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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13747.27-2020

Methods for chemical analysis of zirconium and zirconium alloys - Part 27: Determination of trace impurities content - Inductively coupled plasma mass spectrometry

锆及锆合金化学分析方法 第27部分：痕量杂质元素的测定 电感耦合等离子体质谱法

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Foreword

GB/T 13747 "Methods for Chemical Analysis of Zirconium and Zirconium Alloys" is planned to be divided into 27 parts.

1.Determination of the amount of tin potassium iodate titration and phenylfluorone-polyethylene glycol octylphenyl ether spectrophotometry;

2.Determination of iron content 1,10-phenanthroline spectrophotometry and inductively coupled plasma atomic emission spectrometry;

3.Determination of nickel content diacetyl oxime spectrophotometry and inductively coupled plasma atomic emission spectrometry;

4.Determination of chromium content Diphenylcarbazide spectrophotometry and inductively coupled plasma atomic emission spectrometry;

5.Determination of aluminum content Chromazurol S-tetradecylpyridinium chloride spectrophotometric method;

6.Determination of copper content 2,9-dimethyl-1,10-phenanthroline spectrophotometry;

7.Determination of manganese content, potassium periodate spectrophotometry and inductively coupled plasma atomic emission spectrometry;

8.Determination of Drill Volume Nitroso R Salt Spectrophotometry;

9.Determination of magnesium content by flame atomic absorption spectrometry;

10.Determination of Tungsten Content Thiocyanate Spectrophotometric Method;

11.Determination of molybdenum content thiocyanate spectrophotometric method;

12.Determination of silicon content molybdenum blue spectrophotometry;

13.Determination of lead content by polarography;

1 Scope

GB/T 13747.27-2020 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 27: determination of trace impurities content - inductively coupled plasma mass spectrometry, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 6 March 2020 by the China Nonferrous Metals Industry Association, and has been in force since 1 February 2021. Classification: ICS 77.120.99, CCS H14. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This part of GB/T 13747 specifies boron, magnesium, aluminum, titanium, vanadium, chromium, manganese, cobalt, nickel, copper, molybdenum, tin, hafnium, tungsten, lead, and lead in zirconium and zirconium alloys. Method for determination of uranium content. This section applies to the content of boron, magnesium, aluminum, titanium, vanadium, chromium, manganese, cobalt, nickel, copper, molybdenum, tin, hafnium, tungsten, lead and uranium in sponge zirconium, zirconium and zirconium alloys The determination. Measuring range. 0.00005%~0.010%. When the measurement range overlaps with other parts of GB/T 13747, Law is the method of arbitration.

2 Principle

The sample is dissolved in nitric acid and hydrofluoric acid, calibrated with thulium internal standard, and directly measured by inductively coupled plasma mass spectrometer, according to the working curve The method calculates the mass concentration of each element and expresses the measurement result in mass fraction.

3 Reagent

Unless otherwise stated, only reagents and laboratory first-grade water confirmed to be pure superior grade are used in the analysis.

3.1 Hydrofluoric acid (rho=1.13g/mL).

3.2 Nitric acid (rho=1.42g/mL).

3.3 Single element standard storage solutions of boron, magnesium, aluminum, titanium,

vanadium, chromium, manganese, cobalt, nickel, copper, molybdenum, tin, hafnium, tungsten, lead, uranium and thulium (national standard sample/ National standard material), the mass concentration is 1000μg/mL.

3.4 Mixed standard solution A. Pipette 1.00mL of each element standard stock solution (except thulium element) (3.3) into 100mL plastic volume. In the bottle, add 2 mL of nitric acid (3.2), dilute to the mark with water, and mix. This solution 1mL contains boron, magnesium, aluminum, titanium, vanadium, chromium, manganese, cobalt, nickel, Copper, molybdenum, tin, hafnium, tungsten, lead, and uranium each 10μg.

3.5 Mixed standard solution B. Pipette 10.00mL mixed standard solution A (3.4) into a 100mL plastic volumetric flask, add 2mL nitric acid (3.2), dilute to the mark with water, and mix well. This solution 1mL contains boron, magnesium, aluminum, titanium, vanadium, chromium, manganese, cobalt, nickel, copper, molybdenum, tin, hafnium, tungsten, lead and Uranium is 1μg each, ready to use when used.

3.6 Thulium internal standard solution. pipette 1.00mL thulium element standard stock solution (3.3) into a 1000mL plastic volumetric flask, and add 2mL nitric acid (3.2), dilute to the mark with water, and mix well. This solution 1mL contains 1μg thulium.

3.7 Liquid argon or high-purity argon (purity>=99.99%).

4 Instruments

4.1 Inductively coupled plasma mass spectrometer. mass resolution is better than 0.8u±0.1u (at 10% peak height); equipped with hydrofluoric acid resistant injection system System; equipped with components that can eliminate interfering ions such as 38Ar1H, 40Ar and 40Ar16O.

4.2 The isotope determination of each element is shown in Table 1.

5 Samples

Process the sample into chips with a length not greater than 5mm.

6 Test procedure

6.1 Sample Weigh 0.10g sample (Chapter 5), accurate to 0.0001g.

6.2 Number of determinations Do two tests in parallel and take the average value.

6.3 Blank test Make a blank test with the sample.

6.4 Preparation of analysis test solution Place the sample (6.1) in a 100mL polytetrafluoroethylene beaker, blow a small amount of water along the wall of the cup, add 3mL nitric acid (3.2), 1mL Hydrofluoric acid (3.1), heat at low temperature until the sample is completely dissolved, cool, transfer to a 100mL plastic volumetric flask, add 1.00mL

thulium internal standard solution (3.6), dilute to the mark with water, and mix well.

6.5 Preparation of working curve solution Pipette 0mL, 0.10mL, 0.50mL, 1.00mL, 2.00mL, 5.00mL, 10.00mL mixed standard solution B (3.5) in a series In a 100mL plastic volumetric flask, add 1.00mL Thulium internal standard solution (3.6), dilute to the mark with water, and mix.

6.6 Determination

6.6.1 On the inductively coupled plasma mass spectrometer, after the instrument is running stably, under the selected instrument working conditions, perform the same position as recommended in Table 1. Prime mass number, use the prepared standard series solution (6.5) to calibrate the calibration working curve, the correlation coefficient of the working curve of each element should be

0.999 Above, otherwise it is necessary to re-standardize or re-formulate standard series solutions for standardization.

6.6.2 Determination of analysis test solution (6.4) and blank test solution (6.3). The instrument automatically processes data according to the working curve, calculates and outputs each element Element mass concentration.

7 Test data processing

The content of each element is calculated by mass fraction wx, expressed in %, and calculated according to formula (1). Where. rhox

--- the mass concentration of the element to be tested in the test solution, in nanograms per milliliter (ng/mL); rho0

--- The mass concentration of the element to be tested in the blank test solution, in nanograms per milliliter (ng/mL); V

---The volume of the analysis test solution, in milliliters (mL); m

---The mass of the sample, in grams (g). Analysis results Magnesium, aluminum, titanium, vanadium, chromium, manganese, cobalt, nickel, copper, molybdenum, tin, hafnium, tungsten, lead and uranium elements should be expressed to four decimal places; boron element It should be expressed to five decimal places.

8 Tolerance

The difference between the analysis results between the laboratories should not be greater than the relative tolerance listed in Table 2.

9 Test report