Current Status of Standards for Testing Methods of Aluminium Carbon Materials in China

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Abstract

A comprehensive analysis on the development of the standards for testing methods of carbon materials in aluminium electrolysis is provided, and standards established and revised in recent years are introduced in detail. The revision of standards follows the following principles: adopting the latest international standards to align with international standards; expanding testing scopes or adding new testing methods to improve testing efficiency and meet the demands of intelligent control; and integrating and optimizing similar testing methods for ease of use and better alignment with domestic practical conditions in China. The revised standards will further refine China's analysis and testing standard system for carbon materials in aluminium electrolysis, providing technical support for the development of China's aluminium industry.

Keywords: Carbon materials in aluminium electrolysis, Testing, Standards, Current status.

1. Background and Significance of Standards

With the rapid development of China's aluminium industry, carbon materials in aluminium electrolysis have also made significant progress. Since 2002, China has become the world's largest producer and one of the major exporters of carbon materials in aluminium electrolysis, fostering numerous independent enterprises specializing in export-oriented production of carbon materials in aluminium electrolysis. In view of increasingly widespread use of large 600 kA pre-baked cells, further advancement of smelting techniques, fierce competition in the domestic market, complete integration with the international market, and stricter requirements for energy conservation and consumption reduction, carbon materials, as the main raw materials in aluminium electrolysis, whether anode or cathode materials, have received increasing attention in terms of quality [1, 2]. In recent years, domestic production of pre-baked anodes has reached over 22 million tonnes, and production of cathode carbon blocks has reached 350 000 tonnes. In the aluminium industry standard system, the *Carbonaceous Materials Used in the Production of Aluminium* (YS/T 63) series plays a vital role in ensuring the quality of carbon materials in aluminium electrolysis, together with sampling methods of *Sampling of Carbonaceous Materials Used for Aluminium Production - Part 1: Cathode Blocks* (GB/T 26297.3), *Sampling of Carbonaceous Materials Used for Aluminium Production - Part 2: Sidewall Blocks* (GB/T 26297.1), *Sampling of Carbonaceous Materials Used for Aluminium Production - Part 3: Prebaked Anodes* (GB/T 26297.3) and *Sampling of Carbonaceous Materials Used for Aluminium Production - Part 4: Cathodic Pastes* (GB/T 26297.4), used in conjunction with product standards for carbon materials in aluminium electrolysis such as: *Prebaked Anode for Aluminium Electrolysis* (YS/T 285), *Graphitic Cathode Carbon Block for Aluminium Electrolysis* (YS/T 623), *Graphitized Cathode Carbon Block for Aluminium Electrolysis* (YS/T 699), and *Cathodic Pastes for Aluminium Electrolysis* (YS/T 65). *Carbonaceous Materials Used in the Production of Aluminium* (YS/T 63) series covers the determination of indicators such as electrical resistivity at room temperature, thermal expansion coefficient, true density, compressive strength, microelements, volatile, and ash content. This series of standards serves fields such as the production of carbon materials in aluminium electrolysis, trade settlement, analysis and comparison, and electrolytic aluminium production,

providing technical support for the high-quality development of China's carbon materials in aluminium electrolysis.

2. Development of Standards

2.1 Initial Stage

Before 2000, as standards for the analysis and testing of carbon materials were largely used across the non-ferrous, ferrous and other industries, administered by the Ministry of Metallurgical Industry, it was impossible to take into full account the special requirements of carbon products in aluminium electrolysis during establishment of standards. As a result, some standards were not properly applicable to the analysis and testing of carbon products in aluminium electrolysis [3].

2.2 Development Stage

In order to adapt to the rapid development of the aluminium industry and fully integrate with the international markets, it was necessary to attach importance to the establishment and revision of standards for various parts of carbon products in aluminium electrolysis. With strong support from national ministries and commissions, standardization experts consolidated industry expertise and continuously established a comprehensive standard system for carbon products in aluminium electrolysis, including a series of 27 standards for testing methods of carbon materials in aluminium electrolysis, as shown in Table 1. Most of modified standards adopted ISO standards or equivalents. This series features coordination, complementarity, applicability and advancement, basically meeting the needs at that time [4].

