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**Technical requirements of nitride carbonize
and oxidize multi-treatment (QPQ)**

氮碳氧复合处理（QPQ）技术要求

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Technical requirements of nitride carbonize and oxidize multi-treatment (QPQ)

1 Scope

This standard specifies the terms and definitions, equipment requirements, process material requirements, process requirements, quality control and inspection, safety, hygiene and environmental protection requirements of the nitride carbonize and oxidize multi-treatment (QPQ) technology.

This standard applies to the nitride carbonize and oxidize multi-treatment (QPQ) of black metal, cast iron and stainless steel.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 230.1 Metallic materials - Rockwell hardness test - Part 1: Test method (scales A, B, C, D, E, F, G, H, K, N, T)

GB/T 443 L-AN full loss system oil

GB/T 4340.1 Metallic materials - Vickers hardness test - Part 1: Test method

GB 5959.1 Safety in electroheat installations - Part 1: General requirements

GB 5959.4 Safety in electroheat installations - Part 4: Particular requirements for safety resistance heating installations

GB/T 6067.1 Safety regulations for lifting appliances - Part 1: General

GB/T 7232 Terminology of metal heat treatment

GB/T 8121 The terminology of technological materials for heat treatment

GB/T 9452 Testing method for working zone of heat treatment furnace

GB/T 10067.1 Basic specifications for electroheat installations Part 1: General

GB/T 10067.4 Basic technical conditions for electroheat equipment - Part 4:
Indirect resistance furnace

GB/T 11354-2005 Steel-iron parts - Determination of nitrided case depth and
metallographic examination of nitriding structure

GB 14554 Emission standards for odor pollutants

GB 15735 Requirements for the safety and health in production process of
metal heat treatment

GB/T 22560 Gas nitrocarburizing of iron and steel parts

GB/T 27945.1 The management of hazardous solid wastes from heat
treatment salt - Part 1: General management

GB/T 27945.2 The management of hazardous solid wastes from heat
treatment salts - Part 2: Test method of extractives

GB/T 27945.3 The management of hazardous solid wastes from heat
treatment salts - Part 3: Method of innocent treatment

GB/T 27946 Limited value of hazardous substance in the air of heat
treatment working place

GB/T 30822 Environmental protection technical requirements for heat
treatment industry

GB/T 32529 Technical requirement of cleaning liquid waste recycling and
emissions for heat treatment

GB/T 32541 Quality control system for heat treatment

JB/T 6050 Generalization of hardness test for heat treated iron and steel
parts

JB/T 6090 Polishers

3 Terms and definitions

The definitions of GB/T 7232, GB/T 8121, GB/T 11354-2005 and the following
terms and definitions apply to this document.

3.1

**Nitride carbonize and oxidize multi-treatment (Quench-Polish-Quench,
QPQ)**

4.4.5 Oxidation furnace shall have salt leakage alarm device and corresponding safety protection device.

4.4.6 Oxidation furnaces shall have air extraction devices and shall comply with the requirements of 4.9.

4.4.7 Oxidation furnace can choose electric lifting furnace lid or pneumatic lifting furnace lid, and set up the salt addition port on the furnace lid.

4.5 Control system

4.5.1 The external heat crucible resistance furnace shall meet the requirements of GB/T 32541 for equipment and instruments.

4.5.2 Each heating zone shall have two thermocouples, one is connected to temperature control instruments and installed between the crucible and the resistance wire, the other is connected to the temperature recording instrument and installed in the crucible, the instrument shall have the alarm function; the thermocouples shall be checked regularly to ensure normal use.

4.5.3 The control system shall be able to display, control and record the temperature, as well as record and store the whole process. It has the functions of automatic temperature timekeeping, automatic timing alarm and salt leakage alarm, it has the functions of overcurrent, overload and phase failure protection.

4.6 Cleaning device

4.6.1 Cleaning device is divided into pre-cleaning and post-cleaning device, it may use cleaning tank or cleaning machine.

4.6.2 The cleaning tank shall be made of glass steel or stainless steel, with stirring function, and its effective size is determined in accordance with the size of the nitriding furnace crucible.

4.6.3 The temperature control device of the hot water cleaning tank is subject to temperature measurement by a thermocouple or a thermal resister, and temperature control by temperature control instrument, with the operating temperature $\geq 80\text{ }^{\circ}\text{C}$.

4.6.4 The pre-cleaning device shall be provided with a waste water recovery device.

4.6.5 The post-cleaning device shall be provided with a waste salt recovery device.

4.6.6 If cleaning the workpiece with a washing machine, the washing machine

4.10 Lifting system

4.10.1 Lifting machinery shall meet the operational requirements of the QPQ production line and use the electric hoist with the corresponding load in accordance with the QPQ equipment specifications.

