QWISE-HJ-SJZDS-2019 Company Standard

Hunan WISE New Material Technology Co., Ltd.

Steel Bonded Carbide sub-factory

H2 Sintering process

Operating instructions

Approved by: Zhaohui LIU Prepared by: Liyun Yun Xing Wang
Checked by Technical Dept: Yizhong Zhang and Yongshen Gong
Checked by Sub-Factory: Zhengchen Xu and Zhengdao Xiao
August 1, 2019 Release August 1, 2019 Effective

Hunan WISE New Material Technology Co., Ltd. Release

QWISE-HJ-SJZDS-2019

Company Standard

Hunan WISE New Material Technology Co., Ltd.

Steel Bonded Carbide sub-factory Operating instructions

H2 Sintering process

1 main raw and auxiliary materials

- 1.1 Main raw materials: various grades and various types of compacts.
- 1.2 Main auxiliary materials: vacuum grease, Ar, N2, CH4, H2, compressed air, graphite boat, pump oil, filler paper, corundum, wooden box.
- 2 major equipment and tools
- 2.1 Main equipment: low pressure sintering furnace, vacuum dewaxing sintering furnace, H2 dewaxing furnace, drying box, sand blasting machine.
- 2.2 Main tools: wrenches, screwdrivers, loading vehicles, flashlights, platform scales, etc.
- 3 job quality requirements
- 3.1 Operate in strict accordance with the equipment safety technical regulations.
- 3.2 The sintering system of each grade and the sintering process parameters of each grade shall be implemented according to internal control standards or interim standards.
- 3.3 The internal quality of the sintered product shall be implemented in accordance with internal control standards or interim standards.
- 3.4 The geometry of the sintered product shall comply with the relevant product standards or drawings.
- 3.5 The surface of the sintered product is uniform in color, without bubbling, peeling, bending deformation, falling edge, corner drop and so on.
- 4 steps and essentials
- 4.1 Loading the boat
- 4.1.1 Select the boat according to different compacts, and the surface of the boat should be kept flat.
- 4.1.2 For the loading of the dry bag and the extruded compact, the boat and the lining of the appropriate groove type should be selected according to the model. The height of the lining should be placed in the groove of the boat and then the height of the blank is $1\sim10$ mm, $\Phi6$ mm or less. The squeezed blank must be pressed when the boat is loaded.
- 4.1.3 When loading the boat, it is necessary to inspect the compact, and select the compacts with serious falling edges, corners, delamination, cracks, etc., and gently put them while loading,

leaving gaps between the blanks.

- 4.2 Operation before opening the furnace
- 4.2.1 Inspection before opening

Check whether the dewaxing furnace electrical appliances and instruments are normal: check whether the waterway is unblocked, check whether the intake pipe and exhaust pipe are unblocked; check whether the exhaust air is in good condition; check whether the crane is safe before opening the bell-type dewaxing furnace.

