NATIONAL STANDARD OF
THE PEOPLE'S REPUBLIC OF CHINA

中华人民共和国国家标准

GB/T 2524—2019

(replacing GB/T 2524—2010)

Titanium sponge

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1—2009.

This standard replaces GB/T 2524—2010 "Titanium sponge". Compared with GB/T 2524—2010, apart from editorial changes, the main technical changes are as follows:

——the chemical composition indexes of some grades were modified (see Table 1, and Table 1 of the 2010 edition);

——the requirements for Ni, Cr and "the total of other impurity elements" were added (see Table 1);

——the requirements for the particle size specifications of the products were modified (see 3.3.1, and 3.3.1 of the 2010 edition);

——the acceptance provisions for titanium sponge were modified (see 5.1.2, and 5.1.2 of the 2010 edition);

——the former Annex A was modified to Annex B (see Annex B, and Annex A of the 2010 edition);

——the sampling method for titanium sponge was added (see B.2.1.1);

——the requirements for the preparation of test ingots and test samples of titanium sponge were modified (see B. 2.2.3, and A.2.2.3 of the 2010 edition).

This standard was proposed by the China Nonferrous Metals Industry Association.

This standard is under the jurisdiction of the National Nonferrous Metals Standardization Technical Committee (SAC/TC 243).

The responsible drafting organizations of this standard are: Zunyi Titanium Industry Co., Ltd., Baoti Huashen Titanium Industry Co., Ltd., China Nonferrous Metals Techno-Economic Research Institute, Sponge Titanium Branch of Pangang Group Co., Ltd., Luoyang Sunrui Wanji Titanium Industry Co., Ltd., Chaoyang Jinda Titanium Industry Co., Ltd., Western Titanium Technologies Co., Ltd., Western Superconducting Technologies Co., Ltd., Baosteel Special Steel Co., Ltd., Baoti Group Co., Ltd., Yunnan Metallurgical Xinli Titanium Industry Co., Ltd., and AVIC Tianhe (Tangshan) Titanium Industry Co., Ltd.

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The previous editions of the standard replaced by this standard were issued as follows:

——GB/T 2524—1981, GB/T 2524—2002 and GB/T 2524—2010.

1 Scope

This standard specifies the technical requirements, test methods, inspection rules, marking, packaging, transport, storage, quality certificate and contents of the purchase order (or contract) for titanium sponge.

This standard applies to titanium sponge produced from titanium tetrachloride by the magnesium reduction vacuum distillation method (hereinafter referred to as the magnesium process).

2 Normative References

The following documents contain provisions which, through normative reference in this standard, constitute indispensable provisions of this standard. For dated references, only the edition cited applies. For undated references, the latest edition (including all amendments) applies.

Standard No.	Title
GB/T 231.1	Metallic materials — Brinell hardness test — Part 1: Test method
GB/T 4698 (all parts)	Methods for chemical analysis of titanium sponge, titanium and titanium alloys
GB/T 6005	Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings
GB/T 8170	Rules of rounding off for numerical values and expression and judgement of limiting values

3 Technical Requirements

3.1 Product Classification

Titanium sponge products are classified into 7 grades (designations) according to chemical composition and Brinell hardness: MHT-95, MHT-100, MHT-110, MHT-125, MHT-140, MHT-160 and MHT-200.

3.2 Chemical Composition and Brinell Hardness

3.2.1

The chemical composition and Brinell hardness of titanium sponge products shall conform to the provisions of Table 1.

