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Methods for chemical analysis of niobium hafnium alloys - Determination of trace impurity elements content - Inductively coupled plasma mass spectrometry

铌铪合金化学分析方法 痕量杂质元素的测定 电感耦合等离子体质谱法

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1 Scope

This document describes a method for determining the content of trace impurity elements in niobium hafnium alloys.

This document applies to the determination of the content of lithium, beryllium, boron, magnesium, aluminium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, strontium, molybdenum, cadmium, tin, antimony, lead, bismuth and uranium in niobium hafnium alloys. The determination range for iron is 0.000 5 % to 0.010 %; the determination range for the other elements is 0.000 1 % to 0.010 %.

3 Terms and definitions

The terms and definitions established in GB/T 17433 apply to this document.

4 Principle

The test portion is dissolved in hydrofluoric acid and nitric acid. The mass spectrometric intensities of lithium, beryllium, boron, magnesium, aluminium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, strontium, molybdenum, cadmium, tin, antimony, lead, bismuth and uranium are measured by inductively coupled plasma mass spectrometry; the effect of the matrix is corrected by the internal standard method, the mass concentration of each impurity element is calculated by the working

curve method, and the result of the determination is expressed as a mass fraction.

5 Reagents and materials

Unless otherwise stated, only reagents confirmed to be of guaranteed grade or better are used in the analysis.

5.1 Water, GB/T 6682, grade 1.

5.2 Hydrofluoric acid, of density 1.13 g/mL.

5.3 Nitric acid, of density 1.42 g/mL.

5.4 Single-element stock standard solutions of lithium, beryllium, boron, magnesium, aluminium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, strontium, molybdenum, cadmium, tin, antimony, lead, bismuth, uranium and rhodium: certified standard solutions are used, of mass concentration 100 μg/mL.

5.5 Mixed standard solution: 1.00 mL of each of the stock standard solutions (5.4) of lithium, beryllium, boron, magnesium, aluminium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, strontium, molybdenum, cadmium, tin, antimony, lead, bismuth and uranium is pipetted into a 100 mL plastic volumetric flask; 2 mL of nitric acid (5.3) and 1 mL of hydrofluoric acid (5.2) are added, and the solution is diluted to the mark with water and mixed. 1 mL of this solution contains 1 μg of each of lithium, beryllium, boron, magnesium, aluminium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, strontium, molybdenum, cadmium, tin, antimony, lead, bismuth and uranium.

5.6 Rhodium internal standard solution: 1.00 mL of the rhodium stock standard solution (5.4) is pipetted into a 100 mL plastic volumetric flask; 2 mL of nitric acid (5.3) is added, and the solution is diluted to the mark with water and mixed. 1 mL of this solution contains 1 μg of rhodium.

6 Apparatus

6.1 Inductively coupled plasma mass spectrometer: with a mass resolution of not more than 0.8 u; fitted with a sample introduction system resistant to hydrofluoric acid; fitted with a component able to eliminate the interfering argon monoxide ion of mass 56.

6.2 The recommended isotope masses of the elements to be determined and of the internal standard element are given in Table 1. They are: lithium 7, beryllium 9, boron 11, magnesium 26, aluminium 27, vanadium 51, chromium 52, manganese 55, iron 56, cobalt 59, nickel 60, copper 63, zinc 66, gallium 71, arsenic 75, strontium 87, molybdenum 95, cadmium 114, tin 118, antimony 121, lead 208, bismuth 209, uranium 238 and, as internal standard, rhodium 103. A footnote to the table states that for the element iron the collision mode is used to eliminate the interference.

7 Sample

The sample is machined into chips of not more than 5 mm in length.

8 Test procedure

8.1 Test portion. 0.10 g of the sample (Clause 7) is weighed to the nearest 0.000 1 g.

8.2 Parallel tests. Two parallel tests are carried out and the mean of the results is taken.

8.3 Blank test. A blank test is carried out alongside the test portion.

8.4 Preparation of the test solution. The test portion (8.1) is placed in a 100 mL polytetrafluoroethylene beaker and wetted with a little water. 2 mL of hydrofluoric acid (5.2) and 1 mL of nitric acid (5.3) are added, the beaker is covered with its lid, and it is heated gently until the test portion has dissolved completely; it is then removed and cooled to room temperature. The solution is transferred into a 100 mL plastic volumetric flask, 2.00 mL of the rhodium internal standard solution (5.6) is added, and the solution is diluted to the mark with water and mixed. Note: the rhodium internal standard solution (5.6) may also be added on line.