- 4.2.2 into the furnace
- 4.2.2.1 Carefully handle the compact, the height of the compact in the bell-type dewaxing furnace does not exceed 800mm, install the inner lid of the boat, and then cover the furnace. The electric hoist should be stable during operation, and the amplitude should not be too large. And one person must operate one person for monitoring, when lifting and lifting in place, the main point is moving slowly (the horizontal dewaxing furnace is full).
- 4.2.2.2 Check if the corundum in the sealed tank is sufficient and not enough to be replenished.
- 4.2.2.3 Check whether there is any fire source within four meters around the H2 dewaxing furnace. If it is, the hydrogen can be sent to the furnace after the elimination, and a warning sign is hanged to prevent accidents.
- 4.2.3 Opening the furnace
- 4.2.3.1 The open blower delivers hydrogen for 30~40 minutes, the hydrogen flow rate is
- $1.5\sim2.5$ m3/h, and then the blasting test. The blasting test must leave the hydrogen source 4 meters away. After the blasting test is passed, the ignition is turned off.
- 4.2.3.2 Large-open furnace cooling water (the bell-type dewaxing furnace should also open the thyristor cooling water), and the power supply is heated. The bell-type dewaxing furnace should send electricity to the hydrogen-discharging pipeline.
- 4.2.3.3 During the furnace opening process, pay attention to whether the instrument is normal. If problems are found, the duty officer should respond immediately.
- 4.2.3.4 The maximum furnace temperature shall not exceed 900 °C, and the maximum furnace capacity of the bell-type furnace shall not exceed 300 kg.
- 4.2.3.5 Do the "vacuum sintering original record" during the operation, record once every 20~40min.
- 4.2.4 Troubleshooting
- 4.2.4.1 Sudden hydrogen stop: The hydrogen discharge port must be closed immediately, the hydrogen supply port should be closed, the heating power supply should be cut off, the cooling water should be turned on, and the hydrogen supply should be quickly contacted. After the hydrogen is turned on, the test will be performed. If it is qualified, it will be ignited and automatically operated. No hydrogen has been supplied, and the furnace is cooled down to below 100 °C.
- 4.2.4.2 Sudden power failure: cut off the heating power supply, the cooling water will continue to open, keep the hydrogen unblocked, manually raise the temperature to the temperature at the time of power failure, and then automatically run; if it has been in the future, the furnace temperature will drop below 100 °C to stop the furnace.
- 4.2.4.3 Sudden water stop: Cut off the heating power supply, keep the hydrogen flowing smoothly, manually raise the temperature to the temperature at the time of power failure after the call, and then automatically run; if the water has been in the future, the furnace temperature will drop to

below 100 °C to stop the furnace.

4.2.5 Shutdown

Normally shut down, naturally lowering to below 450 °C, the bell-type dewaxing furnace can lift the furnace body to expose the inner tank (the horizontal dewaxing furnace can open the exhaust, open the furnace shell fan), first below 100 °C The hydrogen discharge port is closed, the hydrogen inlet valve is closed, the furnace is discharged below 60 °C, the cooling water is turned off, and the horizontal dewaxing furnace is turned off.

4.2.6 Released

The inner liner is hoisted out, and the pressed product which has been detached from wax is taken out on a product car with a foam or a graphite plate, and the product is matched with the transfer card.

- 4.3 Low pressure sintering operation
- 4.3.1 Inspection and preparation
- 4.3.1.1 Turn on the power of the control cabinet and enter the main screen state.
- 4.3.1.2 Turn on the cooling water system and check if the water system is normal. The inlet pressure is generally 30~50psi (2.1×105.pa~3.4×105.pa).
- 4.3.1.3 Check if the compressed air is normal, generally 70~110psi (4.8×105.~7.6×105.pa).
- 4.3.1.4 Check whether the inert gas Ar, N2 is turned on, the regulating pressure is 0.2~0.5Mpa, and the N2 or Ar keeps normally open when the circuit is open, and the H2 and CH4 gases are adjusted according to the temporary process requirements.
- 4.3.1.5 Open the two furnace doors smoothly, check whether the heating elements of the heating elements in the furnace, the guide rails, etc. are normal, check whether the sealing surface of the furnace door and the sealing strip are normal, otherwise replace and other emergency treatment.
- 4.3.1.6 Check the water ring pump and the water ring pump operation is carried out according to Appendix C.
- 4.3.2 into the furnace
- 4.3.2.1 When the loading car is used, the graphite floor of the product must be kept up and down and left and right aligned with the graphite track in the furnace. It is confirmed that the charging height must be within the furnace sintering space. It is best to use a tape measure before each furnace.
- 4.3.2.2 Each batch of extruded and dry bag products shall have a small sample. During the process of entering the furnace, it shall be ensured that the boat and the compact cannot be in contact with the graphite sintered box, otherwise it should be adjusted accordingly.
- 4.3.2.3 Push in the compact, check it again, and cover the front and rear sintered box cover without any error.
- 4.3.2.4 Clean the door seal and sealing surface with clean hexane, then apply a layer of vacuum grease to clean the paraffin in the wax collecting tank, close the furnace door, and return it to the wet grinding group with the remaining hexane.
- 4.3.2.5 Close the front and rear furnace doors, pay attention to the smooth movement when closing the door, and finally check whether it is closed.
- 4.3.3 Opening the furnace
- 4.3.3.1 Select the sintering procedure according to the grade of the product.
- 4.3.3.2 Vacuum the furnace, when the furnace vacuum <150m (20pa), close the vacuum valve and dewaxing valve. If the vacuum leakage is less than 500m (67pa) after one minute, the leakage will

pass and the heating power can be sent. Start the automatic program run. Otherwise, the furnace door must be reprocessed and then vacuumed. If it is seriously leaked, it should be promptly notified to the mechanic.

