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Methods for chemical analysis of tantalum and niobium - Part 15: Determination of hydrogen content - Pulse infrared absorption method

钽铌化学分析方法 第15部分：氢含量的测定 脉冲红外吸收法

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Table of Contents

Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part

1.Structure and Drafting Rules of Standardization Documents". Drafting. This document is Part 15 of GB/T 15076, "Chemical Analysis Methods for Tantalum and Niobium". GB/T 15076 has the following parts published.

1.Determination of Tantalum Content in Niobium by Inductively Coupled Plasma Atomic Emission Spectrometry;

2.Determination of Niobium Content in Tantalum. Inductively Coupled Plasma Atomic Emission Spectrometry and Chromatographic Gravimetric Method;

3.Determination of Copper Content by Flame Atomic Absorption Spectrometry;

4.Determination of Iron Content by 1,10-Diazophenanthroline Spectrophotometry;

5.Determination of Molybdenum and Tungsten Contents 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 and Separation Phosphomolybdic Blue Spectrophotometry and Inductively Coupled Plasma Bulk atomic emission spectrometry;

---Determination of carbon and sulfur content;

---Determination of iron, chromium, nickel, manganese, titanium, aluminum, copper, tin, lead and zirconium content in tantalum;

10.Determination of Iron, Nickel, Chromium, Titanium, Zirconium, Aluminum and Manganese in Niobium by Direct Current Arc Atomic Emission Spectrometry;

11.Determination of Arsenic, Antimony, Lead, Tin and Bismuth Contents in Niobium by Direct Current Arc Atomic Emission Spectrometry;

---Determination of phosphorus content in tantalum;

5 Reagents and Materials

5.1 Carrier gas. Argon, with a purity (volume fraction) of not less than 99.995%.

5.2 Power gas. Argon or nitrogen, with a purity (volume fraction) of not less than 99.9%.

5.3 Nickel flux. high-purity nickel foil or nickel bladder, wH<=0.0001%, nickel foil thickness not greater than 0.10mm.

5.4 Tin flux. tin flakes or tin granules, wH<=0.0001%.

5.5 High-purity graphite crucible.

5.6 Standard substances or standard samples. Appropriate standard substances should be selected. In principle, the chemical composition of the standard substances should be similar to that of the analytical samples.

6.Instruments and Equipment Inert gas molten infrared hydrogen analyzer, including electrode furnace, carrier gas purification and analytical gas flow conversion system, infrared cell detector, computer and software. Instrument control system. See Appendix A for instrument reference optimization analysis conditions.

7.1 The particle size of tantalum powder should be less than 700 μm, and the particle size of niobium powder should be less than 180 μm. Tantalum powder should be pre-dried under vacuum at 80°C~150°C for 4 hours, and then cooled... Cool to room temperature and vacuum-pack in a composite aluminum foil bag.

7.2 Remove the outer skin of the block sample, cut it from the core of the sample, and process it into particles with a mass of no more than 0.03g.

8 Test Procedure

8.1 Samples Weigh 0.08g to 0.12g of sample (Chapter 7), accurate to 0.0001g.

8.2 Parallel Tests Perform two parallel measurements and take the average value.

8.3 Instrument Preparation

8.3.1 Assemble all components according to the instrument manual, and connect the power supply, carrier gas (5.1), and power gas (5.2).

8.3.2 Before analysis, fully preheat the instrument. When the instrument is completely powered off and then restarted, the preheating time should be no less than 2 hours.

Prolonged instrument operation is prohibited. When ventilating, the preheating time should not be less than 30 minutes.

8.3.3 Use the instrument leak detection procedure or other auxiliary equipment to confirm that there is no air leakage in the instrument.

8.4 Blank Test The blank value includes the blank for both the crucible and the flux. Place either the nickel flux (5.3) or the tin flux (5.4) into the high-purity graphite crucible (5.5). Perform parallel measurements 3 to 5 times, replacing the high-purity graphite crucible (5.5) each time. Take the average value of the measurement results and perform blank compensation according to the blank compensation program. White space deduction. Blank values are no greater than 0.0001%, and the range is no greater than 0.00005%.

8.5 Instrument Calibration

8.5.1 Single Standard Point Calibration Select a certified reference material or standard sample (5.6), perform 3 to 5 parallel determinations, calculate the average value of the results, and determine the hydrogen calibration. The slope of the quasi-curve. A single measurement must not exceed the uncertainty range given in the certificate for the reference material or standard sample (5.6), and must be measured using another... Verify the system using standard substances or standard samples (5.6) to confirm its linearity; otherwise, recalibrate the system.

8.5.2 Multi-standard point calibration Select two or more certified reference materials or standard samples (5.6), and perform parallel determinations 3 to 5 times for each reference material or standard sample. Take the average value of the measurement results of each standard substance or standard sample (5.6) to determine the slope of the hydrogen calibration curve. A single measured value must not exceed the standard value. The uncertainty range given in the certificate for the reference substance or standard sample (5.6) is used, and another reference substance or standard sample used in the calibration is used. (5.6) Verify the system to confirm its linearity; otherwise, recalibrate the system.

8.6 Measurement Select optimized analytical conditions, and place the sample (8.1) in a container of nickel flux (5.3) (the mass ratio of nickel flux to sample should not be less than 6.1) or The sample (8.1) along with the tin flux (5.4) (the mass ratio of tin flux to sample should not be less than 4.1) was placed into the sample injector, and an empty high-purity stone was added. The ink crucible (5.5) is placed on the lower electrode, the mechanical device is raised, the furnace door is closed, and the test begins. The instrument automatically displays the measurement results. (Continuous...) During the subsequent testing process, standard substances or standard samples (5.6) are inserted at intervals to monitor for drift and verify the initial validity.

9.Experimental Data Processing When the hydrogen content w < 0.0010%, the calculation result should be retained to one significant figure; when w >= 0.0010%, the calculation

result should be retained to two significant figures. Words. Numerical rounding shall be performed in accordance with the provisions of GB/T 8170.

10 Precision Under repeatability conditions, the absolute difference between two independent test results does not exceed the repeatability limit (r); otherwise, the result is considered invalid. The percentage is no more than 5%. Under reproducibility conditions, the absolute difference between two independent test results does not exceed the reproducibility limit (R); exceeding the reproducibility limit (R) is considered acceptable. The percentage is no more than 5%.

15 Determination of Hydrogen Content Pulse infrared absorption method

1.Scope This document describes a method for determining the hydrogen content in tantalum and niobium using pulsed infrared absorption. This document applies to the determination of hydrogen content in processed materials such as tantalum and niobium powder, ingots, and wires. Determination range (mass fraction). 0.0002%~0.10%.

3.Terms and Definitions This document does not contain any terms or definitions that need to be defined.

4.Principles The sample and flux were added to a high-purity graphite crucible and heated to melt under an inert gas (argon) protection. The hydrogen in the sample was in the form of hydrogen gas. After precipitation, hydrogen gas is oxidized to water by an oxidant, and then enters the infrared detector with the carrier gas flow. The detector outputs a signal, and the computer system determines the reaction based on the sample. The hydrogen content was calculated by mass, and the result was expressed as a mass fraction.