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Rare Earth Standard of the People's Republic of China

XB/T 617.7-2014

Chemical analysis methods for neodymium iron boron alloy—Part 7: Determination of oxygen and nitrogen contents-Impulse-infrared and impulse-thermal conductance absorption method

钕铁硼合金化学分析方法 第7部分：氧、氮量的测定 脉冲—红外吸收法和脉冲—热导法

(English Translation)

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Foreword

SAC/TC 229 China Rare Earth Standardization Technical Committee is in charge of this English translation. In case of any doubt about the contents of the English translation, the Chinese original shall be considered authoritative.

The XB/T 617 *Chemical analysis methods for neodymium iron boron alloy* consists of the following seven parts:

——*Part 1: Determination of total rare earth content—Oxalate gravimetry;*

——*Part 2: Determination of fifteen REO relative contents;*

——*Part 3: Determination of boron, aluminum, copper, cobalt, magnesium, silicon, calcium, vanadium, chromium, manganese, nickel, zinc and gallium content—Inductively coupled plasma atomic emission spectrometry;*

——*Part 4: Determination of iron content—The potassium dichromate titrimetry;*

——*Part 5: Determination of zirconium, niobium, molybdenum, tungsten and titanium content—Inductively coupled plasma atomic emission spectrometry;*

——*Part 6: Determination of carbon content—High frequency-infrared absorption method;*

——*Part 7: Determination of oxygen and nitrogen contents—Impulse-infrared and impulse-thermal conductance absorption method.*

This part is the 7th part of XB/T 617.

This standard is drafted in accordance with the rules given in the GB/T 1.1-2009.

This standard was prepared by the SAC/TC 229.

Chemical analysis methods for neodymium iron boron alloy—Part 7: Determination of oxygen and nitrogen contents- Impulse-infrared and impulse-thermal conductance absorption method

1 Scope

This part specifies a method for the determination of oxygen and nitrogen contents in neodymium iron boron alloys.

This part is applicable to the determination of oxygen and nitrogen contents in neodymium iron boron alloys, the determination range of oxygen is 0.0020%~0.60%, nitrogen is 0.0020%~0.10%.

2 Principle

In the inert gas atmosphere, the sample in the graphite crucible is heated to a molten state, the oxygen is converted to carbon monoxide and a small amount of carbon dioxide, the carbon monoxide will be oxidized to carbon dioxide by reduction of copper oxide. Measure the carbon dioxide which entered the infrared detector. Nitrogen is emitted as N_2, measure N_2 which entered the thermal conductivity detector.

3 Reagent and Material

3.1 Carbon tetrachloride

3.2 High-purity nickel basket or foil [ω(Ni)≥99%, ω(O)≤0.0020%, ω(N)≤0.0005%], Pre-treated with mixed acid (HNO_3+HPO_4+HAc) to form a corrosion layer.

3.3 Graphite crucible

3.4 Reference material: Select reference material or other applicable reference material of which the main components and the oxygen content, the nitrogen content of the sample are similar to the test sample.

3.5 Helium (φ(He)≥99.99%)

4 Apparatus

Oxygen/Nitrogen analyzer: the sensitivity shall be no less than 0.1 μg/g.

5 Sample

Sample preparation from blocks shall be taken from the core. Clean the sample with carbon tetrachloride (3.1) and air-dry it, and then place it in a nickel foil (3.2) for later use;Sample preparation from powders or fragments shall be wrapped with nickel foil (3.2), which needs to be cleaned and air-dried with carbon tetrachloride

(3.1)before use.

6 Procedure

6.1 Test portion

Weigh 0.05g~0.10g sample to the nearest 0.001g

6.2 Parallel test

Weigh two test portions for parallel determination, calculate the mean value.

6.3 Apparatus preparation

Check the various reagents and materials with the function of purification, dust removal in the instrument to ensure they are available. Turn on the instrument, preheat and carry out the system check according to the requirements of the operating instructions.

6.4 Blank calibration

Turn on the impulse furnace and place the graphite crucible (3.3) on the lower electrode. Place the nickel basket or foil (3.2) in the sample drop block and heat to a molten state after the lower electrode rising and the crucible degassing. At last, the instrument shows a blank value. Repeat former operation determination of the nickel basket or foil 3~5 times. The next step can be made only if the average blank value of oxygen is no more than 0.0020% and the average blank value of nitrogen is no more than 0.0005%.

6.5 Calibration procedure

Take three reference materials (3.4), measure and calibrate according to 6.6 and repeat once. The fluctuation of the measurement result should be within the allowable range of the standard value.

6.6 Determination

Input sample mass value and blank value, put the sample in a nickel flux or nickel foil (3.2), and then place them in the sample loading , turn on the pulse furnace and place the graphite crucible (3.3) on the lower electrode. Close the lower electrode, degas the crucible, put the sample into the crucible and heat to melt, the gas released in the crucible, the measured value shall be displayed in mass fraction.

7 Expression of results

The oxygen and nitrogen contents, expressed as mass fraction ,are calculated by formula(1), %

$$\omega = \omega_1 - \omega_0 \quad \cdots\cdots (1)$$

where,

ω_1——Mass fraction of oxygen or nitrogen in the fluxing agent and sample, %

ω_0——Mass fraction of oxygen or nitrogen in the fluxing agent, %

8 Precision

8.1 Repeatability

The absolute difference, of the determined values obtained from two independent determinations under the repeatability conditions, is not greater than the repeatability limit (r), which is in the range of the following average values. The cases that the absolute difference is greater than the repeatability limits (r) are less than 5%. The repeatability limit (r) is calculated by linear interpolation method on the basis of the data listed in Table 1..

Table 1

Element	Mass fraction/%	Repeatability limit (r) / %
Oxygen	0.012	0.005
	0.23	0.02
	0.37	0.03
Nitrogen	0.0022	0.0009
	0.012	0.003
Note: The repeatability limit (r) is 2.8×Sr , Sr is the repetitive standard deviation		

8.2 Tolerance

The difference of the analytical results among laboratories shall be no more than the tolerances listed in Table 2.

Table 2

Element	Mass fraction/%	Tolerance/%
Oxygen	0.0020~0.010	0.0012
	>0.010~0.030	0.006
	>0.030~0.10	0.012
	>0.10~0.30	0.03
	>0.30~0.60	0.05
Nitrogen	0.0020~0.0050	0.0015
	>0.0050~0.010	0.0030
	>0.010~0.030	0.005
	>0.030~0.10	0.010

9 Quality guarantee and control

Verify the validity of the method by reference materials or controlling sample each week. Check it at least once a month with the standard sample or controlling sample. Try to find out the cause in case of abnormalities and correct the mistakes then check again.