- 4.3.3.3 After the dewaxing starts, adjust the flow rate of N2 or Ar gas so that the pressure difference between 300~8000m (40~1064pa) is maintained inside and outside the sintering box. During the operation, the AVS sintering original record is completed, below 1000 °C. Record once at ~40min, once every 1000~30min at 1000°C.
- 4.3.3.4 Pay close attention to the change of vacuum in the furnace, and start the washing function in time with blockage.

Process requirements:

- 1 Dewaxing stage: $100 \,^{\circ}$ C $\sim 450 \,^{\circ}$ C; vacuum; sintered box 5000 m (665 pa), furnace within 12000 m (1596 pa).
- 2 pre-burning degassing stage: $800 \,^{\circ} \,^{\circ}$
- 3 sintering stage: $1350 \,^{\circ}$ C $\sim 1500 \,^{\circ}$ C; pressure: after warming up to $700 \,^{\sim}$ 735 psi ($4.8 \times 106. \,^{\sim}$ 5.2 \times 106. pa). Beyond this requirement, report to the technician.
- 4.3.3.5 Sudden external power outage during operation, first convert the cooling water to the urban tap water system, reset the electrical switch, and resume operation after the power is restored.
- 4.3.3.6 When an alarm occurs during operation, first silence the sound, and then find out the cause of the alarm. If it is a water alarm, check whether the water level of the cooling water is normal. If the magnetic flow rotor is stuck by mechanical impurities, it must be removed and cleaned in time to restore it to normal.
- 4.3.3.7 Start the gas system before pressurizing, open the gas storage valve, and make the body system active, ready for pressurization.
- 4.3.3.8 After the end of the program, make a record on the AVS Sintering Furnace Original Record every 40~80 minutes. The temperature and pressure should be recorded. When the furnace temperature is cooled below 800 °C, the fan can be started and cooled to below 150 °C. The gas recovery system can be started to recover Ar, and the gas in the furnace is recovered to 50~100 psi (3.4×105.pa~6.9×105.pa) for venting and venting, and special treatment is additionally carried out.

4.3.4 Released

- 4.3.4.1 After the gas recovery is completed, after the residual gas is emptied, open the manual venting valve to open the furnace door.
- 4.3.4.2 The sintering furnace is discharged from the furnace with a special material. When the furnace is discharged, the truck should be pushed into the unloading position, locked, and the track of the skip should be completely aligned with the upper and lower sides of the furnace rail, and the sintered product will be pushed onto the skip slowly. And fix the graphite bottom plate, wear protective products when handling, pay attention to safety.
- 4.3.4.3 When the extruded sintered product is discharged, the card must be placed in the tray. When other sintered products are released, the card should be transferred with the product.
- 4.3.4.4 After the furnace is finished, close the furnace door and vacuum the furnace.
- 4.3.4.5 If the furnace is not opened for a long time, cut off the control power and turn off the water, electricity and gas.