4.10.2 The design, manufacture and installation of the lifting system shall comply with the provisions of GB/T 6067.1.

5 Process material requirements

5.1 Composition of process materials

Process materials consist of base salts, regenerated salts, and oxidized salts.

5.2 Process material properties

5.2.1 Base salt

5.2.1.1 Base salts are mixtures of carbonate, sodium, lithium carbonates and cyanates. The base salt is added into the nitrocarburizing furnace, mainly providing active nitrogen carbon atoms to the salt bath nitrocarburizing of the workpiece, when the liquid level in the nitrocarburizing furnace falls, the base salt shall be added to raise the liquid surface.

5.2.1.2 The base salt is in the form of a white block, powder, or other shape and shall not contain heavy metals; it shall not produce gas when the base salt is added and supplemented.

5.2.1.3 The base salt can be used in the range of 480 °C ~ 650 °C. The cyanide content (mass fraction) in the salt bath at the time of exit-factory is 37% or negotiated between the supplier and the purchaser, the deviation does not exceed $\pm 1\%$.

5.2.2 Regenerated salt

5.2.2.1 The salt bath decomposes naturally during workpiece salt bath nitrocarburizing reaction and heating, the content of cyanide in the salt bath will continuously decrease. The regenerated salt is an organic substance used to adjust the content of the active ingredient cyanate in the salt bath.

5.2.2.2 The regenerated salt is white and shall not contain heavy metals; only traces of gas can be produced when regenerated salt is added.

6.3.2.2 The maximum loading amount per furnace is less than 50% of the salt bath mass.

6.3.2.3 Workpieces shall avoid direct contact with the workpiece and fixtures, to prevent degradation of the nitriding quality of the contact layer.

6.3.2.4 For the workpieces with blind holes or grooves, blind holes or grooves shall be downwards to avoid accumulation of salt.

6.3.2.5 Rod shape and plate shape workpieces shall be clamped vertically to reduce distortion.

6.3.2.6 When the workpiece is processed, the fixture for mounting the workpiece shall be suspended in the crucible; the distance between the bottom end of the fixture and the bottom of the crucible shall be ≥ 50 mm, the distance between the top of the fixture and the salt bath surface in crucible shall be ≥ 50 mm.

6.3.3 Pre-cleaning

6.3.3.1 The cleaning tank shall meet the requirements of 4.6.

6.3.3.2 The clamped workpiece shall be cleaned with a metal cleaner solution, cold water and hot water, to remove the oil and dirt from the workpiece.

6.3.3.3 Wash the workpiece with a washing machine, remove the oil and dirt from the workpiece, blow (bake) dry, then clamp it.

6.3.3.4 Metal cleaner shall meet the requirements for use.

6.3.3.5 The cleaning process must not have a detrimental effect on the surface of the workpiece.

6.3.4 Preheating

6.3.4.1 The preheating furnace shall meet the requirements of 4.2.

6.3.4.2 The role of the preheating process is to remove the moisture from the surface of the workpiece and blind holes, to prevent the salt solution from splashing and injuring people as water enters the nitrocarburizing furnace, ensure that the temperature of the nitrocarburizing furnace does not decrease too much, and improve the workpiece surface quality.

6.3.4.3 The preheating temperature and the preheating time are properly selected in accordance with the workpiece material, the loading amount per furnace, and the like.

6.3.4.4 The preheating temperature is generally $350\text{ }^{\circ}\text{C} \sim 400\text{ }^{\circ}\text{C}$. The

6.3.5.5 The nitrocarburizing temperature in the salt bath and the nitrocarburizing time in the salt bath for commonly used materials are given in Appendix C.

6.3.5.6 Through the inspection of preliminary heat treatment specimens, reasonably determine the salt bath nitrocarburizing processing parameters.

6.3.5.7 Correct the salt bath nitrocarburizing process parameters by checking the test specimens.

6.3.5.8 After the workpiece enters the nitrocarburizing furnace, if the furnace temperature does not drop by more than 20 °C, the nitrocarburizing time starts from the point when the workpiece enters the furnace. If the furnace temperature drops by more than 20 °C, the nitrocarburizing time shall start from the point when the furnace temperature rises to the setting temperature.

6.3.5.9 The content of cyanate in the nitrocarburizing salt bath shall be strictly controlled. The content of cyanate in the salt bath at the beginning of production shall be measured every day. The cyanate can be measured once or twice a week after the production enters into normal conditions.