Table 1. Standards in the development stage.

Standard No.	Standard Title	Standard Code
YS/T 63.1-2006	*Preparation of Roasted Test Pieces, Determination of Loss on Roasting and Apparent Density after Ramming*	ISO14427-2004 and ISO 20202-2004
YS/T 63.2-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 2: Cathode Blocks and Prebaked Anodes - Determination of Electrical Resistivity at Ambient Temperature*	ISO 11713-2000
YS/T 63.3-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 3: Determination of the Thermal Conductivity Using a Comparative Method*	ISO 12987-2004
YS/T 63.4-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 4: Determination of the Thermal Expansion Coefficient*	ISO 14420-2005
YS/T 63.5-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 5: Cathode Blocks - Determination of Expansion due to Sodium Penetration with Application of Pressure*	ISO 15379-1: 2004
YS/T 63.6-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 6: Determination of Open Porosity Using a Hydrostatic Method*	ISO 12985-2: 2000
YS/T 63.7-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 7: Determination of Apparent Density Using a Dimensions Method*	ISO 12985-1: 2000
YS/T 63.8-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 8: Determination of the Density in Xylene by a Pyknometric Method*	ISO 9088:1997
YS/T 63.9-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 9: Determination of Truth Density by Helium Pyknometry Method*	ISO/CD 21687

YS/T 63.10-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 10: Determination of the Air Permeability*	ISO/DIS 15906:2005
YS/T 63.11-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 11: Determination of the Reactivity to Air - Loss in Mass Method*	ISO 12989-1: 2000
YS/T 63.12-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 12: Prebaked Anodes - Determination of the Reactivity to Carbon Dioxide - Loss in Mass Method*	ISO 12988-1: 2000
YS/T 63.13-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 13: Determination of Young's Modulus*	
YS/T 63.14-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 14: Determination of Bending/Shear Strength by a Three-Point Method*	ISO 12986-1: 2000
YS/T 63.15-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 15: Determination of Compressive Strength*	ISO/CD 18515
YS/T 63.16-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 16: Analysis Using an X-ray Fluorescence Method*	ISO 12980-1: 2000
YS/T 63.17-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 17: Determination of Volatile Matter Content*	ISO/TS 14425: 1999
YS/T 63.18-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 18: Determination of Water Content*	ISO 11412: 1997
YS/T 63.19-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 19: Determination of Ash Content*	ISO 8005: 1984
YS/T 63.20-2006	*Carbonaceous Materials Used in the Production of Aluminium - Part 20: Determination of Sulfur Content*	ISO 5931: 2000
YS/T 63.21-2007	*Carbonaceous Materials Used in the Production of Aluminium - Part 21: Cathodic Pastes - Determination of Expansion/Shrinkage during Baking*	ISO 14428: 2004
YS/T 63.22-2009	*Carbonaceous Materials Used in the Production of Aluminium - Part 22: Determination of Baking Level Expressed by Equivalent Temperature*	ISO 17499: 2006
YS/T 63.23-2012	*Carbonaceous Materials Used in the Production of Aluminium - Part 23: Prebaked Anodes - Determination of the Reactivity to Air - Thermogravimetric Method*	ISO 12989-2: 2004
YS/T 63.24-2012	*Carbonaceous Materials Used in the Production of Aluminium - Part 24: Prebaked Anodes - Determination of the Reactivity to Carbon Dioxide - Thermogravimetric Method*	ISO 12988-2: 2004
YS/T 63.25-2012	*Carbonaceous Materials Used in the Production of Aluminium - Part 25: Cathode Blocks - Determination of Expansion due to Sodium Penetration without Application of Pressure*	ISO 15379-2: 2004
YS/T 63.26-2012	*Carbonaceous Materials Used in the Production of Aluminium - Part 26: Dense Refractory Bricks - Determination of Cryolite Resistance*	ISO 20292: 2009
YS/T 63.27-2015	*Carbonaceous Materials Used in the Production of Aluminium - Part 27: Determination of the Fracture Energy for Prebaked Anodes*	ISO 11706: 2012

