

TECHNICAL NOTE

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Performance Evaluation of Gas Atmosphere Delubing and Sintering in Sintervac® Furnaces

In the following report I Fred Andersen of GCA/Vacuum Industries, Somerville, Mass, describes the use of low pressure sweep gas for delubrication of tungsten carbide preforms. This method allows the removal of paraffin lubricant with minimal or no effect from breakdown products. It also offers the carbide sintering specialist the potential of optimising the overall delube/presinter/sinter cycle while simultaneously achieving a consistently high and readily controlled quality level with a minimum of furnace maintenance.

Many factors affect the quality of tungsten carbide parts and the productivity of the sintering process used during their manufacture. A major influence during the overall sintering process is that portion of the cycle in which lubricants are removed from the pressed parts. The lubricant has only a transitory purpose, playing no role in the final structure of the product. Its constituents, however, may have adverse effects on both the carbide product and on the production equipment. While other lubricants have been the subject of study, this report focuses on the delubrication of carbide parts pressed with paraffin using a partial pressure carrier gas or vacuum in a batch operation.

THE LUBRICANT AND ITS PURPOSES

Paraffin, a byproduct of the petroleum industry, is the most commonly used lubricant in carbide manufacture. The lubricant is applied to the powder prior to forming or pressing and serves no purpose after this. Paraffin is removed from the heated parts by one of three principal methods:

- (1) Flowing hydrogen blanket, above atmospheric pressure.
- (2) Vacuum
- (3) Sweep gas, flowing N₂, or Ar, at subatmospheric pressure.

Polyethylene glycol is also used as a lubricant. This material, although a good lubricant and a biodegradable material, imposes restrictions on the user in that it is necessary to use hydrogen during delubrication. Therefore, this report will be devoted to methods of removing paraffin in either a vacuum or a partial pressure sweep gas.

PRINCIPLES OF PARAFFIN REMOVAL

Removal in Vacuum

During paraffin wax removal in vacuum - in a conventional 'cold-wall' resistance heated furnace - several things either do or can take place:

- (1) As heat is applied to the part, the paraffin is liquified and moves to the part's surface from which it vaporises. In the case of extruded parts or similar parts

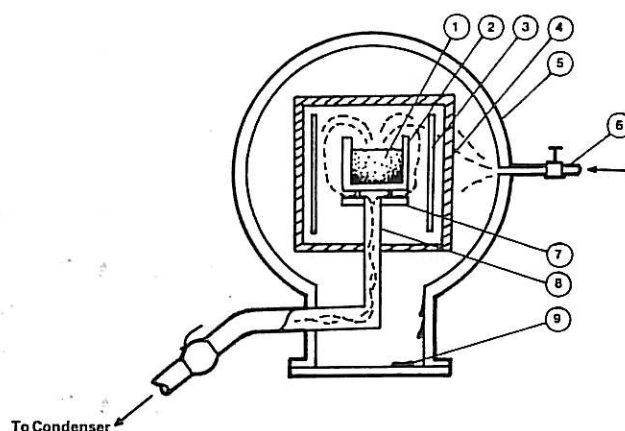
with high wax content, a certain amount may run off as a liquid.

- (2) The wax vapour travels through the porous graphite felt insulation and is deposited on the warm chamber wall where it runs off into the wax condensing system.
- (3) If the wax vapour, which represents the bulk of the residual atmosphere during delubrication, is heated above 400C, free carbon will be produced which may affect the product adversely. (Some users report that a certain amount of carbon liberated from the paraffin may compensate for carbon deficiency caused by reactions with residual oxygen).

- (4) The paraffin from the parts must be removed from the furnace interior and the vacuum pumping system, thus requiring maintenance and causing potential operating problems.
- (5) Heating too fast during delubrication may cause high enough gas pressure levels in the parts to burst them.