6.3.5.10 After the nitrocarburizing salt bath treatment for a certain period of time, the regenerated salt shall be added at a suitable time in accordance with the change of the cyanate content.

6.3.5.11 When adding regenerated salt, the salt bath temperature shall be controlled at $540\text{ °C} \pm 10\text{ °C}$, so that the regenerated salt reaction is more fully, reducing the evaporation of the regenerated salt and increasing the yield.

6.3.5.12 The salt bath shall react for 1 h after adding the regenerated salt before it can continue production; when a large amount of regenerated salt is needed to substantially increase the content of cyanate, it shall follow the principles of more number of additions but less amount of additions.

6.3.5.13 When the liquid level of the nitrocarburizing salt bath drops, the base salt shall be added. The amount of base salt added shall be determined by the liquid level of the nitrocarburizing salt bath.

6.3.5.14 The slag removal shall be performed after the nitrocarburizing salt bath treatment for 10 h.

6.3.5.15 The slag extracted from the nitrocarburizing salt bath is broken after cooling and placed in an oxidization salt bath.

6.3.5.16 The addition of base salts is usually performed in conjunction with the regenerated salt addition and the slag removal process.

6.3.5.17 In case of salt bath nitrocarburizing, if it requires leading compressed

6.3.7.5 The water in the first cold water tank shall be periodically treated and drained after reaching to the environmental protection requirements.

6.3.7.6 The second cold water tank is cleaned with running water and the wastewater can be discharged normally.

6.3.7.7 The cleaning process shall not have a detrimental effect on the surface of the workpiece.

6.3.8 Polishing

6.3.8.1 The purposes of polishing is to remove the loose layer from the surface of the workpiece and reduce the roughness of the surface of the workpiece, in order to greatly improve the corrosion resistance of the workpiece after secondary oxidation.

6.3.8.2 Reasonably select the polishing method and polishing equipment in accordance with the workpiece shape, technical requirements, etc.

6.3.8.3 The polishing equipment shall meet the requirements of 4.7.

6.3.8.4 Helical vibration polishers and centrifugal vibratory polishers are suitable for shallow polishing of small parts and irregular parts.

6.3.8.5 Rod-shaped centerless grinding and polishing machine is suitable for the polishing of rod and rod-shaped parts such as piston rod and pin shaft.

6.3.8.6 Ordinary wheel-type polisher is suitable for the polishing of parts with large size and relatively regular shape. Select different types of polishing wheels to achieve different polishing results.

6.3.8.7 Reasonable control the polishing degree in accordance with the appearance of the workpiece and performance requirements.

6.3.8.8 Polishing process shall not adversely affect the surface of the workpiece.

6.3.9 Secondary salt bath oxidation

6.3.9.1 The secondary salt bath oxidation temperature is generally 400 °C ~ 450 °C, salt bath oxidation time is generally 10 min ~ 60 min.

6.3.9.2 Others shall meet the requirements of 6.3.6.

6.3.10 Re-cleaning

Re-cleaning shall be performed in accordance with the requirements of 6.3.7.

6.3.11 Oil immersion

compound layer is measured, the depth of the diffusion layer is measured only when required.

7.2.5 Metallographic organization

Metallographic organizations shall meet the technical requirements of the normal organization, it shall not be less than the requirements of GB/T 11354.

7.2.6 Distortion

Distortion shall meet the technical requirements of the workpiece.

7.2.7 Others

In accordance with the technical requirements of the workpiece, the relevant standards can be followed to perform test of such items of the corresponding specimens as mechanical properties, wear resistance, corrosion resistance and so on.

8 Safety, hygiene and environmental protection requirements

8.1 Safety and hygiene requirements shall comply with the provisions of GB 15735, GB 5959.1, GB 5959.4 and GB/T 30822.

8.2 The waste water, waste liquid, waste gas, and waste residue generated from the process material production process and process implementation shall be collected and treated in a manner that does not adversely affect the environment.

8.3 Waste gas and wastewater discharge during the production process and process implementation of process materials shall meet the requirements of GB 14554, GB 15735, GB/T 27946, GB/T 30822 and GB/T 32529.

8.4 The discharge of solid waste generated during the production process and process implementation of process materials shall be performed in accordance with GB/T 27945.1 and GB/T 27945.2.

8.5 In the event of sudden power failure or water stoppage during production, the equipment shall have corresponding protective measures to protect the safety of personnel, equipment and parts.