Table 1 — Chemical composition and Brinell hardness of titanium sponge

Product grade	Product designation	Ti, not less than	Fe	Si	Cl	C	N	O	Mn	Mg	H	Ni	Cr	Other impurity elements, total	Brinell hardness HBW10/1500/30, not greater than
Grade 0A	MHT-95	99.8	0.03	0.01	0.06	0.01	0.010	0.050	0.01	0.01	0.003	0.01	0.01	0.02	95
Grade 0	MHT-100	99.7	0.04	0.01	0.06	0.02	0.010	0.060	0.01	0.02	0.003	0.02	0.02	0.02	100
Grade 1	MHT-110	99.6	0.07	0.02	0.08	0.02	0.020	0.080	0.01	0.03	0.005	0.03	0.03	0.03	110
Grade 2	MHT-125	99.4	0.10	0.02	0.10	0.03	0.030	0.100	0.02	0.04	0.005	0.05	0.05	0.05	125
Grade 3	MHT-140	99.3	0.20	0.03	0.15	0.03	0.040	0.150	0.02	0.06	0.010	—	—	0.05	140
Grade 4	MHT-160	99.1	0.30	0.04	0.15	0.04	0.05	0.20	0.03	0.09	0.012	—	—	—	160
Grade 5	MHT-200	98.5	0.40	0.06	0.30	0.05	0.10	0.30	0.08	0.15	0.030	—	—	—	200

NOTE 1: The other impurity elements generally include (but are not limited to) Al, Sn, V, Mo, Zr, Cu, Er, Y, etc.; for products of grade 1 and above, the content of each of the impurity elements Al and Sn shall not be greater than 0.030%, which is not included in the "other impurity elements, total" specified in this table; the content of Y shall not be greater than 0.005%; the supplier and the purchaser shall negotiate and state it in the purchase order (or contract).

3.2.2

The mass fraction of titanium is the balance obtained by subtracting the sum of the measured values of the impurities in Table 1 from 100%.

3.2.3

The significant figures of the analytical values in the chemical composition analysis report and the measured values of the Brinell hardness shall be consistent with those of the corresponding limiting values for the relevant product grade (designation) in this standard, and the digits after the significant figures shall be rounded off in accordance with the rules specified in GB/T 8170.

3.2.4

For the analytical data of impurity elements other than those specified in Table 1, when the purchaser does not require them, the supplier need not provide them, but shall monitor their contents, which shall meet the requirement of "other impurity elements, total" specified in Table 1; when the purchaser has special requirements for other impurities, the supplier and the purchaser shall negotiate and state them in the purchase order (or contract).

3.3 Particle Size

3.3.1

The particle size specifications of titanium sponge products shall conform to the provisions of Table 2.

Table 2 — Particle size specifications of titanium sponge

Product type	Particle size specification
Standard size	0.83 mm~25.4 mm
Small size	0.83 mm~12.7 mm
Fine size	0.83 mm~5.0 mm

3.3.2

For products of standard size, the weight of the particles larger than the upper limit shall not be greater than 5% of the total weight of the lot, of which the largest particle shall not be greater than 40 mm; the weight of the particles smaller than the lower limit shall not be greater than 5% of the total weight of the lot.

3.3.3

For products of small size, the weight of the particles larger than the upper limit shall not be greater than 5% of the total weight of the lot, and the weight of the particles smaller than the lower limit shall not be greater than 5% of the total weight of the lot.

3.3.4

For products of fine size, the weight of the particles larger than the upper limit shall not be greater than 5% of the total weight of the lot, and the weight of the particles smaller than the lower limit shall not be greater than 5% of the total weight of the lot.

3.3.5

When products of other particle size specifications are required, or there are special requirements for the particle size, the supplier and the purchaser shall negotiate and state them in the purchase order (or contract).

3.4 Visual Quality

3.4.1

The products shall be light grey or silver grey sponge, with clean surface, free from visible inclusions; a small number of defective sponge titanium lumps are permissible.

3.4.2

Defective sponge titanium lumps refer to: sponge titanium lumps with obvious dark yellow or bright yellow oxidation; sponge titanium lumps oxidized and nitrogen-rich with dark yellow or bright yellow traces; sponge titanium lumps with obvious residual chlorides; sponge titanium lumps with residue; sponge titanium lumps with high iron and its associated elements; sponge titanium lumps with dark grey surfaces showing traces of moisture absorption, etc. Some typical samples of defective sponge titanium lumps are shown in Annex A; if there are special requirements, they shall be stated in the purchase order (or contract).

