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GB/T 24583.2-2019

Replacing GB/T 24583.2-2009

Vanadium-nitrogen alloy - Determination of the nitrogen content - Thermal conductivity method after fusion in an inert gas stream

钒氮合金 氮含量的测定 惰性气体熔融热导法

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Foreword

GB/T 24583 Vanadium-nitrogen alloy analysis method is divided into 8 parts: - GB/T 24583.1 Vanadium-nitrogen - Determination of vanadium content - Ammonium ferrous sulfate titration method; - GB/T 24583.2 Vanadium-nitrogen - Determination of nitrogen content - Thermal conductimetric method after fusion in a current of inert gas; - GB/T 24583.3 Vanadium-nitrogen - Determination of nitrogen content - Distillation-neutralization titration method; - GB/T 24583.4 Vanadium-nitrogen - Determination of carbon content - Infrared absorption method; - GB/T 24583.5 Vanadium-nitrogen - Determination of phosphorus content - Bismuth molybdenum blue spectrophotometric method; - GB/T 24583.6 Vanadium-Nitrogen alloy - Determination of sulfur content - The infrared absorption method; - GB/T 24583.7 Vanadium-nitrogen - Determination of oxygen content - Infrared absorption method; - GB/T 24583.8 Vanadium-nitrogen - Determination of silicon, manganese, phosphorus, aluminum content - Inductively coupled plasma atomic emission spectrometric method. This Part is Part 2 of GB/T 24583. This Part is drafted in accordance with the rules given in GB/T 1.1-2009. This Part replaces GB/T 24583.2-2009 "Vanadium-Nitrogen alloy - Determination of nitrogen content - Thermal conductimetric method after fusion in a current of inert gas". Compared with GB/T 24583.2-2009, the main technical changes in this Part are as follows: - CHANGE the measuring range (mass fraction) to "8.00%~20.00%" (see Clause 1; Clause 1 of the 2009 edition); - Modify "Normative references" (see Clause 2; Clause 2 of the 2009 edition); - CHANGE the graphite crucible from high-temperature graphite crucible to suitable-for-instrument

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high-purity graphite crucible (see 4.12;

4.9 of the 2009 edition); Vanadium-nitrogen - Determination of nitrogen content - Thermal conductimetric method after fusion in a current of inert gas Caution - The personnel using this Part shall have hands-on experience in formal laboratory work. This Part does not address all possible safety problems. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions set by the relevant national regulations.

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1 Scope

China's national method for determining nitrogen in vanadium-nitrogen alloy by inert gas fusion with thermal conductivity detection. It is Part 2 of GB/T 24583 and specifies the determination of the nitrogen content of the alloy. Nitrogen is half the product. Vanadium-nitrogen alloy exists because vanadium alone is only half of the microalloying reaction - the precipitate that refines the grain and strengthens the steel is a carbonitride, and nitrogen is needed to form it - so the alloy is bought on the vanadium content and on the nitrogen content and on the ratio between them. A shipment low in nitrogen is not the product that was ordered even if the vanadium assay passes. Nitrogen in a solid is difficult to determine because it is not extractable by dissolution: it is present as a nitride, chemically bound, and any wet method has to liberate it first. Inert gas fusion avoids that entirely. The sample is dropped into a graphite crucible in a stream of helium and heated by a high current until it melts and the nitrogen is released as gas; the carrier stream carries it through the system, the carbon monoxide and other gases are removed, and the nitrogen is measured by the change in thermal conductivity of the stream, which is large because nitrogen and helium differ greatly in thermal conductivity. The result is fast, needs no reagents and no dissolution, and is what a works laboratory uses for routine control. What the standard fixes is the crucible and the flux, the temperature programme, the gas purification and flow, the calibration against certified reference materials, the blank, and the precision. Issued on 4 June 2019 and in force since 1 May 2020, it replaces GB/T 24583.2-2009.

This Part of GB/T 24583 specifies the determination of nitrogen content in vanadium-nitrogen alloy by thermal conductimetric method after fusion in a current of inert gas. This Part applies to the determination of nitrogen content in vanadium-nitrogen alloy. Measuring range (mass fraction): 8.00%~20.00%.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

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GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions

GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

GB/T 20567 Vanadium-nitrogen

5.2.1 The carrier gas system includes a helium container, a two-stage pressure regulator, and a sequential control part which ensures proper pressure and rated flow.

5.2.2 The power gas source includes power gas (nitrogen or compressed air), a two-stage pressure regulator, and a sequential control part which ensures proper pressure and rated flow.

5.3 High-temperature electrode furnace It shall meet the requirements for the fusion temperature of the test portion.

5.4 Control system Control functions include crucible loading-unloading, coil base lift, dust collector cleaning, analysis condition selection settings, analysis process monitoring and alarm interruption, analysis data acquisition, calculation, correction, and processing, etc.

5.5 Measurement system It is mainly composed of an electronic balance (sensitivity is

0.1 mg) controlled by a microprocessor, a thermal conductivity cell detector, and an electronic measuring element.

6 Sampling and sample preparation

In accordance with the provisions of GB/T 20567, the sample shall be taken and prepared. The sample shall all pass through the

0.125 mm sieve pore.

7 Analytical procedures

7.1 Number of determinations The same sample is determined at least twice independently.

7.2 Test portion WEIGH

0.05 g of sample, accurate to 0.0001 g.

7.3 Analysis preparation

7.3.1 According to the instruction manual of the instrument, debug and check the

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instrument (mainly air-tightness check), to keep the instrument in a normal stable state.

7.3.2 Choose to set the best analysis conditions.

7.3.3 USE the sample and flux to perform two test trials according to 7.6, to determine if the instrument is normal.

7.4 Blank test At least 3 blank tests are performed along with the test portion. The nitrogen blank value shall be less than 2 μg. Calculate the average. Refer to the instrument manual. INPUT the average as a blank to the azotometer. The instrument, when measuring the test portion, will automatically perform the electronic compensation of the blank value.

7.5 Calibration test According to the nitrogen content of the sample to be tested, establish a corresponding analysis curve; choose at least 2 standard samples (The nitrogen content range of standard sample shall cover the nitrogen content of the sample to be tested). The calibration and check shall be carried out in turn. The fluctuation of the determination result shall be within the range of reproducibility limit, to confirm the linearity of the system. Otherwise, the linearity of the system shall be adjusted according to the instrument manual.

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7.6 Determination

7.6.1 According to the type of graphite crucible used in the azotometer, one of the following combinations is selected as a flux:

a) Tin capsule (see 4.6)+nickel capsule (see 4.7);

b) Tin capsule (see 4.6)+nickel basket (see 4.8);

c) Nickel capsule (see 4.7)+

0.20 g of tin granule (see 4.5).

7.6.2 LOAD the test portion (see 7.2) into the selected tin capsule (see 4.6) or nickel capsule (see 4.7); ADD another flux selected in 7.6.1; carefully SQUEEZE and wrap; PLACE in the instrument's feeding device. PLACE the graphite crucible (see 4.12) on the crucible holder; operate according to the instrument manual; determine and read the result.

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