Debinding Options For Hard Metals

Scott K. Robinson, Centorr/Vacuum Industries, Nashua, N.H.

Binders used to hold powders together in various metals and ceramic forming processes, such as isostatic pressing, hot pressing, powder compaction and injection molding, must be removed completely before sintering. A number of options are available to handle this operation efficiently and cost effectively.

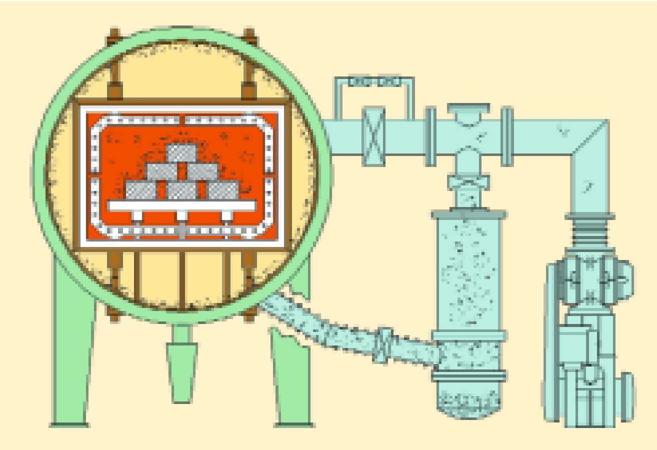
Powder processing formulations include the use of binder and lubricant additives to help in handling the powder compacts and in processing the compacts. Commonly used lubricants and binders in tungsten-carbide (WC) hard-metals processing are paraffin wax and polyethylene glycol (PEG). In addition, carbon-containing binder systems are used in ceramics and hard metals to supply carbon content during sintering to achieve a stoichiometric formulation. While binder and lubricant manufacturers are continually working to optimize these additives, the parts producer is working to ensure that the additives are removed before producing the final component, as it is necessary to remove all traces of binders and lubricants in a debinding step prior to reaching final sintering temperatures. Common debinding methods used to handle WC are vacuum dewax, Sweepgas and positive-pressure flow through (using inert gas or hydrogen).

Discoloration on chamber walls indicating cracked wax

Vacuum dewax

Developed more than 20 years ago, vacuum dewax was the first process to allow debinding and sintering of hard metals in one furnace, saving production time and capital expense. By using a vacuum and leveraging the low vapor pressure of the paraffin wax binder, a fast, efficient debinding process was developed compared with positive-pressure thermal debinding.

Vacuum dewax uses low vacuum levels (typically 10^{-1} to 10^{-2} torr) and low temperatures to evaporate paraffin wax off the load. By bringing off the wax at temperatures below their cracking point, there is less chance of unintentional carbon pick up. There is no retort in the furnace, so wax by-products must diffuse through the graphite felt hot-zone insulation to get to the outer water-cooled shell, where the wax immediately condenses and solidifies on the casing. To reduce this buildup, later units were fitted with both cold and hot water flow in the jacket so the casing could be run above 60°C (140°F), causing the solidified wax to liquify and flow to the bottom of the tilted chamber where it runs into a wax cup reservoir.



Vacuum dewax system used to delube hard metals

At the end of each process cycle, a hot water flush is run on the chamber walls to make chamber cleaning easier and faster. The balance of the wax leaves the furnace chamber through the roughing-vacuum manifold to a wax condenser, and the exhaust passes through to the pumping system. The vacuum level is not controlled in any way other than to operate at the ultimate vacuum of the pumping system (typically 10^{-1} to 1 torr). For different binders, the wax condenser can be replaced with other types of traps, such as chilled liquid-nitrogen vessels with baffled plates.

The main disadvantage of the vacuum-dewax design is that evaporating wax must flow past the heating elements and furnace insulation to exit the hot zone. Because the heating elements can be up to 100°C (180°F) higher than the load temperature, cracking of the paraffin wax can occur as wax-laden vapors contact the elements. This forms a carbon-rich environment, which usually is considered in the part formulation.