2.2 Improvement Stage

In recent years, with the promotion and application of *Carbonaceous Materials Used in the Production of Aluminium* series as modified by adopting ISO international standards, some problems have also been discovered. As ISO international standards are used globally and are

required to meet the technical requirements of various countries, they may not represent the most advanced methodologies. Some methods only outline general principles, lacking specific operable guidelines. With the rapid development of China's aluminium industry, the domestic production processes and product ranges of some carbon materials in aluminium electrolysis have changed, requiring modification or improvement of determination scope, technical parameters and other indicators in the standards. The demand for new testing items in China, widespread application of new determination methods and continuous update of ISO standards have necessitated additions and revisions to this series of standards. The new versions of the standards have been released successively, as shown in Table 2.

Table 2. Release of new versions of standards.

Standard No.	Standard Title	Date of Standard Implementation
YS/T 63.2-2023	*Carbonaceous Materials Used in the Production of Aluminium - Part 2: Determination of Electrical Resistivity at Ambient Temperature*	November 1, 2023
YS/T 63.4-2023	*Carbonaceous Materials Used in the Production of Aluminium - Part 4: Determination of the Thermal Expansion Coefficient*	November 1, 2023
YS/T 63.7-2024	*Carbonaceous Materials Used in the Production of Aluminium - Part 7: Determination of Apparent Density Using a Dimensions Method*	May 1, 2025
YS/T 63.8-2023	*Carbonaceous Materials Used in the Production of Aluminium - Part 8: Determination of Truth Density - Pyknometric Method*	November 1, 2023
YS/T 63.11-2024	*Carbonaceous Materials Used in the Production of Aluminium - Part 11: Determination of the Reactivity to Air*	May 1, 2025
YS/T 63.12-2024	*Carbonaceous Materials Used in the Production of Aluminium - Part 12: Determination of the Reactivity to Carbon Dioxide for Prebaked Anodes*	May 1, 2025
YS/T 63.14-2023	*Carbonaceous Materials Used in the Production of Aluminium - Part 14: Determination of Bending/Shear Strength by a Three-Point Method*	November 1, 2023
YS/T 63.15-2023	*Carbonaceous Materials Used in the Production of Aluminium - Part 15: Determination of Compressive Strength*	November 1, 2023
YS/T 63.20-2023	*Carbonaceous Materials Used in the Production of Aluminium - Part 20: Determination of Sulfur Content*	November 1, 2023
YS/T 63.27-2024	*Carbonaceous Materials Used in the Production of Aluminium - Part 27: Determination of the Fracture Energy for Prebaked Anodes*	May 1, 2025

3. Overview of Major Standard Additions and Revisions

3.1 Determination of Electrical Resistivity at Ambient Temperature (YS/T 63.2-2023)

This standard applies to all carbon materials in aluminium electrolysis, including cathode carbon blocks, sidewall carbon blocks and pre-baked anodes and cathode pastes; the precision of the vernier callipers is better than 0.02 mm, aligning with the precision of widely available vernier callipers for ease of procurement and use by personnel; sampling provisions, sample sizes, and distance between probes and precision requirements for sidewall carbon blocks, hot ramming pastes and cold ramming pastes are clearly defined, more suitable for the current testing status with requirements for electrical resistivity in the cathode paste product standards and requirement of electrical resistivity testing for sidewall carbon blocks; in order to meet the demands of automated and intelligent testing, with requirements for the on-site testing methods of the

resistivity of the entire block during the enterprise's production control, Appendix A On-site Testing Methods of Resistivity of the Entire Block is added, laying a solid foundation for further intelligent testing.