Removal in Sweep Gas

A carrier gas or sweep gas at sub-atmospheric pressure has been used for some time by several manufacturers to remove the transport lubricant away from the product. If sweep gas is used during delubrication, additional variables resulting from a more complex process are introduced. On balance,



Test with sweep gas:
 (average of 11 identical runs)

Load: paraffin 800 grams
 Gas: nitrogen 120 l/hr
 Heat: ambient - 600C in 25 min
 Collection in condenser: 97.8%
 Collection in chamber: .6%
 Collection rate total: 98.4%
 Pressure range: 11-20 mbar

1 Lubricant - paraffin
 2 Crucible - graphite
 3 Heater - graphite

4 Insulation - graphite felt
 5 Chamber - warmed by hot water
 6 Sweepgas inlet
 7 Hearth
 8 Pump out - graphite
 9 Lubricant accumulated in chamber

Test with vacuum only:
 (single test only)

Load: paraffin 800 grams
 Heat: ambient 600C in 2.5 min
 Collection in condenser: 15%
 Collection in chamber: 69%
 Conditions otherwise same as with sweep gas

FIG. 1 Test set up for evaluation of sweep gas and condenser

reason, more rapid heating rates during certain portions of the cycle are possible.

Maintenance -

Minimising paraffin contamination on furnace parts and system components contributes to reduced maintenance.

FURNACE DESIGN CONSIDERATIONS

Recognising the benefits of effective delubing for the carbide sintering process, it is important to design and build furnaces with adequate attention to the requirements of the delubrication portion of the cycle. Evaluation and test of many designs has led to the conclusion that the following features are most important (Fig. 2):

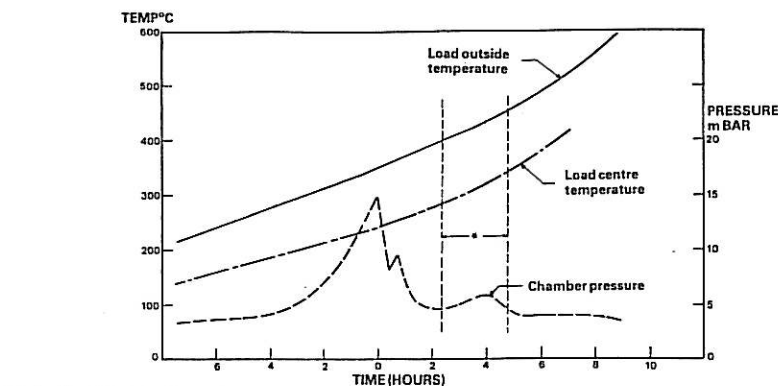
- A direct pumpout connection from the hot zone leading directly to a valved condenser system. Graphite was found to be a suitable material, allowing modular construction for design flexibility and adjustment.
- Provision for a work box of user's choice. Box may be permanently installed or may be considered part of the load. A hole located in the bottom plate mates with the kragen on the pumpout pipes if a movable box is being used. (The kragen is a special collar which provides a tight coupling).
- Provision for a gas system which allows flow control, typically about 200 litres per hour at 3-20 millibar chamber pressure. (For pressure readings, thermocouple gauges were found to be unreliable. A diaphragm type with dial or electronic read out is preferred).
- Provision for load centre thermocouples.
- A heating system (hot water or electric) to provide adequate temperature to avoid clogging of lubricant in manifolding and certain portions of the condensing system.
- A system for separating paraffin from the sweep gas (see following section).

CONDENSING SYSTEM FOR SEPARATION OF SWEEP GAS AND LUBRICANT

A good condensing system by itself cannot do anything for the quality of the product, but an effective system is essential if the user wants to operate at full rate of production without having to service the pumps frequently. Sweep gas operation, in particular, requires a special condensing system, so that the gas will not collect previously condensed lubricant and pass it on to the pump.

It was determined that the typical hot liquid film or hot packed bed condenser would not work with sweep gas, because the paraffin would be re-entrained in the gas and carried to the pump. After a series of experiments, a system was designed that is working well. It has the following features (Fig. 2):

- Primary collection takes place as the mixture cools and the paraffin precipitates in the first stage collector.
- Additional paraffin is repelled by a heated filter and falls into the second stage collector.
- In an expansion chamber, the remaining particles are cooled and dried, passed



Typical load: 300-400 Kg, 1.5% paraffin
Sweep Gas: Argon at 200-300 litres/hr
*Note: Potential for cracking in this range. Paraffin present while temperatures in load outside go from 400-460°C.