The other major disadvantage is that over time, wax infiltrates the graphite-felt lining. Later in the process cycle during sintering, the insulation is subjected to much higher temperatures where the condensed wax becomes gaseous and again cracks due to the high temperature, adding more carbon to the system. This is typically not desired.

The felt also stiffens over time as the carbon particulate solidifies in the felt increasing its density, eventually reaching the point where its thermal conductivity increases causing it to becomes a less effective insulator. Eventually, the hot zone will no longer be able to achieve desired ramping rates with a full load during sintering and may even fail to attain maximum temperatures.

Sweepgas™ for hard metals

Because industry wanted to improve productivity and lower maintenance costs, Sweepgas technology became the next generation debinding technique. In this design, a graphite retort is inserted in the hot zone in an effort to retain all the off-gassing taking place inside the hot zone and to create a barrier through which the paraffin cannot return. A special graphite pump-out tube with optical baffles (to prevent heat loss through the opening) connects the retort directly to the condensing trap and pumping system.

CFC (carbon-fiber composite) tubes can be used to lower the heat loss out this port by as much as 25%, improving furnace temperature uniformity. To improve the wax collection and speed up debinding time, an argon sweepgas is used as a carrier gas.

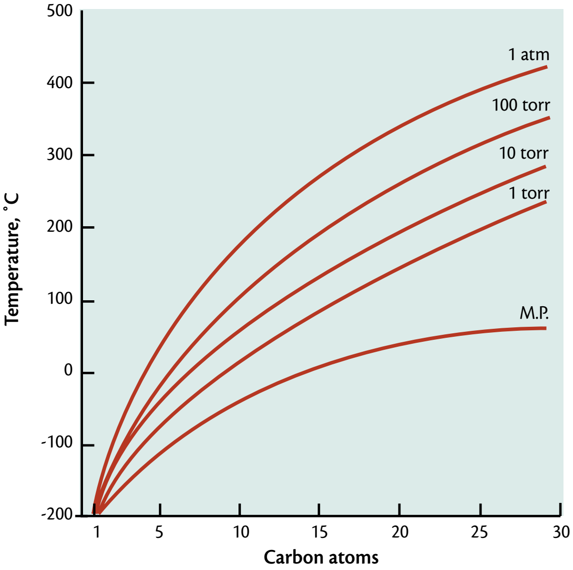
Because of the added gas load, vacuum levels of 10^{-1} torr are no longer possible. The system introduces a measured flow of inert gas into the chamber, which diffuses into the graphite retort "sweeping away" binder off-gassing. The system is designed to operate at pressures of approximately 8 torr in the retort and 10 torr in the chamber, trying to ensure a 1 to 2 torr differential between chamber and retort. This pressure head outside the retort minimizes the escape of off-gassing wax vapor into the chamber. The binder-laden gas is pumped out the bottom of the retort through the graphite pump-out tube. A closed-loop controller takes the pressure signal input from the retort capacitance manometer and the output automatically adjusts an electromechanical modulating valve to regulate pumping speed and provide constant-pressure control within the retort during debinding. The binder is trapped in the condensing system before reaching the vacuum pumps.

While sweepgas furnaces still include hot- an cold-water manifolding, newer units don't require hot-water circuits on the chamber due to the efficiency of the debinding system, but only for the wax condenser to allow for cleaning at the end of a cycle.