3.4.3

For grade 0A, grade 0 and grade 1 products, the weight of defective sponge titanium lumps shall not exceed 0.05% of the total weight of the lot; for grade 2 and grade 3 products, the weight of defective sponge titanium lumps shall not exceed 0.1% of the total weight of the lot.

4 Test Methods

4.1

The chemical composition analysis of the products shall be carried out in accordance with the provisions of GB/T 4698; the analytical methods for the impurity elements not specified in GB/T 4698 shall be determined by the supplier, and when the purchaser has special requirements, they shall be determined through negotiation between the supplier and the purchaser.

4.2

The Brinell hardness test of the products shall be carried out in accordance with the provisions of GB/T 231.1. The determination of the indentation position is given in B.2.3 of Annex B.

4.3

The particle size inspection of the products shall be carried out in accordance with the provisions of B.5.2 (or the method agreed upon between the supplier and the purchaser).

4.4

The visual quality inspection of the products shall be carried out in accordance with the provisions of B.5.1.

5 Inspection Rules

5.1 Inspection and Acceptance

5.1.1

The titanium sponge products shall be inspected by the supplier to ensure that the product quality conforms to the provisions of this standard and the purchase order (or contract), and a quality certificate shall be filled in.

5.1.2

The purchaser shall inspect the products received in accordance with the provisions of this standard and the purchase order (or contract). When the inspection results of the chemical composition and Brinell hardness do not conform to the provisions of this standard and the purchase order (or contract), the matter shall be raised with the supplier within 60 days from the date of receipt of the products; when the inspection results of the particle size and visual quality do not conform to the provisions of this standard and the purchase order (or contract), the matter shall be raised with the supplier within half a year from the date of receipt of the products; the matter shall be settled through negotiation between the supplier and the purchaser. If arbitration is required, the samples for arbitration shall be taken by the supplier and the purchaser jointly from the products received by the purchaser in accordance with the provisions of B.4.

5.2 Batching

The titanium sponge products shall be submitted for acceptance in lots. Each lot shall consist of products from the same furnace, of the same grade (designation) and the same particle size specification. The lot weight shall be 500 kg~12 000 kg.

5.3 Inspection Items and Sampling Provisions

The inspection items and sampling of the products shall conform to the provisions of Table 3.

Table 3 — Inspection items and sampling provisions

Inspection item	Provision for sampling and sample preparation	Clause for requirements	Clause for test method
Chemical composition	Annex B	3.2.1	4.1
Brinell hardness	Annex B	3.2.1	4.2
Particle size	B.5.2	3.3	4.3
Visual quality	B.5.1	3.4	4.4

5.4 Judgement of Inspection Results

5.4.1

When the chemical composition inspection result is non-conforming, the lot shall be judged non-conforming.

5.4.2

When the Brinell hardness inspection result is non-conforming, the lot shall be judged non-conforming.

5.4.3

When the particle size inspection result is non-conforming, the lot shall be judged non-conforming.

5.4.4

When the visual quality inspection result is non-conforming, the lot shall be judged non-conforming.

6 Marking, Packaging, Transport, Storage and Quality Certificate

6.1 Marking

The products shall be packed in drums, and the outside of each drum shall be marked with:

a) name of the supplier and its registered trademark of the products;

b) product name and particle size specification;

c) lot number, grade or designation, net weight and gross weight;

d) date of packaging and rainproof mark.

6.2 Packaging, Transport and Storage

6.2.1

The products shall be packed in drums with a net weight of 70 kg~250 kg each. The packaging drum shall be a zinc-plated iron drum with a removable cover, lined with a polyvinyl chloride film bag inside, and sealed with vacuum rubber.

6.2.2

After packaging, the drum shall be evacuated and filled with nitrogen, or the air in the drum shall be displaced with nitrogen; a mark enabling identification of whether the packaging is intact shall be added at the joint between the drum cover and the drum body.

6.2.3

The packed products shall be stored in a dry warehouse, and shall not be stacked in the open air or stored together with corrosive articles such as acids and alkalis.