- 4.4 Operation of vacuum sintering furnace
- 4.4.1 Preparation and inspection
- 4.4.1.1 Check if the water system is normal. The general inlet pressure range is 0.15~0.40Mpa (GCA: 20~40psi).
- 4.4.1.2 Check if the compressed air supply is normal, the pressure is generally 0.4~0.7Mpa (GCA: 70~110psi).
- 4.4.1.3 Open the furnace door and check whether there are any abnormalities in the graphite heating parts, thermocouples and furnace door sealing surfaces in the furnace. If there are any problems, report the relevant personnel in time.
- 4.4.1.4 Check the vacuum pump set and oil level. If it is normal, start the mechanical pump, open the gas vibration valve, preheater vibration drainage, mechanical pump is replaced by water ring pump, then check the water ring pump, operate the water ring pump according to Appendix C. . 4.4.2 into the furnace
- 4.4.2.1 Squeeze and dry bag products should have small samples for each batch. Carefully move the product boats into the four bins, mark the corresponding cards, store them in the designated position, and cover the sintered box cover.
- 4.4.2.2 Clean the paraffin in the wax canister, handle the seal and put it back in place.
- 4.4.2.3 Clean the sealing surface of the furnace door with hexane or alcohol, then apply a thin layer of vacuum grease on the sealing strip and return it to the wet grinding unit with the remaining hexane.
- 4.4.2.4 Close the furnace door locking fixture.
- 4.4.3 Opening the furnace
- 4.4.3.1 According to the product and process requirements, the corresponding sintering procedure should be called up. If the special program section needs to be changed, it must be carefully confirmed.
- 4.4.3.2 Start the vacuum pump, open the vacuum valve, and vacuum the furnace.
- 4.4.3.3 When the vacuum degree of the furnace is less than 50pa, the vacuum valve is closed. If the vacuum leakage does not exceed 50pa within one minute, the leak detection will pass, and the valve can be turned to water, and the heating power supply can be sent to start the automatic program.
- 4.4.3.4 During the operation, record in the "vacuum sintering original record", record once at 1000 °C for 20~40min, record at 1000 °C for 20~30min, pay close attention to the furnace operation, hot water temperature is running 2 After ~3 hours, it reaches 50~70 °C. After dewaxing, the cooling water temperature must be between 5 °C and 40 °C. Timely handling general alarms. If there is a sudden external power outage, you must turn the valve in time, turn the tap water, cut off the control power supply, stick to the post, and wait for the power to recover. When other serious anomalies occur during operation, the furnace should be shut down in time. After the power is cut off, Ar or H2 gas protection should be provided to the furnace, and the relevant personnel should be notified to deal with it.
- 4.4.3.5 After the program is finished, evacuate to $1000\,^{\circ}$ C and close the valve to charge Ar. The Ar gas is generally at $0.02{\sim}0.04$ Mpa ($4{\sim}15$ inHg), forced cooling, and every $40{\sim}80$ minutes after the program is finished. The degree of vacuum and temperature should be recorded in the cooling stage above $1000\,^{\circ}$ C, and the degree of vacuum and temperature should not be recorded below $1000\,^{\circ}$ C.

- 4.4.3.6 Start the fan when the furnace temperature is cooled to 800 °C. When turning on the fan, first open the front and rear damper flaps.
- 4.4.3.7 Cool down to 75 °C to turn off the fan.
- 4.4.4 Released
- 4.4.4.1 Vacuum sintering furnace When the temperature of the furnace drops to 75 °C, the manual inflation valve is opened. After the pressure inside and outside the road is balanced, the furnace door can be opened.
- 4.4.4.2 When the vacuum sintering furnace is baked, wear protective equipment and pay attention to safety.
- 4.4.4.3 When the extruded sintered product is discharged, the card must be entered into the tray. When the other sintered products are discharged, the card should be transferred with the product, and the inside of the furnace should be cleaned and kept clean. If the electrode and carbon felt are found to have significant oxygen, measures should be taken to deal with the group's report.
- 4.4.4.4 After the furnace is finished, close the furnace door and vacuum the furnace.
- 4.4.4.5 If the furnace is not opened for a long time, cut off the control power and turn off the water, electricity and gas.
- 4.5 Unloading and sandblasting
- 4.5.1 Unload the qualified sintered products, sandblasting, and put them into the wooden box. The alloy in the wooden box should be consistent with the transfer card. The products after sandblasting should be non-sticky graphite, no oxidation, and the surface color is uniform. Process parameters: corundum particle size is $355{\sim}250\mu m$ ($40{\sim}60$ mesh), pressure pressure is $0.2{\sim}0.8Mpa$.
- 4.5.2 In the process of unloading, sandblasting and cartoning, it should be handled gently to prevent product collision, try to avoid collision products, and the card should be transferred with the product, so it should not be confused.
- 4.6 Treatment of the boat after use
- 4.6.1 Place the used boat near the air vent, use a brush to clean the surface debris and paint, especially remove the dirt in the groove.
- 4.6.2 Apply a special coating on the surface of the boat. The cover should be slightly thicker. The paint must be brushed or evenly sprayed. No blank should be left.
- 4.6.3 Place the boat with the paint in the oven and dry it. The temperature is set to $4\sim8$ grids and kept for $1\sim2$ hours.
- 4.6.4 Take the dried boats out on the boat rack and sort them in neat rows.
- 4.7 New boat handling