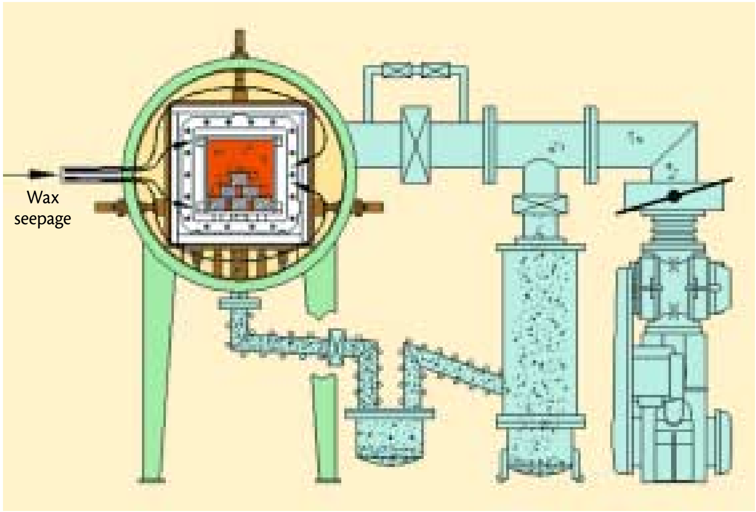
The wax condenser is the heart of the Sweepgas system. Older designs used the Lutts and Anderson-style wax condenser, where low-pressure carrier gas plus binder first enter an empty wax pot through a heated/insulated debind manifold. When the wax molecules enter this large volume cavity, a portion immediately condenses due to the change in pressure, leaving a majority of the wax condensate in the pot. The remaining vapor passes through a heated bronze wool mechanical filter where additional wax condenses and drips into the pot below.

To remove the smaller diameter wax particles, filters will not work unless they are so restrictive that they reduce the pumping conductance excessively. After the bronze wool filter, the gas passes through a restrictive orifice causing a vortex-type flow around a water-cooled cylindrical cone. Particles entrained in the gas flow will flow to the outer walls due to their higher momentum. If the inner and outer walls of the condenser are water cooled, the cyclone effect causes significant condensation of all but the smallest wax particles.

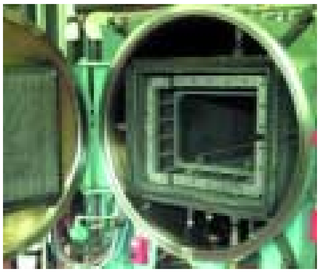
The vapor then passes through an optically dense path of water-cooled "D" baf-



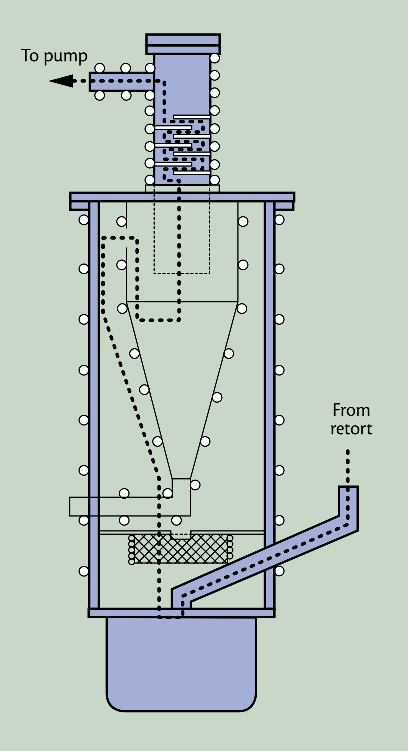
Vapor pressure and melting point of normal C1 to C29 paraffin waxes



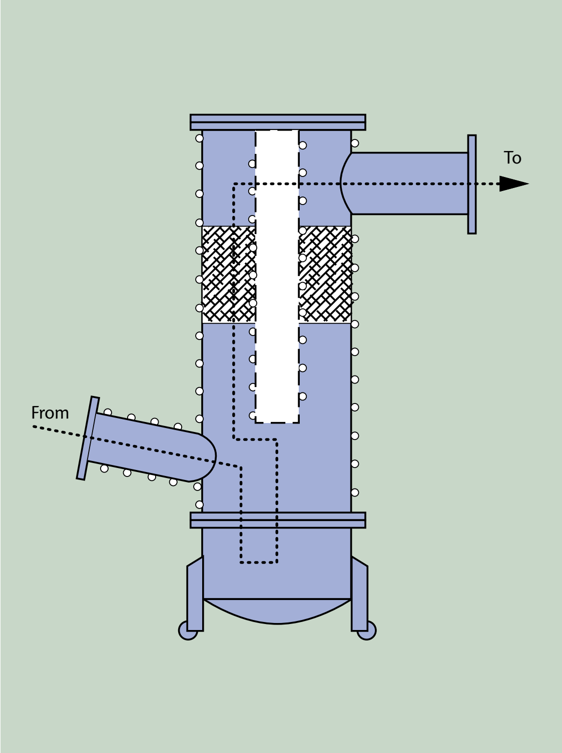
Sweepgas process used to delube hard metals



