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Methods for chemical analysis of tantalum and niobium - Part 4:Determination of iron content - 1,10-Phenanthroline spectrophotometry

钽铌化学分析方法 第4部分:铁量的测定 1，10—二氮杂菲分光光度法

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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 15076.4-2020 is the Chinese national standard on methods for chemical analysis of tantalum and niobium - part 4:determination of iron content - 1,10-phenanthroline spectrophotometry, 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 1,10-phenanthroline spectrophotometric method for the determination of iron content in tantalum and niobium. This section applies to the determination of iron content in tantalum, niobium and their hydroxides and oxides. Measuring range. >0.020%~0.50%.

2 Principle

The sample is dissolved with hydrofluoric acid and nitric acid, tartaric acid-boric acid complexes the main body, fluorine and other impurity elements, and the ferric ion is reduced by ascorbic acid It is a divalent iron ion. In a buffer solution with a pH of about 5, the divalent iron ion forms a red complex with 1,10-phenanthroline. Measure the absorbance at 510nm with a meter.

3 Reagents and materials

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

3.1 Hydrofluoric acid (rho=1.14g/mL).

3.2 Hydrochloric acid (rho=1.18g/mL).

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

3.4 Acetic acid (rho=1.05g/mL).

3.5 Hydrochloric acid (1 1).

3.6 Acetic acid (1 1).

3.7 Tartaric acid. analytically pure.

3.9 Ascorbic acid. analytically pure.

3.10 Ammonium acetate. analytically pure. 3.11 1,10-phenanthroline. analytically pure.

3.12 Tartaric acid-boric acid solution. Weigh 10g tartaric acid (3.7) and 6g boric acid (3.8) into a 250mL beaker, add 80mL water, add After hot dissolution, cool, transfer to a 100mL volumetric flask, dilute to the mark with water, and mix.

3.13 Ascorbic acid solution (20g/L). Weigh 2g ascorbic acid (3.9) into a 250mL beaker, add 50mL water, stir to dissolve it After all, transfer to a 100mL volumetric flask, dilute to the mark with water, and mix well. Available now.

3.14 Acetic acid-ammonium acetate buffer solution (pH=5). Weigh.200g of ammonium acetate (3.10) into a 500mL volumetric flask, add.200mL of water, and add Heat to dissolve completely, cool, add 50mL acetic acid (3.6), transfer to a 1000mL volumetric flask, dilute to the mark with water, and mix. 3.15 1,10-phenanthroline ethanol solution (4g/L). Weigh 4g of 1,10-phenanthroline (3.11) into a 100mL beaker and add 20mL After stirring and dissolving ethanol, transfer to a 1000mL volumetric flask, dilute to the mark with water, and mix well.

3.16 Niobium metal (wNb>=99.99%, wFe<=0.0001%).

4 Equipment

Spectrophotometer.

5 Samples

5.1 The particle size of tantalum powder is less than 700μm; the particle size of niobium powder is less than 180μm.

5.2 Tantalum powder should be dried in vacuum at 80°C~150°C for 4h in advance, cooled to room temperature, and vacuum packaged in a composite aluminum foil bag; Substances and oxides should be pre-baked at 105°C~110°C for 2h, and placed in a desiccator to cool to room temperature for later use.

5.3 From the top 10cm of the tantalum ingot or niobium ingot to any part of the middle, use

a planer to remove the skin and sample the shavings. The size of the debris should be Uniform, and the particles should be as small as possible.

6 Test procedure

6.1 Sample Weigh 0.20g sample, accurate to 0.0001g.

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

6.3 Blank test Do a blank test with the sample.

6.4 Determination

6.4.1 Place the sample (6.1) in a 30mL platinum crucible and wet it with water. Add 2mL of hydrofluoric acid (3.1) to the tantalum sample or 4mL of the niobium sample Hydrofluoric acid (3.1), add nitric acid (3.3) dropwise, heat at low temperature until the sample is completely dissolved, remove and cool to room temperature, add 20mL tartaric acid-boric acid to dissolve Solution (3.12), transfer the solution into a 100mL polyethylene volumetric flask pre-added with 10mL tartaric acid-boric acid solution (3.12), dilute with water to Scale and mix well. When the oxide is not easy to dissolve to clarification by this method, a hot-pressure digestion device can be used to dissolve the sample.

6.4.2 Divide the above solution (6.4.1) according to Table 1 into a 25mL colorimetric tube, add 2mL ascorbic acid solution (3.13), 5mL acetic acid-ethyl Ammonium acid buffer solution (3.14), 2.5mL 1,10-phenanthroline ethanol solution (3.15), (each reagent needs to be mixed), diluted with water to Shake well and place for 5min.

6.4.3 Move part of the solution (6.4.2) into a cuvette according to Table 1, take water as a reference, and measure its absorption at a wavelength of 510 nm on a spectrophotometer. Luminosity.

6.4.4 Subtract the absorbance of the blank solution accompanying the sample, and check the corresponding iron content from the working curve.

6.5 Drawing of working curve

6.5.1 Pipette 0mL, 1.00mL, 2.00mL, 3.00mL, 4.00mL, 5.00mL, 6.00mL iron standard solution B (3.20) into In a 25mL colorimetric tube, add 5mL tartaric acid-boric acid solution (3.12), and the rest of the operation is carried out according to 6.4.2~6.4.3.

6.5.2 Subtract the absorbance of the reagent blank, use the iron content as the abscissa and the absorbance as the ordinate to draw the working curve.

7 Test data processing

The iron content is calculated as the mass fraction of iron wFe, calculated according to