6.2.4

During transport and storage, the products shall be handled with care, strictly protected from rain and moisture, and the packaging drums shall be prevented from damage by collision.

6.2.5

During packaging, transport and storage, attention shall be paid to fire prevention, and contact with ignition sources shall be avoided. If the products catch fire during packaging, argon shall be used for extinguishing; if they catch fire during transport or storage, dry sand, potassium chlorate, sodium chloride, a mixture of potassium chlorate and sodium chloride, dolomite powder or asbestos cloth may be used for extinguishing. Water or foam shall not be used for extinguishing.

6.3 Quality Certificate

Each lot of products shall be accompanied by a quality certificate, on which the following shall be stated:

a) name, address, telephone and fax of the supplier;

b) product name;

c) lot number, particle size, grade or designation, weight and number of drums;

d) analysis results and the seal of the inspection department;

e) number of this standard and number of the purchase order (or contract);

f) date of inspection.

7 Contents of the Purchase Order (or Contract)

The purchase order (or contract) for the products listed in this standard shall include the following contents:

a) product name;

b) grade or designation;

c) impurity content and particle size (when required by the purchaser);

d) quantity;

e) number of this standard;

f) others.

Annex A (Informative) — Figure Samples of Defective Sponge Titanium Lumps

Some typical samples of defective sponge titanium lumps are shown in Figure A.1.

The captions of the samples in Figure A.1 are as follows:

a) sponge titanium lump oxidized in the reduction–distillation process;

b) sponge titanium lump oxidized in the crushing process;

c) sponge titanium lump with chlorides;

d) sponge titanium lump with residue;

e) sponge titanium lump with high iron content;

f) yellowed sponge titanium lump;

g) sponge titanium lump with high nitrogen content;

h) foamy sponge titanium lump;

i) sponge titanium lump with contaminated surface;

j) sponge titanium lump with bright round (columnar) outer surface.

(Figure A.1 — Figure samples of defective sponge titanium lumps)

Annex B (Normative) — Sampling of Titanium Sponge Products, Preparation of Test Samples and Test Ingots, and Methods for Visual Quality and Particle Size Inspection

B.1 Scope

This annex specifies the sampling methods for titanium sponge products and the methods for preparing the test samples and test ingots used for the chemical composition analysis and hardness determination of the products.

This annex applies to the determination of the quality of a lot of titanium sponge products.

B.2 Methods for Sampling by the Supplier and Preparation of Test Ingots of Titanium Sponge Products

B.2.1 Sampling Methods

B.2.1.1

In principle, sampling shall be carried out before the lot of titanium sponge products is packaged, and an automatic sampling device is recommended for continuous sampling.

B.2.1.2

The sampling and sample preparation process shall make the sample taken as representative as possible of the average composition of the lot of products.

B.2.1.3

The tooling used for taking samples shall ensure that the samples do not deteriorate or become contaminated.

B.2.1.4

The samples of titanium sponge shall be taken by the following methods; if there are special requirements, they may be settled through negotiation between the supplier and the purchaser:

a) Taking of primary samples

After the lot of titanium sponge products is crushed, sorted and thoroughly mixed, samples shall be taken from the continuously flowing titanium sponge at certain time intervals in small quantities of 30 g~300 g by an automatic sampling device; or after the batch of titanium sponge products is thoroughly mixed, it shall be placed in a rectangular stainless steel container, spread to a uniform thickness, divided equally according to its area, and samples shall be taken from each part in small quantities of not less than 300 g with a stainless steel square shovel of capacity not less than 1 000 g. The total quantity of samples taken shall be more than 0.5% of the lot quantity, and shall be not less than 20 kg.

b) Taking of secondary samples

The samples collected in a) shall be spread flat on a stainless steel plate and reduced by the quartering method to a sample of not less than 4 kg to obtain the secondary sample; the secondary sample shall then be divided equally into 4 portions, each of not less than 0.9 kg. The secondary sample represents the average composition of this lot

of titanium sponge products and serves as the supply sample for preparing the test samples for the chemical composition analysis and hardness test of the products, of which one portion is the test sample of the supplier, and the remaining portions are kept for preservation.