Chamber walls basically free of wax build-up using Sweepgas debinding process



Lutts and Anderson Sweepgas condenser design



Griffin Sweepgas condenser design

fles, where even small particles condense and adhere to the chilled surfaces. For larger hot zones, a newer modified sweepgas condenser was developed, which allows for a reasonable pressure differential in larger size hot zones without requiring excessive sweepgas flows and correspondingly larger pumping systems.

The modified condenser is similar to the Lutts and Anderson design, except the cyclone chamber is replaced with dual mechanical filtration media. The first chamber contains a basket of lightweight, high surface-area aluminum stampings, called pall rings. The upper basket is filled with a finer stainless steel wool medium. Both baskets are removable for easy cleaning using a heat torch, steam or solvent bath. Condensers are cleaned at the end of each cycle by running a hot water flush on the jacket, causing trapped wax to liquify and run into the pot below. The bottom pot should be emptied after each cycle or at least weekly based on the percentage of binder in the system. The condenser body typically is cleaned once each quarter.

Only with efficient wax trapping can reliable, consistent pump performance be expected, with little binder residue entering the pump chamber and contaminating the oil. Time between oil changes typically is between 30 and 50 cycles for mechanical pumps and up to 200 cycles for the roots blower. More expensive pumping designs, such as liquid ring and dry pumps, can be avoided for this process due to the efficiency of the trap.

The higher efficiency of Sweepgas debinding compared with vacuum degas is shown in the following table:

	Vacuum dewax	Sweepgas
Collection in chamber	69%	<2%
Collection in condenser	15%	95%
Total	84%	97%

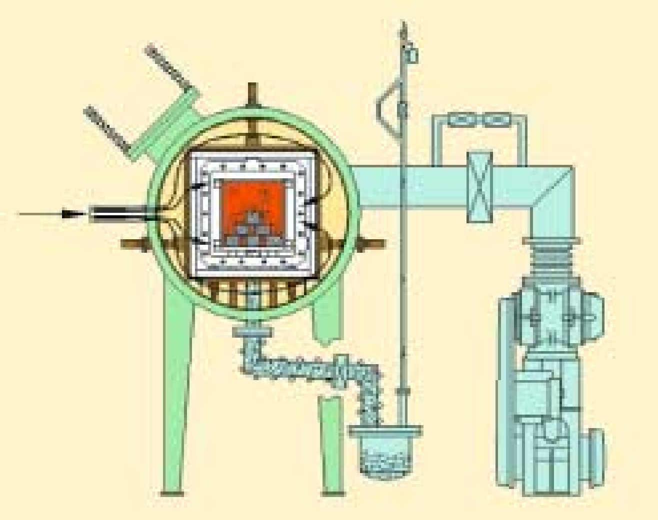
The primary advantage of the Sweepgas technique is the lower amount of wax buildup in the furnace chamber, which must be cleaned after every run. Hot-zone life also is improved due to preventing wax infiltration/reaction with the insulation and elements.

However, caution should be taken before considering a change from vacuum dewax to sweepgas. While the advantages are worthwhile and cost effective, many carbide producers find they have to reformulate their powder mix due to the lower carbon contents resulting from the more efficient binder removal.