3.2 Determination of the Thermal Expansion Coefficient (YS/T 63.4-2023)

The temperature ranging from 20 ℃ to 300 ℃ is modified to from room temperature to 300 ℃, in which room temperature serves as the starting temperature for final calculations, and any temperature within the room temperature range is acceptable, with no strict requirement for laboratory temperature; a calibration sample "borosilicate glass with thermal expansion coefficient of 3.3×10^{-6}/K" is added: since quartz has a thermal expansion coefficient one order of magnitude lower than that of carbon materials in aluminium electrolysis, the addition of borosilicate glass - whose properties align more closely with carbon materials in aluminium electrolysis - ensures better calibration of equipment; the rounding rules for calculated results is changed from rounding to two decimal places to one decimal place: since single decimal place precision can meet the requirements for product quality control by manufacturing enterprises and users, the relevant product standards provide the requirement of rounding to one decimal place.

3.3 Carbonaceous Materials Used in the Production of Aluminium - Part 8: Determination of Truth Density - Pyknometric Method (YS/T 63.8-2023)

Currently, domestic enterprises mainly employ the water boiling method instead of xylene method for determining the true density of carbon materials in aluminium electrolysis: this method utilizes distilled water as the solvent, eliminating operational safety concerns, and uses pycnometer as the testing apparatus, which is cost-effective and allows simultaneous test of dozens of samples, significantly improving the testing efficiency, reducing the labour intensity of the testing personnel, and lowering the risk of occupational diseases. The standard title is revised to *Carbonaceous Materials Used in the Production of Aluminium - Part 8: Determination of Truth Density - Pyknometric Method*, more suitable for the current testing status.

3.4 Carbonaceous Materials Used in the Production of Aluminium - Part 11: Determination of the Reactivity to Air (YS/T 63.11-2024)

The requirements for thermocouples are changed: currently, Type K or N thermocouples are widely used and capable of meeting standard requirements; the parameter of reactivity to air is changed: the parameter descriptions are modified to align with industry practices; calculation formula for the reactivity is changed: original formula occasionally resulted in sum failure to reach 100 % due to rounding errors, and the revised formula eliminates this issue; The thermogravimetric method for testing reactivity to air is added, integrating the content of *Carbonaceous Materials Used in the Production of Aluminium - Part 23: Determination of the Reactivity to Air - Thermogravimetric Method* (YS/T 63.23-2012), enhancing the applicability.

3.5 Determination of the Reactivity to Carbon Dioxide for Prebaked Anodes (YS/T 63.12-2024)

The requirements for reagents are changed: currently, 99.99 % CO_2 gas is widely used, and tightening CO_2 gas purity is more conducive to the authenticity and comparability of test data; The requirements for thermocouples are changed: currently, Type K or N thermocouples are widely used and capable of meeting standard requirements; the temperature for sample taking is changed: at 550 ℃, the sample taken will react with the air, affecting the authenticity of the test results, while below 300 ℃, the sample will not react with the air; the parameter of reactivity to CO_2 is changed: the parameter descriptions are modified to align with industry practices; calculation formula for the reactivity is changed: original formula occasionally resulted in sum

failure to reach 100 % due to rounding errors, and the revised formula eliminates this issue; the thermogravimetric method for testing reactivity to CO_2 is added, integrating the content of *Carbonaceous Materials Used in the Production of Aluminium - Part 24: Baked Anodes - Determination of the Reactivity to Carbon Dioxide - Thermogravimetric Method* (YS/T 63.24-2012), enhancing the applicability.