8.5 Preparation of the working curve solutions. 0 mL, 0.10 mL, 0.50 mL, 1.00 mL, 2.00 mL, 5.00 mL and 10.00 mL of the mixed standard solution (5.5) are pipetted into a set of 100 mL plastic volumetric flasks; 2 mL of hydrofluoric acid (5.2), 1 mL of nitric acid (5.3) and 2.00 mL of the rhodium internal standard solution (5.6) are added to each, and the solutions are diluted to the mark with water and mixed. The concentration gradient of the working curve may be adjusted according to the mass concentration of the elements to be determined in the test solution, and shall include at least 5 standard points.

8.6.1 The inductively coupled plasma mass spectrometer is started up and its parameters are adjusted. Once the instrument is stable, the working curve solutions (8.5) are introduced into the inductively coupled plasma mass spectrometer, and the mass spectrometric intensities of the elements to be determined are measured at the isotope masses recommended in Table 1. The working curve is plotted with the mass concentration of the element to be determined as the abscissa and the mass spectrometric intensity as the ordinate; the linear correlation coefficient of the working curve shall be not less than 0.999.

8.6.2 The blank test solution (8.3) and the test solution (8.4) are introduced into the inductively coupled plasma mass spectrometer, the mass spectrometric intensities of the elements are measured, and the mass concentration of each element is calculated from the working curve.

9 Treatment of the test data

The content of the element to be determined, expressed as a mass fraction, is calculated by formula (1), in which the mass concentration of the element to be determined in the test solution and the mass concentration of that element in the blank test solution are in nanograms per millilitre (ng/mL), the volume of the test solution is in millilitres (mL), and the mass of the test portion is in grams (g).

When the mass fraction is less than 0.010 %, the calculated result is expressed to four decimal places. When the mass fraction is 0.010 %, it is expressed to three decimal places, rounded in accordance with GB/T 8170.

10 Precision

10.1 Repeatability. For the measured values of two independent test results obtained under repeatability conditions, within the range of mean values given in Table 2, the absolute difference between the two test results does not exceed the repeatability limit r, and the cases in which the repeatability limit r is exceeded do not exceed 5 %. The repeatability limit r is obtained from the data of Table 2 by linear interpolation or extrapolation. The statistical data obtained from the interlaboratory test results are given in Annex A. Table 2 pairs a mass fraction of 0.000 5 % with a repeatability limit of 0.000 1 %, 0.001 0 % with 0.000 2 %, 0.003 0 % with 0.000 3 %, 0.006 0 % with 0.000 4 %, and 0.009 0 % with 0.000 6 %.

10.2 Reproducibility. For the measured values of two independent test results obtained under reproducibility conditions, within the range of mean values given in Table 3, the absolute difference between the two test results does not exceed the reproducibility limit R, and the cases in which the reproducibility limit R is exceeded do not exceed 5 %. The reproducibility limit R is obtained from the data of Table 3 by linear interpolation or extrapolation. The statistical data obtained from the interlaboratory test results are given in Annex A. Table 3 pairs a mass fraction of 0.000 5 % with a reproducibility limit of 0.000 2 %, 0.001 0 % with 0.000 4 %, 0.003 0 % with 0.000 5 %, 0.006 0 % with 0.000 7 %, and 0.009 0 % with 0.001 1 %.

11 Test report

The test report shall give at least the following: the object of the test; the number of this document; the calculated results; any departures from the basic analytical procedure; any unusual phenomena observed; the date of the test.

A Annex A (informative) Statistical data obtained from the interlaboratory test results

The participating laboratories carried out a joint test on 5 different levels of niobium hafnium alloy, each laboratory making 9 independent determinations on each level under