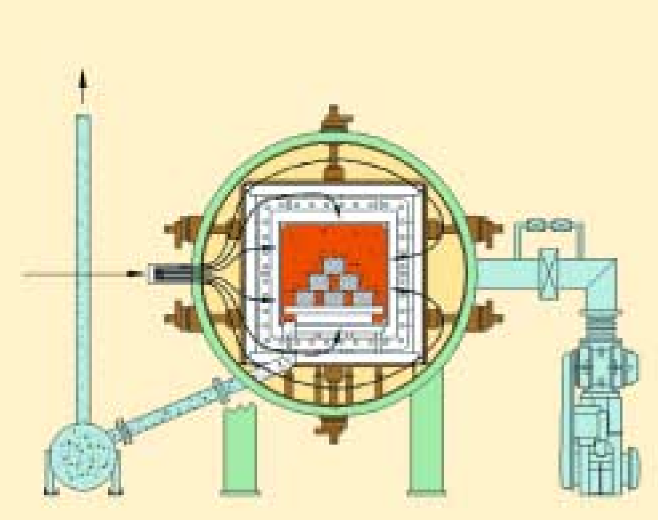
Positive-pressure flow-through debinding

Some binders do not lend themselves to vacuum debinding. The materials have high vapor pressures, which make the use of vacuum ineffective, or they decompose into tar-like, sticky phases, which would destroy vacuum pumping systems in a short time. Here, a positive-pressure environment is desirable.

With this design, a flow of inert gas or hydrogen process gas is used to purge the chamber of binder buildup and related process off-gassing as the hot zone is gently heated up. The flow-through gas enters the retort and sweeps across the parts, carrying the binder vapors out the bottom through a pump-out tube to the debind manifold, which is heat traced and insulated to prevent binder condensation. The gases enter an empty binder pot below the cham-



Positive-pressure gas flow-through system for debinding



Positive-pressure gas flow-through system with incinerator

ber, where most of the binder condenses, and the balance flows out an exhaust tower mounted off the pot through a propane/natural-gas operated burn-off. The manifolding also is heat traced and insulated.

When using hydrogen process gas (common for polyethylene glycol, or PEG, binder systems), the hydrogen exhaust tower is mounted off the binder pot instead of the top of the chamber. While this design is effective for small amounts of binder, it is not designed for quantities over 4 to 5%. The propane tower will combust most gases, but excessive particulate will clog the orifice or pass through the flame tower uncombusted. The binder will then cool in the upper air stream, condense, and rain back down on the furnace chamber.

To combat this problem, a variation of this system has been developed for nonoxide ceramic sintering. Gas is flowed into the retort to entrain binder/process offgases and direct it out the pump-out tube, while gas flow into the chamber prevents process gas leakage out of the retort (where it can attack the graphite hot zone elements and insulation) and minimizes binder condensation on the cool chamber walls.

The binder pot and tower are replaced with an inert gas-purged, full-size thermal oxidizer to manage volatile organic compounds (VOCs) and to meet state and federal environmental regulations. The robust design is required because the binder residue is corrosive to less expensive insulation liners.

Custom-designed "Griff" valves are used to seal the chamber during vacuum operation, plunge the orifice on an intermittent stroke to ensure minimal clogging, and to control the rate of exhaust of positive pressure flow-through gases. With this design, large binder contents, and tarry, sticky binders can be handled with proper maintenance.

The primary advantage of positive-pressure systems over vacuum debinding is the ability to "bathe" the parts in warm gas, which provides more uniform heating than radiation heating in a vacuum environment due to the thermal transfer from gas conduction and convection. **IH**

Selected references

- S. W. Kennedy and C.W.P. Finn, "Metal Injection Molded Part Debinding and Sintering in Vacuum Furnaces," *Powder Metallurgy Science&Technology,* 1990, Vol. 1, No. 3, p 37
- C.W.P. Finn, "Vacuum Binder Removal and Collection," *The International Journal of Powder Metallurgy,* Vol. 27, No. 2, 1991

For more information: *Scott Robinson is market manager, Ceramics and Powder Materials, Centorr/Vacuum Industries, 55 Northeastern Blvd., Nashua, NH 03062; tel: 603-595-7233; fax: 603-595-9220; e-mail: skr@centorr.com*