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Methods for chemical analysis of aluminium and aluminium alloys - Part 30: Determination of hydrogen content - Heating extraction-thermal conductivity method

铝及铝合金化学分析方法

第 30 部分：氢含量的测定 加热提取热导法

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Foreword

GB/T 20975, Methods for chemical analysis of aluminium and aluminium alloys, is divided into 31 parts:

-- Part 1: Determination of mercury content;

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-- Part 3: Determination of copper content;

-- Part 4: Determination of iron content - Orthopenanthroline photometric method;

-- Part 5: Determination of silicon content;

-- Part 6: Determination of cadmium content - Flame atomic absorption spectrometric method;

-- Part 7: Potassium periodate spectrophotometric method;

-- Part 8: Determination of zinc content;

-- Part 9: Determination of lithium content - Flame atomic absorption spectrometric method;

-- Part 10: Determination of tin content;

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-- Part 15: Determination of boron content;

-- Part 16: Determination of magnesium;

-- Part 17: Determination of strontium content - Flame atomic absorption spectrometric method;

-- Part 18: Determination of chromium content;

-- Part 19: Determination of zirconium content;

Methods for chemical analysis of aluminium and aluminium alloys - Part 30: Determination of hydrogen content - Heating extraction-thermal conductivity method

Caution -- The personnel who uses this Part shall have hands-on experience in formal laboratory work. This Part does not address all possible security issues. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions which are set by the relevant national regulations.

1 Scope

This Part of GB/T 20975 specifies the method for the determination of hydrogen content in solid aluminum and aluminum alloys by heating extraction-thermal conductivity method.

This Part applies to the determination of hydrogen content in solid aluminum and aluminum alloys. Determination range: 0.05 μg/g ~ 1.0 μg/g.

Note: This method only performs inter-laboratory test on the specified measurement range. However, the laboratory can extend the measurement range of this method through experiments such as method sensitivity, precision and bias in method validation.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 8170-2008, Rules of rounding off for numerical values & expression and judgement of limiting values

3 Method summary

Place the sample in a high-purity graphite crucible; heat to just below the melting point to drive off the surface hydrogen; then, continue to heat under a

Weigh a certain mass of the sample (6.1); accurate to 0.001 g. The sample mass for pulse heating method should be 1 g ~ 4 g; the sample mass for high-frequency heating method should be 2 g ~ 6 g.

6.3 Sample number

Two samples are prepared in parallel for each sample.

6.4 Sample size

The sample size should be determined according to the requirements of the instrument and the test conditions.

6.5 Sample preparation

6.5.1 Rough machining

Use the precision lathe to remove all the coats of the sample and process it into a cylindrical shape; the size of the sample after processing shall meet the requirements of 6.4; put it into the sample bag; write the sample identification and sampling date; store it in a closed and dry container.

Note: in order to meet the machining allowance of the sample, the diameter of the sample after rough machining shall be more than 1 mm, and the length shall be more than 5 times of the requirements.

6.5.2 Fine machining

The sample after rough machining will be fine-machined on the day of inspection (the laboratory should have a special finishing machine for hydrogen measurement). Before the usage of the lathe, the cutters and clamps should be cleaned with diethyl ether or carbon tetrachloride (5.5). In order to avoid overheating of the sample during processing, the tool amount and the speed shall be controlled. If necessary, use absolute ethanol or acetone (5.6) for cooling. The processed sample shall be taken with a clean tweezers; it is not allowed to touch by hand to prevent contamination. The surface roughness Ra shall be no more than 1.6 μm.

6.6 Sample placement

6.6.1 The fine-machined sample (6.5.2) should be tested immediately.

6.6.2 When the sample cannot be tested immediately, it should be protected by appropriate methods. The sample can be placed in diethyl ether or carbon tetrachloride (5.5) for a period of not more than 4 hours. Otherwise, the sample shall be reworked.

Under the same test conditions as the sample, use aluminum alloy standard sample or quality control sample (5.1) that is approved by both parties to calibrate the working standard, according to the instrument analysis software.

7.6 Sample test

7.6.1 Use the quality control sample to verify the calibrated working curve.

7.6.2 Test the sample according to the instrument analysis software.

7.6.3 After the test of the sample, observe the state of the melt sample; it's appropriate to be spherical and pourable.

7.6.4 Each sample (6.3) is tested independently.

7.7 Recalibration of the working curve

When any of the following conditions occurs during the test, 7.5 should be repeated.

a) large fluctuations of the instrument;

b) the test interval of the sample exceeds 2h;

c) replace samples with large differences in composition;

d) replace the carrier gas;

e) make analytical parameter adjustments (not recommended for daily adjustments);

f) replace pot furnace of different batches;

g) other abnormalities occur.

8 Result calculation

8.1 The instrument automatically displays the test results. The test results are expressed to three decimal places; the rounding-off value is according to 3.2, 3.3 of GB/T 8170-2008.

8.2 Take the average of the two test results. If the two test results are not within the repeatability limit, it should be retested.

9 Precision

9.1 Repeatability

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