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High purity silicon carbide - Determination of trace elements

高纯碳化硅 微量元素的测定

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Warning - Personnel using this standard shall have practical experience in formal laboratory work. This standard does not indicate all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with relevant national laws and regulations.

1 Scope

This standard specifies a method for determining the content of trace elements in high-purity silicon carbide, by inductively coupled plasma atomic emission spectroscopy (ICP-OES) method and inductively coupled plasma mass spectroscopy (ICP-MS) method.

This standard is applicable to the determination of 16 elements such as aluminum, arsenic, calcium, chromium, copper, iron, mercury, potassium, magnesium, manganese, sodium, nickel, lead, sulfur, titanium, zinc in high-purity silicon carbide materials, which have a mass fraction content of more than or equal to 99.9%. The measurement range of each element is shown in Table 1 (based on mass fraction).

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/T 602 Chemical reagent -- Preparations of standard solutions for impurity

GB/T 6682 Water for analytical laboratory use -- Specification and test methods

3 Principles

3.1 ICP-OES method

After the sample is melted and decomposed with sodium carbonate and borax at high temperature or dissolved with acid pressurization, it is excited in an inductively coupled plasma torch flame, to emit the characteristic spectrum lines of the elements contained. The content of the corresponding elements is determined according to the intensity of the characteristic spectrum lines of the elements to be measured.

3.2 ICP-MS method

After the sample is dissolved with acid pressurized, it is excited in an inductively coupled plasma torch flame. The ionized ions are separated by a quadrupole and collected by a photomultiplier tube. The content of the corresponding element is determined based on the intensity of the mass number of elements to be measured.

4 Reagents or materials

Unless otherwise stated, only reagents that are confirmed to be superior or superior or above are used in the analysis.

4.1 Water, first-grade water specified in GB/T 6682.

4.2 Sodium carbonate and borax.

4.3 Hydrochloric acid: density 1.19 g/mL.

4.4 Hydrochloric acid solution (1 + 1): Take hydrochloric acid (4.3) and water; dilute it 1:1 in volume ratio.

4.5 Hydrofluoric acid: Density 1.15 g/mL.

4.6 Sulfuric acid: Density 1.84 g/mL.

4.7 Nitric acid: Density 1.41 g/mL.

4.8 Single-element standard stock solution: Each single-element standard stock solution is prepared according to the method specified in GB/T 602, or the national standard solution is directly used, its mass concentration is 1.000 mg/mL.

4.9 ICP-OES calibration standard solution series: The ICP-OES calibration standard solution series is diluted and grouped by single-element standard stock solution. The recommended mass concentrations of each element are shown in Appendix A.

Weigh four portions of 0.2 g sample, accurate to 0.0001 g.

6.3.2 Sodium carbonate borax melting method

6.3.2.1 This method is suitable for the determination of all elements except potassium and sodium in the ICP-OES method.

6.3.2.2 Mix a portion of sample, 1g of sodium carbonate, 1 g of borax into a platinum crucible; cover it. Place it in a muffle furnace; heat it from room temperature to 800 °C at a heating rate of no more than 10 °C/min; then heat it to 1000 °C at a heating rate of no more than 5 °C/min; keep it in heat for 30 minutes; take it out with the furnace to cool below 100 °C.

6.3.2.3 After the platinum crucible is cooled to room temperature, add 20 mL ~ 25 mL of water; soak until the frit is loose; transfer to a 250 mL beaker. Add 10 mL of hydrochloric acid solution (1 + 1) in a total amount of 10 mL to the platinum crucible and beaker; rinse the wall of the platinum crucible with water and cover, until there is no residue; heat and boil until all the salts dissolve; cool to room temperature. Transfer the solution into a 100 mL volumetric flask; wash the platinum crucible and cap with water; transfer the wash solution into the volumetric flask; dilute it with water to the marking line; shake it well to prepare for testing.

6.3.3 Pressurized acid dissolution method

6.3.3.1 This method is suitable for the determination of all elements tested by ICP-MS method.

6.3.3.2 Place a portion of the sample in a polytetrafluoroethylene inner tank; add 5 mL of hydrofluoric acid and 8 mL of nitric acid; then slowly add 5 mL of sulfuric acid.

6.3.3.3 After covering the inner cover, put it in the stainless steel jacket; cover the outer cover; tighten the center screw; put it in an electric oven at 240 °C for 16 hours. After cooling, unscrew the center screw; open the outer tank; remove the inner tank; transfer the solution to the platinum evaporation dish; clean the inner tank with deionized water three times; add the washing liquid to the evaporation dish. Place the platinum dish on the electric stove; heat it until no white sulfuric acid smoke appears. After cooling, add 5 mL of nitric acid and a small amount of water. After heating and dissolving, transfer the residue into a 25 mL volumetric flask; wash the evaporation dish 3 times; add the washing solution to the volumetric flask; use water to dilute it to the mark.

6.4 Blank solution

Take the same amount of all reagents as 6.3.2 and 6.3.3, but do not add samples.

7 Test steps

7.1 Calibration curve

7.1.1 ICP-OES method

According to the ICP-OES instrument instructions, the recommended working conditions and the wavelength of the spectral line analysis for each element (see Appendix A), ignite the plasma torch flame. After the torch flame is stabilized, the spectral intensity of the mixed standard series solution (recommended in Appendix A) is sequentially measured on ICP-OES; the intensity of each element is used as the dependent variable; the concentration of the element is used as the independent variable to perform linear regression, to draw the correction curve; the intercept (a), slope (b), linear correlation coefficient (R^2) are calculated. If the linear correlation coefficient (R^2) is less than 0.999, re-draw the calibration curve.

7.1.2 ICP-MS method

According to the ICP-MS instrument instructions, the recommended working conditions, the mass of each element (see Appendix A), ignite the plasma torch flame. After the torch flame is stabilized, the mass intensity of the mixed standard series solution (recommended in Appendix A) is sequentially determined on ICP-MS. The intensity of each element's mass number is used as the dependent variable; the concentration of the element is used as the independent variable to perform linear regression to draw the correction curve; the intercept (a), slope (b), linear correlation coefficient (R^2) are calculated. If the linear correlation coefficient (R^2) is less than 0.995, re-draw the calibration curve.

7.2 Blank test

The blank test shall be carried out in parallel with the test (7.3) and the same steps shall be taken.

7.3 Test

Four tests are performed in parallel.

According to the instrument conditions set in 7.1, the spectral intensity of each measured element in the sample solution (6.3) is measured; the concentration of each measured element is calculated from the calibration curve.

If the concentration of the element being measured exceeds the maximum concentration value of the standard solution, refer to Appendix A to prepare a mixed standard solution with a maximum concentration value slightly higher than the concentration value of the element being measured; repeat the operation steps 7.1 ~ 7.3.

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