FIG. 3 Representative temperature and pressure response in a sintering furnace during lubricant removal with sweep gas

- through a centrifugal separator and then into a series of flow line interruptors.
- A water/electric, cold/hot temperature control system allows temperatures to be kept to a minimum at all stages in order to prevent re-entrainment without the risk of clogging.
 - By-pass valves are used between furnace internal pumpout manifold and condenser and between condenser and pump so that the condenser can be isolated (while the furnace is in the cooling mode for example), stripped and emptied in preparation for the next cycle.
- Systems with these features have been used in connection with GCA's sweep gas test programme. Examination of manifolding and pumps has shown that, under regular circumstances, almost no solid material passes through the system. Possible gaseous components will pass through and may account for lower percentage collection rates in certain cases when severe cracking of the lubricant has occurred.

CYCLE CONSIDERATIONS

Furnace cycles established for sweep gas de-lube generally follow the same time/temperature programmes as cycles for vacuum de-lube. Some time saving is possible when paraffin removal is enhanced by the carrier gas. Most test runs emphasised zero effect on carbon content from the lubricant. In other words, the goal was to achieve high material quality, totally avoiding the contribution of carbon from paraffin cracking products.

Carbide manufacturers use time/temperature programmes which are designed for their particular product, load size, etc. The following are, however, some cycle parameters which apply universally when sweep gas is used.

After loading and closing, the furnace is pumped down through the main valve leading via a manifold to the pump/blower. During this time, hot water is run through the chamber and other sections of the system in which paraffin build up is not wanted. Note that, at this time, the paraffin condensing system is inactive.

When the pressure has reached the nominal blank off level, the main valve is closed and the condenser system isolating valves are

opened. Sweep gas flow is started and furnace power is applied. Gas flow of argon or nitrogen during tests has been from 50-500 litres/hr at pressures from 2 to 60 millibars. Actual values for specific furnaces and loads will vary according to application and furnace and pumping system characteristics.

Furnace temperature is raised at a controlled rate so that the outer portion of the load will reach lubricant melting point, but the hot zone components will not reach 400°C in the possible presence of paraffin vapours. Use of multiple thermocouples is a great help in achieving this. A retractable thermocouple for reading load interior temperature is recommended in addition to the control thermocouple whose reading can then be related to furnace maximum temperature. Poor temperature control, resulting in 'On-Off' operation of heating elements, can cause the heaters to reach transient temperatures high enough to crack paraffin vapour.

The heat penetrates the work load and the paraffin liquefies. Upon reaching the load surface, the lubricant is entrained in the gas and swept into the condensing system. As the temperature continues to increase, the paraffin is transported in vapour form and its pressure now adds to the base pressure of the sweep gas. It is important that pressure measurements are made with a diaphragm type gauge with either a dial or electronic readout.

The combined paraffin/sweep gas pressure will rise as more paraffin is released while the centre of the load heats. If a load centre thermocouple is being used, it can be used to determine when all the lubricant has been released. At this temperature there should be a corresponding drop in pressure. By observing the load centre temperature and total pressure, the conclusion of delubing can be established. Subsequent increase of temperature to the presinter level may be done with sweep gas until no evidence of lubricant entrainment remains. The sweep gas flow is then stopped, but the furnace is pumped through the condensing system for some time in order to maximise removal and collection of condensables from the pores in the parts. Then the condenser is isolated and the main valve opened. A typical experimental cycle illustrates the above parameters (Fig. 3).

