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Replacing GB/T 26050-2010

Hardmetals - Determination of contents of metallic elements - X-ray fluorescence spectrometry

硬质合金 金属元素含量的测定 X射线荧光光谱法

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1 Scope

The document describes an X-ray fluorescence method for the contents of cobalt, chromium, iron, manganese, molybdenum, niobium, nickel, tantalum, titanium, vanadium, tungsten and zirconium in hardmetals after fusion; the contents of metallic elements in refractory metal carbides may also be determined by reference to it.

It applies to the carbides of niobium, tantalum, titanium, vanadium, tungsten and zirconium and to the presintered and sintered hardmetals made from those carbides. Table 1 gives the measuring range of each element as a mass fraction in per cent: cobalt 0.05 to 50.00; chromium 0.05 to 5.00; iron 0.05 to 10.00; manganese 0.05 to 2.50; molybdenum 0.05 to 5.00; niobium 0.05 to 15.00; nickel 0.05 to 20.00; tantalum 0.05 to 30.00; titanium 0.05 to 30.00; vanadium 0.05 to 4.00; tungsten 45.00 to 95.00; zirconium 0.05 to 2.00. Clause 3 states that the document defines no terms.

4 Principle and interfering elements

The test portion is first converted to oxides by an oxidation or an acid dissolution method, then fused with a suitable flux into a borate glass bead. The intensity of the characteristic X-ray line of each element to be determined is measured and read against a calibration curve, with correction for interelement effects, to obtain the mass fraction of each element.

Clause 5 on interfering elements gives titanium and tungsten acting on vanadium as an example of interference with the spectral lines. The Chinese sentence of this clause is incomplete in the original and has been rendered as it stands.

6 Reagents

Water complying with GB/T 6682-2008, grade 3 or better.

Anhydrous lithium tetraborate, or a mixed flux of anhydrous lithium tetraborate and lithium metaborate, in the anhydrous dry state; otherwise it is ignited at 500 °C to 600 °C for 4 h, cooled and kept in a desiccator.

Hydrofluoric acid of density 1.12 g/mL, analytical grade; nitric acid (1+1); sulfuric acid (1+1).

Release agent: lithium bromide solution at 240 g/L; potassium iodide solution, lithium iodide solution and the like may also be used. A note recommends that the concentration of the solution be made up to suit the case in hand.

7 Apparatus and samples

The apparatus comprises an X-ray fluorescence spectrometer; a high-frequency fusion machine; a high-temperature furnace; a flat-bottomed platinum crucible of 95 % platinum and 5 % gold; a platinum alloy plate with a polished surface, of 85 % platinum, 10 % rhodium and 5 % gold or of 95 % platinum and 5 % gold; a brass ring or a heat-resistant steel or graphite cylinder; a grinding device; a porcelain boat; and a balance with a scale interval of 0.1 mg.

A sample for the oxidation method shall pass a 0.18 mm sieve and a sample for the acid dissolution method a 2 mm sieve.

9 Test procedure

A test portion of 1.0 g is weighed to 0.0001 g on the balance, and the test is run twice in parallel, the mean of the two being taken.

The test portion is converted to oxides by the oxidation method or by the acid dissolution method; the oxidation method is not to be used where the molybdenum content is above 0.10 %. For the oxidation method the test portion is placed in a porcelain boat of constant

mass, raised from room temperature to 700 °C to 950 °C in the furnace with air passing over it and oxidized for about 1 h until oxidation is complete. For the acid dissolution method the test portion is placed in a platinum crucible, 15 mL of nitric acid and 2 mL of hydrofluoric acid are added and the mixture is heated until the test portion has dissolved; 1 mL to 2 mL of sulfuric acid is then added to dry it and it is heated at about 600 °C until no more sulfur trioxide is evolved, and cooled.

The ignition factor is calculated from the mass of the boat or crucible with the test portion after ignition, the mass of the empty boat or crucible before ignition and the mass of the test portion, all in grams; the equation itself is damaged in the source text and is not reproduced here.

For the sample bead 0.2 g of the pretreated test portion and 6 g of the anhydrous lithium tetraborate or of the mixed flux are weighed to 0.001 g, mixed thoroughly in the platinum crucible, 0.7 mL of the lithium bromide release agent is added, and the mixture is fused in the high-frequency fusion machine at 1 000 °C to 1 100 °C for 10 min to 15 min until the oxides have melted into an even glass disc; the amounts of test portion, flux and release agent may be increased or reduced to suit the case. Where the high-temperature furnace is used instead, the crucible is heated at 750 °C to 850 °C until melting is complete and the reaction has ended, then covered and held at about 1 000 °C to 1 100 °C for 10 min to 15 min, the melt being swirled to make it uniform. The brass ring or the heat-resistant steel or graphite cylinder is set on the platinum alloy plate on a heater at 300 °C to 400 °C, the melt is poured into the preheated ring and cooled to room temperature so that the borate disc comes loose from the plate. The disc is then ground to a smooth surface, rinsed at once and dried; if a smooth surface is not obtained, a final wet or dry grinding on 220 grit paper is allowed, and in dry grinding one specimen shall not be contaminated by another through the paper.

Standard beads are made to establish the working curve. The standard samples used for them shall be close to the analytical sample in chemical composition and structure, shall cover the content range of the elements to be determined and shall be suitably graded; they may be made up by mixing metals or their compounds of accurately known content, and the beads are prepared in the same way as the sample beads.

Table 2 gives the recommended analytical lines and analysing crystals; the K alpha 1,2 line is recommended for cobalt, chromium, iron, manganese, molybdenum, niobium, nickel, titanium, vanadium and zirconium and the L alpha 1 line for tantalum and tungsten. Background correction or internal standard correction may be applied where needed. In the measurement the standard beads are run under the chosen conditions, a suitable model is used to correct the calibration curve, the working curve is drawn, a suitable standard bead is chosen as the drift correction bead, the instrument drift is corrected, and the fluorescence intensity of the sample bead is finally measured and converted to the content of the element through the calibration curve.

10 Processing of the test data, permissible difference and test report

The content of each element is expressed as a mass fraction in per cent and is obtained from the ignition factor and the mass fraction read from the working curve; the equation is damaged in the source text and is not reproduced here. Results are expressed to two decimal places and are rounded in accordance with GB/T 8170.

Table 3 fixes the permissible difference between two measured values as a mass fraction in per cent: 0.01 for contents from 0.05 to 0.10; 0.03 for contents above 0.10 up to 0.50; 0.10 for contents above 0.50 up to 2.00; 0.25 for contents above 2.00 up to 10.00; 0.40 for contents above 10.00 up to 30.00; and 1.00 for contents above 30.00 up to 95.00.

The test report shall give at least the object tested, the number of the document, the analytical result and its expression, any departure from the basic analytical procedure, any abnormal phenomenon observed, and the date of the test.