Before being used in production, the new boat must be treated with de-F, S, P and other impurities and de-melted melt by boiling in boiling water at $100\,^{\circ}$ C for 24 hours, then washing with tap water, and in an electric drying oven. The inner drying, drying temperature is set to 4 to 8 grids, and then calcined in a vacuum furnace at $1500\,^{\circ}$ C for 100 to 150 minutes.

4.8 Sintering of production identification samples

The sintering system is generally carried out according to the sintering system of the same brand of large quantities of products. Without the same system, it can be sintered with the furnace.

Management focus: After the furnace is completed, the actual sintering system is written on the "Production Identification Sintering Instruction Card" that has not been completed by the appraisal team.

Water ring vacuum unit operation steps:

Sintering process requirements:

- D.1 1~ vacuum dewaxing sintering furnace;
 - A) Dewaxing stage: room temperature ~ 750 ° C, furnace vacuum ≤ 100 pa.
 - B) Pre-burning stage: >750°C~1250°C, furnace vacuum ≤100pa.
 - C) Sintering stage: >1250 ° C ~ sintering temperature, furnace vacuum ≤ 100 pa.
 - D) Report to the technician or team leader beyond this range.
- D.2 The remaining vacuum dewaxing sintering furnace:
 - A) Dewaxing stage: room temperature ~ 750 ° C, furnace vacuum ≤ 180 pa.
 - B) Pre-burning stage: >750°C~1250°C, furnace vacuum degree ≤120pa.
 - C) Sintering stage: >1250 ° C ~ sintering temperature, furnace vacuum ≤ 300 pa.
 - D) Report to the technician or team leader beyond this range.
- D.3 AVS pressure sintering furnace
- D.3.1. No atmosphere adjustment process
- A) Dewaxing stage: room temperature ~ 750 ° C, vacuum degree: within the sintered box 5000M (665pa), the furnace is less than 12000M (1596pa).
- B) Pre-burning stage: >750°C~1150°C, vacuum degree: within the sintered box 2000M (226pa), the furnace is less than 4000M (452pa).
- C) Vacuum sintering stage: >1150 ° C \sim sintering temperature, vacuum degree: within the sintering box 4500M (595pa), furnace 8000M (1070pa) or less.
- D) Pressure sintering stage: After the end of the heat preservation, the pressure is increased to $700\sim735$ psi $(4.8\times106.\text{pa}\sim5.2\times106.\text{pa})$
 - E) Regulating valve: 0~100%.
 - F) Gas flow rate: 0~201/min.
 - G) Reporting to the technician or team leader beyond this range.
- D.3.2. No atmosphere adjustment process
- A) Dewaxing stage: room temperature ~ 750 ° C, vacuum degree: within the sintered box 5000M (665pa), the furnace is less than 12000M (1596pa).
- B) Pre-burning stage: >750°C~1250°C, vacuum degree: within the sintered box 5000M (565pa), the furnace is less than 10000M (1330pa).
- C) Vacuum sintering stage: >1250 ° C \sim sintering temperature, vacuum degree: within the sintering box 4500M (595pa), furnace 8000M (1070pa) or less.
- D) Pressure sintering stage: After the end of the heat preservation, the pressure is increased to $700\sim735$ psi $(4.8\times106.pa\sim5.2\times106.pa)$
 - E) Regulating valve: 0~100%.
 - F) Gas flow rate: 0~301/min.
 - G) Reporting to the technician or team leader beyond this range.

Approved by: Zhaohui LIU Prepared by: Liyun Yun Xing Wang
Checked by Technical Dept: Yizhong Zhang and Yongshen Gong
Checked by Sub-Factory: Zhengchen Xu and Zhengdao Xiao
August 1, 2019 Release August 1, 2019 Effective

Hunan WISE New Material Technology Co., Ltd. Release