Observation and measurement of lubricant recovered can be related to product quality. Substantial amounts of tar, oil, and grease in the collected lubricant along with low recovery rates were observed in several cases in which product evaluation indicated it was exposed to excess carbon.

however, the results with this process are potentially more consistent. Investigations conducted in the GCA laboratory as well as in several experimental field installations, have utilised argon and nitrogen as sweep gases. The following observations define the basic sweep gas paraffin removal process:

- (1) As heat is applied, paraffin is liquified and transported to the surface. Wax vapour is entrained by the gas and carried with it and in its direction of flow.
- (2) The gas/paraffin mixture is pumped directly from openings in the hot zone (or from corresponding openings in a work container) into a lubricant separating system. Such a system must include a wax condenser specifically designed for use with sweep gas.
- (3) In contrast to vacuum delubing where the paraffin vapour travels to cover the entire hot zone and chamber, the directionality of the sweep gas flow restricts escape paths for the paraffin through the hot zone insulation (which would be in directions other than the gas flow). Tests indicate that with direct pumping from the hot zone, there is substantially less paraffin collected on the chamber wall with sweep gas than with vacuum alone. (Fig. 1)
- (4) What effect overheating a gas/paraffin mixture has on carbon formation and soot deposition vs. paraffin vapour only (i.e. in vacuum) has not yet been quantified. It has been noted that overheating work while paraffin is still present does produce limited quantities of soot.
- (5) The gas/paraffin mixture is directed through several separating/condensing stages on the way to the vacuum pump. A multi-stage system with a dry final stage was used for the experiments. It was possible to account for more than 95% of the original theoretical wax content with no evidence of the balance in either the manifold or the pump.

QUALITY, PRODUCTIVITY AND MAINTENANCE IMPLICATIONS OF THE DELUBRICATION PROCESS

The delubing process directly influences the final quality of the product. The process also takes up a big portion of overall cycle time. Lastly, it produces effluents which can contribute heavily to maintenance.

The chief objective of the delubricating process should be to remove the lubricant, yet maintain the proper proportions of tungsten, carbon, cobalt and additive constituents. The delube equipment should include provisions for separating the lubricant from the gas being pumped so that no trace of lubricant can be found in the pumps or associated manifold.

As shown above, the traditional batch furnace with vacuum delube allows the paraffin to freely cover the entire chamber, coating and saturating insulation materials, chamber walls, etc. If ample delube time is available, a suitable interval can be allowed for the paraffin to escape before the temperature is raised to the point where cracking may occur. In many cases, however, one or more of the following things may happen, with accompanying effects on quality:

- (a) Too rapid an increase in heating element temperature, combined with exposure to paraffin, can result in chemical changes and release of carbon and other components detrimental to the product.
- (b) Heating elements raised to a higher temperature than required (this could be intermittent - the result of unstable control action at low temperatures) can overheat parts closest to them with resultant breakdown of lubricant on these parts plus potential internal pressure build up.
- (c) Parts on the outside of the work load may be heated to a temperature high enough to cause cracking of paraffin vapour being released from parts in lower temperature regions of the work load.

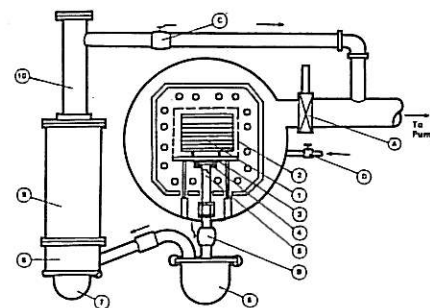
USING SWEEP GAS

Tests were run in the GCA laboratory in a vacuum furnace with resistance heating elements directly facing the work load to compare paraffin deposits on the vacuum chamber wall in sweep gas mode compared to conventional vacuum operation. The only modification to the standard furnace was the addition of a connection from the interior of the hot zone to the pumping line leading to the vacuum system (Fig. 1). The tests showed that, during straight vacuum operation, 69% of the paraffin condensed on the walls. With sweep gas, under the conditions shown, 0.6% of the paraffin was condensed.

These tests lent credence to the practice of inserting an additional barrier, one through which the paraffin cannot return, between the work and the heating elements. Such a barrier is termed a load box or work cell. If a work cell is used, it is obvious that the paraffin and the sweep gas can be pumped directly to a condenser system. It should be noted, however, that sweep gas techniques do not absolutely require the use of a work cell. Our experimental results have indicated that many of the benefits of using sweep gas may be realised without such a container. Among users, varied opinions exist on this subject.

