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Methods for chemical analysis of tantalum and niobium - Part 11:Determination of arsenic,antimony,lead,tin and and bismuth contents in niobium - Direct current arc atomic emission spectrometry

钽铌化学分析方法 第11部分:铌中砷、锑、铅、锡和铋量的测定 直流电弧原子发射光谱法

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Foreword

GB/T 15076 "Methods for Chemical Analysis of Tantalum and Niobium" is divided into 16 parts.

1.Determination of tantalum content in niobium by inductively coupled plasma atomic emission spectrometry;

2.Determination of the amount of niobium in tantalum inductively coupled plasma atomic emission spectrometry and chromatography gravimetric method;

3.Determination of copper content by flame atomic absorption spectrometry;

4.Determination of iron content 1,10-phenanthroline spectrophotometry;

5.Determination of the amount of molybdenum and tungsten by inductively coupled plasma atomic emission spectrometry;

6.Determination of silicon content by inductively coupled plasma atomic emission spectrometry;

7.Determination of phosphorus content in niobium 4-methyl-pentanone-[2] extraction separation phosphomolybdenum blue spectrophotometry and inductively coupled plasma Bulk atomic emission spectroscopy;

8.Determination of carbon and sulfur content by high-frequency combustion infrared absorption method;

9.Determination of the amount of iron, chromium, nickel, manganese, titanium, aluminum, copper, tin, lead and zirconium in tantalum by DC arc atomic emission spectrometry;

10.Determination of the content of iron, nickel, chromium, titanium, zirconium, aluminum and manganese in niobium by DC arc atomic emission spectrometry;

11.Determination of arsenic, antimony, lead, tin and bismuth in niobium by DC arc atomic emission spectrometry;

12.Determination of Phosphorus in Tantalum by Ethyl Acetate Extraction and Separation of Phosphomolybdenum Blue Spectrophotometry;

13.Determination of nitrogen content inert gas fusion thermal conductivity method;

1 Scope

GB/T 15076.11-2020 is the Chinese national standard on methods for chemical analysis of tantalum and niobium - part 11:determination of arsenic,antimony,lead,tin and and bismuth contents in niobium - direct current arc atomic emission 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 15076 specifies the determination of arsenic, antimony, lead, tin and bismuth in niobium by DC arc atomic emission spectrometry. This section applies to the determination of arsenic, antimony, lead, tin and bismuth in niobium and niobium hydroxide, niobium oxide and niobium carbide. The measurement range is shown in Table 1.

2 Principle

The metal niobium and its hydroxides and carbides are burnt to convert into niobium pentoxide, and a certain proportion of graphite powder and sodium fluoride are mixed as a buffer. Granules, DC arc anode excitation, spectrometry.

3 Reagents or materials

3.1 Niobium pentoxide. wNb2O5>=99.99% (wAs<=0.0001%, the content of other single measured elements wx<=0.00001%), in muffle Burn in the furnace at 850°C for 2h~3h, put it in a desiccator and cool to room temperature for later use.

3.2 Arsenic trioxide (wAs2O3>=99.99%).

3.3 Antimony trioxide (wSb2O3>=99.99%).

3.4 Lead dioxide (wPbO2>=99.99%).

3.5 Tin dioxide (wSnO2>=99.99%).

3.6 Bismuth trioxide (wBi2O3>=99.99%).

3.7 Sodium fluoride. pure superior grade.

3.8 Graphite powder. pure spectrum.

3.9 Buffer. Weigh 9.40g graphite powder (3.8), add 0.60g sodium fluoride (3.7), and grind well.

3.10 Compression mold. made of plexiglass rod car, the top is 2.9mmx2mm.

3.11 Argon (volume fraction not less than 99.99%).

3.12 Graphite electrode. pure spectrum, the structure and size of the upper electrode and the lower electrode are shown in Figure 1. Unit is mm

a) Upper electrode

b) Lower electrode Figure

4 Equipment

DC arc atomic emission spectrometer.

5 Samples

5.1 The particle size of niobium carbide is less than 150μm, and the particle size of niobium powder is less than 180μm.

5.2 Niobium hydroxide and niobium oxide should be pre-baked at 105°C~110°C for 2h, placed in a desiccator and cooled to room temperature for later use.

5.3 From the top 10cm of the niobium ingot to any part of the middle, use a planer to remove the skin and sample the shavings. The size of the shavings should be uniform, and And the particles should be as small as possible.

6 Test procedure

6.1 Sample processing Take about 1g of the sample and place it in a porcelain crucible or platinum crucible, in a box-type resistance furnace, heat up to 900 °C and burn for 1 hour (niobium wire and niobium ingot need to be burned 4h or more), make it completely converted into niobium pentoxide, take it out, and cool to room temperature.

6.2 Parallel test Do two tests in parallel and take the average value.

6.3 Preparation of Spectral Sample

6.3.1 Weigh an appropriate amount of oxide sample (6.1) and buffer (3.9), mix at a ratio of 2.1, and grind uniformly.

6.3.2 Load the mixture (6.3.1) into the graphite electrode (3.12) and press it with a compression mold (3.10) to make the sample 1mm lower than the electrode port for measurement.

6.4 Determination

6.4.1 Standard sample preparation Accurately weigh 9.3944g niobium pentoxide (3.1) in an agate mortar, and add 0.1320g arsenic trioxide (3.2) in sequence, 0.1197g antimony trioxide (3.3), 0.1154g lead dioxide (3.4), 0.1270g tin dioxide (3.5), 0.1115g bismuth trioxide (3.6), grind well, prepare a master standard sample with a content of 1% of each measured element, and then gradually dilute it with niobium pentoxide (3.1) into a standard sample series. See Table 2 for sample element content. Weigh appropriate amounts of each standard sample and buffer (3.9) and treat them as per 6.3.

6.4.2 Recommended measurement conditions The measurement conditions of the instrument are as follows:

---Light source. DC arc, current 15.5A;

--- Electrode spacing. 2mm;

---Pre-burning time. 2s;

---Exposure time. 50s;

---Refer to Table 3 for the recommended analysis spectrum of each element.

6.4.3 Measurement In the DC arc atomic emission spectrometer (4), according to the recommended analysis line, determine the working curve standard (6.4.1) and analyze the sample (6.3.2), check the background of the spectral lines of each element and correct it at the appropriate position, and the computer will automatically give each measured element in the niobium pentoxide The quality score of the element.

7 Test data processing

The content of the measured element is calculated as the mass fraction wM of the measured element, calculated according to formula (1).

8 Precision