B.2.2 Preparation of Test Samples

B.2.2.1 Pressing of the Consumable Electrode

About 0.9 kg of the supplier's test sample (the quantity is determined by the capacity of the pressing die) shall be put into a clean pressing die and pressed into a consumable electrode on a hydraulic press; the electrode forming pressure shall be 300 MPa~500 MPa, and the pressure holding time shall be 3 s~5 s.

B.2.2.2 Vacuum Melting and Casting of the Titanium Ingot

The pressed consumable electrode shall be put into a drying oven and dried for 20 min~40 min at a drying temperature of 90 ℃~105 ℃; after drying, it shall be loaded into a vacuum consumable arc furnace for melting and casting. Before melting, the system shall be inspected and pre-evacuated; the absolute vacuum degree in the furnace shall not be greater than 0.133 Pa, and the leakage rate shall not be greater than 0.667 Pa/min. During melting, the current intensity shall be 900 A~1 100 A, the working voltage shall be 24 V~45 V, and the cooling water flow rate shall be 9 L/min~12 L/min. The ingot shall be cooled in the furnace for not less than 20 min.

B.2.2.3 Preparation of the Test Ingot and Test Samples

The titanium ingot taken out of the water-cooled copper crucible shall be clamped on a lathe and turned at a speed of not greater than 405 r/min to remove the shrinkage cavity portion at the head of the ingot; a titanium layer 10 mm thick at the head and 5 mm thick on the side shall be turned off. The feed rate and depth of cut of the lathe shall be such that no oxidation occurs on the surface of the test ingot. The sampling positions are shown in Figure B.1.

The test samples of each type shall be prepared as follows:

a) Preparation of the test samples for oxygen, nitrogen and hydrogen analysis

A titanium ring with an outer diameter of not less than 35 mm, a width of 5 mm~7 mm and a thickness of 2 mm~4 mm shall be turned from the middle-upper part of the ingot, with a depth of 15 mm, for the analysis of O, N and H.

b) Preparation of the test samples for the analysis of silicon, iron, manganese, magnesium, carbon and other elements

With the spindle speed of the lathe not greater than 450 r/min and a feed rate of 0.031 mm/r, filamentary turning samples with a width of 1 mm~3 mm, a thickness of 0.05 mm~0.2 mm, an appearance without discoloration and a mass of not less than 20 g shall be prepared for the analysis of silicon, iron, manganese, magnesium, carbon and other elements.

c) Preparation of the test sample for chlorine analysis

A high-speed steel twist drill of φ8 mm~10 mm shall be used; with the spindle speed of the bench drilling machine at 560 r/min, 5 holes shall be drilled evenly along the length direction on the residual end of the unmelted consumable electrode. After removing the surface layer of 3 mm~5 mm, drilling chips of 20 g shall be taken on the premise that the electrode is not drilled through and the chips do not change color, for chlorine analysis.

d) Preparation of the ingot for the Brinell hardness test

After the preparation of the analysis samples, the titanium ingot shall be machined into an ingot for the Brinell hardness test at a lathe speed of not greater than 450 r/min and with a feed rate and depth of cut such that no oxidation occurs on the surface of the ingot. The ingot shall have a diameter of not less than 40 mm and a height of not less than 15 mm. The surface of the ingot for the Brinell hardness test shall be smooth, with a roughness parameter Ra of not greater than 1.6 μm, and the upper and lower faces shall be parallel; four points shall be measured with a micrometer in the perpendicular direction, and the maximum difference shall not be greater than 0.05 mm; the surface of the test ingot shall be free from porosity or cracks.

(Figure B.1 — Preparation of test samples: 1 — ingot; 2 — titanium ring for the analysis of O, N and H; 3 — filamentary turning sample for the analysis of Si, Fe, Mn, Mg, C and other impurity elements; 4 — ingot for the Brinell hardness test; d — diameter of the ingot for the Brinell hardness test, in mm; h — height of the ingot for the Brinell hardness test, in mm.)