When using sweep gas techniques for delubrication, some distinct operating characteristics have been observed:

- (a) The atmosphere during most of the sweep gas delube cycle is substantially inert gas, whereas in vacuum delube it is almost entirely vaporized paraffin.
- (b) The paraffin generally leaves the work as a vapour and is picked up by and carried away with the gas as a function of its flow rate.
- (c) The direction of sweep gas flow is from the outside to the inside, therefore directing the lubricant away from the chamber walls, furnace insulation and heating elements.
- (d) If a work container is used and if a pumping connection is made directly to it, the container itself can serve as an effective barrier between work and heating elements*. With inert gas flowing from the outside of the container, the possibility of paraffin contacting the heaters is drastically reduced.
- (e) The inert gas atmosphere offers some heat transfer benefits, especially when



- 1 Work
 - 2 Work box (alternative)
 - 3 Hearth plate/work box bottom with pump out
 - 4 Kragen - joins pump out tube to hearth plate opening
 - 5 Pump out tube - distribution system
 - 6 First stage collector - operates warm
 - 7 Second stage collector - operates warm
 - 8 Filter stage - operates warm
 - 9 Expansion and centrifugal separator - operates cold
 - 10 Flow line interrupter - operates cold (hot for stripping)
- A Main valve. Closed in delube cycle
 B Drain valve. Open in delube cycle. Closed in sinter
 C Bypass valve. Open in delube cycle. Closed in sinter
 D Sweep gas valve. Open during delube with sweep gas

FIG. 2 SWEEP GAS™ system for delubrication

the work load consists a large number of parts, each representing a barrier to radiative heat transfer.

EFFECT OF SWEEP GAS ON QUALITY, PRODUCTIVITY AND MAINTENANCE

The above characteristics of the sweep gas process have the following influences:

Quality -

- (1) Sweep gas delube offers conditions which result in reduced carbon generation and provide a consistent and controllable atmosphere.
- (2) Paraffin contamination on insulation is minimised and therefore should not become a problem during presinter and sinter segments of the cycle.

Productivity -

Using a load box with direct pump out, the heating element temperature is no longer the dominating factor regarding paraffin cracking. The container temperature becomes the significant factor. For this

* Carbide manufacturers use many types of trays, racks, shelves, etc, to support the work. Load containers have often been used or have been recommended by furnace manufacturers as a remedy for localised heating element effects and as barriers against cracking products, in both sweep gas and vacuum delubing. In many cases these containers are considered part of the work charge and are loaded and unloaded with the product inside. In some cases, a permanent box or retort that remains in the furnace is used.

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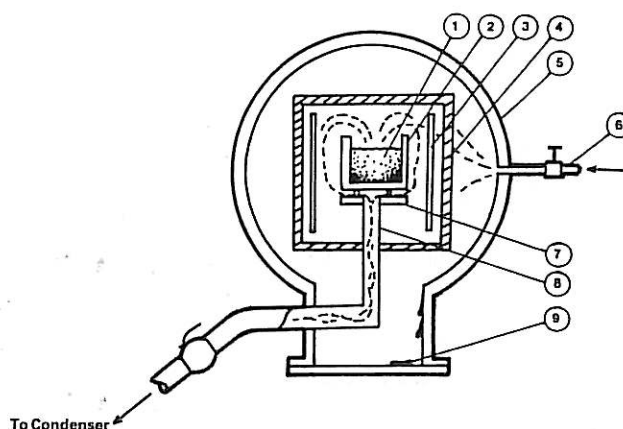
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Removal in Sweep Gas

A carrier gas or sweep gas at sub-atmospheric pressure has been used for some time by several manufacturers to remove the transport lubricant away from the product. If sweep gas is used during delubrication, additional variables resulting from a more complex process are introduced. On balance,



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- 2 Crucible - graphite
- 3 Heater - graphite
- 4 Insulation - graphite felt
- 5 Chamber - warmed by hot water
- 6 Sweep gas inlet
- 7 Hearth
- 8 Pump out - graphite
- 9 Lubricant accumulated in chamber

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 (single test only)

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FIG. 1 Test set up for evaluation of sweep gas and condenser