the crucible. Do not cover the electric furnace during this process.

7.3 Each coal specimen is tested three times in succession. After three tests, carefully pour out the coke residue from the crucible. Measure the coke shape after the coke residue cools to room temperature. If the range of three measured values exceeds 1/2, it shall add two single tests. If the range of the five measured values exceeds 1, it shall check the equipment, to make 5 repeated measurements.

Note: Between two tests, cover the electric heating furnace cover, to bring the furnace temperature back to the preset temperature as quickly as possible.

7.4 After the test is completed, burn off the residue on the crucible and crucible cover;

wipe them clean.

8 Result expression and reporting

8.1 Crucible swelling number of coal sample

The crucible swelling number of the coal sample is determined and expressed according to the following method:

a) Swelling number 0: Coke slag is not sticky or powdery;

b) Swelling number 1/2: The coke residue adheres to the coke block without swelling. Place the coke block on a flat hard board; carefully apply a 500 g load to crush it or break into more than 2 pieces;

c) Swelling number 1: The coke residue adheres to the coke block without swelling. After adding a 500 g load, it cannot be crushed or broken into no more than 2 hard pieces.

d) Swelling number 1½ ~ 9: The coke residue adheres to the coke block and swells. Place the coke block under the coke block observation tube. Rotate the coke block, to find the maximum side shape. Chen compare it with the side shape of a set of standard coke blocks with serial numbers (Figure 5). Take the number of the closest standard side shape as its swelling number;

e) Swelling number greater than 9: The coke residue adheres to the coke block and swells. Place the coke block under the coke block observation tube. Rotate the coke block. The maximum side shape exceeds the standard coke block's side shape 9 (Figure 5), which is recorded as ">9" or "9^{+}".

8.2 Result reporting

Take the arithmetic mean of 3 measurement results, which have a range of no more than 1/2 for the same coal sample; round it to 1/2 units and report it in accordance with GB/T 483; round up 3 and round off 2 for the digits after the decimal point; if 5 measurements are taken, take the arithmetic mean of 5 measurement results; round it to 1/2 units and report it.

9 Method precision

The repeatability limit of the swelling number of bituminous coal crucible is: the range of the results of 3 repeated measurements is not greater than 1/2; the range of the results of 5 repeated measurements is not greater than 1.

The critical difference in reproducibility is as follows: the difference between the

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