B.2.3 Determination of the Indentation Positions

B.2.3.1

On the upper surface of the ingot for the Brinell hardness test, the indentation positions shall be determined in a diamond-shaped arrangement of 1/2 and 2/3 (as shown in Figure B.2).

(Figure B.2 — Indentation positions)

B.2.3.2

The Brinell hardness values of four points on the upper surface of the same test ingot shall be measured, and their average value shall be taken as the average Brinell hardness value of the titanium ingot. If the difference between the Brinell hardness value of one of the four points and the average Brinell hardness value exceeds 10 units, the average value of the Brinell hardness values of the other three points shall be taken as the average Brinell hardness value of the titanium ingot; if the differences between the Brinell hardness values of two points and the average Brinell hardness value both exceed 10 units, the ingot shall be re-cast and re-tested.

B.3 Methods for Sampling by the Purchaser and Preparation of Test Ingots of Titanium Sponge Products

B.3.1 Sampling

B.3.1.1

20% of the number of drums (not less than 3 drums; when the lot consists of fewer than 3 drums, all drums shall be sampled) shall be randomly taken from the lot of products received; not less than 20 kg of products shall be taken from different parts of the drums, mixed evenly, and reduced to a sample of 4 kg.

B.3.1.2

The 4 kg sample obtained by reduction shall be divided equally into three portions, of which one portion is used for pressing the consumable electrode and the other two portions are kept as spare samples.

B.3.2 Preparation of the Test Ingots

The pressing of the consumable electrode, melting and casting, and the preparation of each test sample shall be carried out in accordance with B.2.2.

B.4 Methods for Arbitration Sampling and Preparation of Test Ingots of Titanium Sponge

B.4.1 Sampling

For arbitration sampling, the supplier and the purchaser shall be present at the same time, and sampling shall be carried out by the following method:

a) 20% of the number of drums (not less than 3 drums; when the lot consists of fewer than 3 drums, all drums shall be sampled) shall be randomly taken from the lot of products in dispute, and not less than 20 kg of products shall be taken evenly from the upper, middle and lower parts of each drum, mixed evenly, and reduced to a sample of 4 kg.

b) The 4 kg sample obtained by reduction shall be divided equally into three portions, one portion being sent to the arbitration organization, one portion kept by the supplier, and one portion kept by the purchaser.

B.4.2 Preparation of the Test Ingots

The pressing of the consumable electrode, melting and casting, and the preparation of each test sample shall be carried out in accordance with B.2.2.

B.5 Methods for Visual Quality and Particle Size Inspection of Titanium Sponge

B.5.1 Visual Quality of Titanium Sponge

10% of the number of drums (not less than 3 drums; when the lot consists of fewer than 3 drums, all drums shall be sampled) shall be randomly taken from the lot of products, placed on a clean stainless steel plate, and inspected by visual examination. The defective sponge titanium lumps shall be selected in accordance with the provisions of 3.4.2 and weighed, so as to determine whether their weight exceeds the provisions of this standard.

B.5.2 Particle Size Inspection Method

B.5.2.1

In accordance with the requirements of GB/T 6005, sieves meeting the requirements shall be selected, and the nominal sizes of the openings shall correspond to the upper and lower limits of the particle size requirements of the products.

B.5.2.2

10% of the number of drums (not less than 2 drums) shall be randomly taken from the lot of products.

B.5.2.3

The two layers of sieves corresponding to the upper and lower limits of the particle size requirements of the products shall be placed on a vibrating machine, and the products taken out shall be put into the upper sieve tray in portions; after vibrating for 2 min~5 min and confirming that the sieving is complete, the undersize of the lower sieve tray and the oversize of the upper sieve tray shall be taken out and weighed respectively, and the calculation

and judgement shall be made as to whether the weight conforms to the requirements of this standard and the purchase order (or contract).