3.6 Carbonaceous Materials Used in the Production of Aluminium - Part 14: Determination of Bending/Shear Strength by a Three-Point Method (YS/T 63.14-2023)

The scope is expanded to include carbon slabs, carbon bricks and other carbon materials, broadening the applicability to meet the needs of the industry; the precision of the vernier callipers is modified from ± 0.5 % to better than 0.02 mm, aligning with the precision of widely available vernier callipers for ease of procurement and use by personnel; an oven is added: the temperature can be controlled at 110 ℃ ± 5 ℃; The sample preparation part is added, the dimensional requirements after sample processing is added, and the calculation formula for prismatic samples is added to meet the needs of some enterprises for prismatic sample testing; the rounding rules for calculated results is changed from rounding to two decimal places to one decimal place: since single decimal place precision can meet the requirements for product quality control by manufacturing enterprises and users, the relevant product standards provide the requirement of rounding to one decimal place or an integer.

3.7 Carbonaceous Materials Used in the Production of Aluminium - Part 15: Determination of Compressive Strength (YS/T 63.15-2023)

The scope is expanded to include products with applicable compressive strength: carbon slabs, carbon bricks and other carbon materials, broadening the applicability to meet the needs of the industry; the specimen size of the cube with a processing side length of 45.0 ± 0.2 mm is added to meet the needs of some enterprises for testing of cube samples; in addition to the existing requirement for loading rate of "0.5 N/mm^2 per second" in a destructive test, in combination with the performance of the actual electronic tester, the equivalent rate of "5 mm/min" is added.

3.8 Carbonaceous Materials Used in the Production of Aluminium - Part 20: Determination of Sulfur Content (YS/T 63.20-2023)

The measurement range is modified from ≥ 0.15 % to ≥ 0.10 %: although currently, the sulphur content of some carbon products is very low, this method can meet the accuracy requirements for low-content detection, and expanding the range can meet the detection requirements; the principle, equipment, determination method and precision of the combustion-infrared absorption method are added. In the current standards, as a traditional chemical gravimetric method, the Eschka reagent method is complicated and time-consuming (one or two days for each sample) to operate, posing high requirements for personnel, which is not conducive to the efficiency of the testing. With the current status of the promotion of intelligent manufacturing in the carbon industry, it is necessary to include equipment that can be intelligently controlled. For this purpose, combustion-infrared absorption method is widely used at present, under which the sample is placed in a resistance furnace or high-frequency furnace for combustion at high temperature under an oxygen flow, and after sulphur is oxidized into sulphur dioxide gas, an infrared analyser is used to determine the production of sulphur dioxide, followed by calculation of sulphur content in the sample. This method is simple to operate and takes a short time (one or two minutes), significantly enhancing work efficiency. The newly added method can meet the current requirements for testing and intelligent control of carbon materials in aluminium electrolysis in China.

4. Significance of Standard Implementation

With respect to the revisions to this standard series, modifications of *Determination of the Thermal Expansion Coefficient*, *Determination of Bending Strength by a Three-Point Method* and *Determination of Compressive Strength* adopt latest ISO standards, ensuring the industry standards in line with international standards, and are based on the actual conditions in China to better serve domestic and foreign users; *Determination of Electrical Resistivity at Ambient Temperature*, *Determination of Truth Density - Pyknometric Method* and *Determination of Sulfur Content* improve the testing efficiency, reduce the use of organic reagents and meet the needs of intelligent control by expanding the testing range or inclusion of new testing methods in combination with the actual situation and demands of the industry; *Determination of the Reactivity to Air* and *Determination of the Reactivity to Carbon Dioxide for Prebaked Anodes* integrate and optimize similar testing methods for applicability, thus aligning with actual conditions in China. The release and implementation of relevant standards will provide technical basis for the quality control and product acceptance of carbon materials in aluminium electrolysis, for the enterprises involved in production and use of carbon materials in aluminium electrolysis, conducive to improving the quality of analysis and testing personnel, and standardizing product quality control in the industry, meeting the current requirements for the testing and quality control of carbon materials in aluminium electrolysis in China. The revised standards will further refine China's analysis and testing standard system for carbon materials in aluminium electrolysis, and facilitate standardization of production quality control and trade of aluminium industry in China, providing technical support for the development of the China's aluminium industry.

5. References

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