8.6 Warning signs shall be set up in all places that may cause personal injury.

Appendix B

(Normative)

Cyanate chemical analysis method

B.1 Reagents

B.1.1 Mixed indicator. WEIGH 0.03 g of methyl red, DISSOLVE it in 50 ml of absolute ethanol, USE distilled water to dilute it to 100 mL; WEIGH 0.015 g of methylene blue, DISSOLVE it in 15 mL of water. After the two are completely dissolved, MIX them and CONTAIN it into the dropping bottle.

B.1.2 ADD 0.1 g of phenolphthalein (100% high purity) into 100 mL of ethanol (ethanol solution concentration 90%) to obtain a 0.1% mass fraction phenolphthalein solution.

B.1.3 PREPARE the formaldehyde (1:1) into 500 mL or 1000 mL in accordance with the specimen requirements. MEASURE the blank value once every preparing the solution (100mL of formaldehyde plus 100 mL of distilled water).

B.1.4 BOIL the 10% (mass fraction) NaOH in distilled water to prepare it (e.g. WEIGH 10 g of NaOH, USE 90 mL of boiled distilled water to dissolve it).

B.1.5 1 mol/L H₂SO₄ [e.g. 28 mL of 95% ~ 98% (mass fraction) concentrated sulfuric acid, distilled water 472 mL].

B.1.6 Configuration and calibration of 0.1 mol/L NaOH solution.

B.2 Analytical determination

B.2.1 Sampling

USE a stainless steel rod of about Φ30 mm to insert about 100 mm into the nitriding salt bath, then quickly LIFT it up. After the salt flakes around the rod fall off, COLLECT them in a container and prepare for the test. Specimens are ground fine in a mortar, PLACE it in an oven at 80 °C ~ 90 °C, BAKE it for 1 h, COOL it in a desiccator (a new salt sample is taken and tested immediately, without the need of baking). WEIGH 5 g of salt sample in a small beaker, ADD 50 mL of distilled water, HEAT it on an electric furnace to make it completely dissolve, TAKE it off and COOL it, TRANSFER it into a 250 mL volumetric flask, USE distilled water to make its volume reach to the mark, SHAKE it uniformly, USE rapid quantitative filter paper to filter it dry to prepare for use.

B.2.2 Determination

PIPETTE 10 mL of the above solution, ADD 20 mL of distilled water in a 250 mL flask, ADD 5 mL of H₂SO₄ solution (2N), COVER with a small funnel, HEAT to boil it for 2 min, REMOVE and COOL it down. ADD 2 drops of mixing indicator, SHAKE it uniformly, the solution is purple red [If it is green, add 2 mL of H₂SO₄ (1 mol/L) boil for 2 min, the solution shall still be purple], ADD 10% (mass fraction) NaOH solution to neutralize the solution to green (add in drops and shake it uniformly). Then ADD 1 mol/L H₂SO₄ dropwise until the solution turns red. USE a small amount of distilled water to rinse the bottle wall, ADD 0.1 mol/L NaOH dropwise until it is green, ADD 10 mL of formaldehyde (1:1) solution, SHAKE it uniformly and LET it be standing for 1 min, ADD 4 drops of phenolphthalein indicator, USE the 0.1 mol/L NaOH standard solution to titrate it, the solution turns bright red from purple to red, USE a steady reddish color as the end point, RECORD the titration millimeters V₁. TAKE another 10 mL of formaldehyde (1:1) in a 250 mL conical flask, ADD 5 mL of distilled water, ADD 4 drops of phenolphthalein, USE 0.1 N NaOH standard solution to titrate it to the slight reddish color, RECORD the number of milliliters of titration. This is blank value V₂.

B.2.3 Calculation

The cyanate ion content is calculated by the formula (B.1).

$$w(\text{CNO}^-) = \frac{N \times 42 \text{ g/mol} \times (V_1 - V_2)}{G \times 1000 \text{ mL/L}} \times 100\% \quad \text{..... (B.1)}$$

Where:

w (CNO⁻) - Mass fraction of cyanate ions;

N - The amount of substance concentration of sodium hydroxide standard solution, in moles per liter (mol/L);

V₁ - The volume of sodium hydroxide standard solution consumed when titrating the sample and reagent mixture, in milliliters (mL);

V₂ - The volume of sodium hydroxide standard solution consumed when titrating the reagent, in milliliters (mL);

G - The mass of the specimen in the determined sample solution, in grams (g).

B.3 Preparation of 0.1 mol/L NaOH standard solution

B.3.1 Weigh 40 g of NaOH (excellent grade) into a 1000 mL beaker, ADD 400

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