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Methods for chemical analysis of aluminium and aluminium alloys - Part 27: Determination of cerium, lanthanum, scandium - Inductively coupled plasma atomic emission spectrometry method

铝及铝合金化学分析方法

第 27 部分：铈、镧、钪含量的测定

电感耦合等离子体原子发射光谱法

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Foreword

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

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- Part 5: Determination of silicon content;
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- Part 18: Determination of chromium content;

Methods for chemical analysis of aluminium and aluminium alloys - Part 27: Determination of cerium, lanthanum, scandium - Inductively coupled plasma atomic emission spectrometry method

1 Scope

This Part of GB/T 20975 specifies the method for determination of cerium, lanthanum, scandium content in aluminium and aluminium alloys.

This Part is applicable to the determination of cerium, lanthanum, and scandium content in aluminium and aluminium alloys. The aluminium in aluminium and aluminium alloys interferes with the determination of cerium, lanthanum, and scandium. The same amount of aluminium is added to the working curve to eliminate the interference. Cerium, lanthanum, and scandium do not interfere with each other, and other coexisting elements do not interfere with the determination. Measurement range: cerium: 0.005%~0.90%; lanthanum: 0.005%~0.50%; scandium: 0.0002%~0.50%.

2 Method principle

After the sample is dissolved, in dilute hydrochloric acid medium, using inductively coupled plasma atomic emission spectrometer (ICP-AES), the content of cerium, lanthanum, and scandium elements is determined. By matrix matching method, the influence of the matrix on the measurement results is corrected.

3 Reagents and materials

Unless otherwise stated, in the analysis, only Grade 2 water is used.

3.1 Hydrogen peroxide (ρ=1.10 g/mL).

3.2 Hydrochloric acid (ρ=1.19 g/mL), analytically pure.

3.3 Nitric acid (ρ=1.42 g/mL), analytically pure.

3.4 Argon (>99.99%).

3.5 Hydrochloric acid (1+1).

3.6 Sodium hydroxide (200 g/L).

3.7 Aluminium [w(Al)≥99.99%], before use, dip-rinse with a small amount of acid; then WASH off the acid with water, RINSE 2~3 times with absolute ethyl alcohol, and air dry.

3.8 Aluminium matrix solution (20 mg/mL): WEIGH 10.00 g of aluminium (3.7) into a 500 mL beaker; COVER the watch glass; and ADD a total of 300 mL of hydrochloric acid (3.5) in portions. After the violent reaction is stopped, ADD a few drops of hydrogen peroxide (3.1); slowly HEAT until completely dissolved; then BOIL for a few minutes, COOL; and TRANSFER the solution to a 500 mL volumetric flask; USE water to dilute to volume, and MIX well.

3.9 Standard stock solution of cerium (1 mg/mL): WEIGH 0.3071 g of cerium oxide [w(CeO_2)≥99.99%, pre-burned at 1000 °C for 1 h, and cooled in a desiccator] in a 100 mL beaker; ADD 15 mL of nitric acid (3.3). PLACE for a while and HEAT to boil; then ADD 1 mL of hydrogen peroxide (3.1); HEAT at low temperature for 4 times~5 times until complete dissolution; HEAT to boil, and COOL. TRANSFER to a 250 mL volumetric flask; USE water to dilute to volume, and MIX well.

3.10 Standard stock solution of lanthanum (1 mg/mL): WEIGH 0.2932 g of lanthanum trioxide [w(La_2O_3)≥99.99%, pre-burned at 1000 °C for 1 h, and cooled in a desiccator] in a 100 mL beaker; ADD 50 mL of hydrochloric acid (3.5); HEAT until complete dissolution, and COOL. TRANSFER to a 250 mL volumetric flask; USE water to dilute to volume, and MIX well.

3.11 Standard stock solution of scandium (1 mg/mL): WEIGH 0.3835 g of scandium oxide [w(Sc_2O_3)≥99.99%, pre-burned at 800 °C for 1 h, and cooled in a desiccator] into a 100 mL beaker; ADD 25 mL of nitric acid (3.3); dropwise ADD hydrogen peroxide (3.1) until completely dissolved. Then BOIL for a few minutes, COOL; and TRANSFER to a 250 mL volumetric flask; ADD 25 mL of nitric acid (3.3); USE water to dilute to volume, and MIX well.

3.12 Cerium standard solution: PIPETTE 10.00 mL of standard stock solution of cerium (3.9) into a 100 mL volumetric flask; USE water to dilute to volume, and MIX well, to obtain cerium standard solution A (100 μg/mL). By stepwise dilution in the same manner, cerium standard solution B (10 μg/mL) can be obtained.

3.13 Lanthanum standard solution: PIPETTE 10.00 mL of standard stock solution of lanthanum (3.10) into a 100 mL volumetric flask; USE water to dilute to volume, and MIX well, to obtain lanthanum standard solution A (100 μg/mL).

6.3 Blank test

WEIGH the same amount of aluminium (3.7) as the test portion; and PERFORM a blank test along with the test portion.

6.4 Determination

6.4.1 When the silicon's mass fraction in the sample is ≤2%, PLACE the test portion (6.1) in a 100 mL beaker; ADD 10 mL of hydrochloric acid (3.5), and COVER the watch glass. After the reaction is stopped, ADD a few drops of hydrogen peroxide (3.1); slowly HEAT until completely dissolved; and then BOIL for 3 min~5 min, COOL. TRANSFER the solution to a 100 mL volumetric flask; USE water to dilute to volume, and MIX well. The test solution is introduced into a plasma atomic emission spectrometer for determination.

6.4.2 When the silicon's mass fraction in the sample is >2%, PLACE the test portion (6.1) in a 100 mL polytetrafluoroethylene beaker; ADD 10 mL of sodium hydroxide solution (3.6), and COVER the watch glass. After the reaction is completed, COOL to room temperature; ADD 15 mL of hydrochloric acid (3.5), and COOL. TRANSFER the solution to a 100 mL volumetric flask; USE water to dilute to volume, and MIX well. The test solution is introduced into a plasma atomic emission spectrometer for determination.

When the linear correlation coefficient of working curve is ≥0.99, according to the relationship between the spectral intensity and the concentration, the concentration of cerium, lanthanum, and scandium elements in the sample is automatically given by the computer.

6.5 Drawing of working curve

6.5.1 When the mass fraction of cerium, lanthanum, scandium is ≤0.10%: respectively PIPETTE 0 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL, 10.00 mL of cerium, lanthanum, scandium standard solution B (3.12~3.14) into a set of 100 mL volumetric flasks; ADD 5 mL of aluminium matrix solution (3.8), 5 mL of hydrochloric acid (3.5); USE water to dilute to volume, and MIX well.

6.5.2 When the mass fraction of cerium, lanthanum, scandium is >0.10%: respectively PIPETTE 0 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL, 10.00 mL of cerium, lanthanum, scandium standard solution A (3.12~3.14) into a set of 100 mL volumetric flasks; ADD 5 mL of aluminium matrix solution (3.8), 5 mL of hydrochloric acid (3.5); USE water to dilute to volume, and MIX well.

6.5.3 Appropriate analytical conditions are selected for the determination. With the concentration of Ce, La, and Sc as the abscissa, and the corresponding spectral intensity as the ordinate, the standard curve is drawn (automatically drawn by computer).

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