

Vacuum Binder Removal and Collection

Charles W. Finn*

Thermal removal of binders from powder metal (P/M) and metal injection molded (MIM) parts is aided by the application of vacuum, particularly when combined with a carrier gas. However, collection of the removed binders is difficult and can be costly, as uncollected binder can result in damage to vacuum pumping systems. A novel solution to this problem is presented based on thermal vacuum debinding.

Introduction

Metal Injection Molding (MIM) technology combines the advantages of net shape processing used for many years in the plastics industry with the technology of metal sintering developed by the powder metallurgy (P/M) industry. Unlike classic P/M parts, which are fabricated in a die using limited amounts (½ to 1%) of lubricant to minimize die wear, MIM uses large amounts of binder materials, sometimes in excess of 50 volume percent. Indeed, one can think of green MIM parts as metal reinforced plastics! This high volumetric fraction of plastic allows the techniques of injection molding to be applied to MIM parts. However, once the molding process has been completed, the binder has to be eliminated. Various techniques (thermal, chemical and mechanical) are in use. This paper discusses thermal vacuum debinding specifically. After the binder has been eliminated the MIM parts are very fragile, rather like a sand castle that had dried in the sun. They then have to be heated to the sintering temperature to produce a useful product. Often a post sintering heat treatment is applied to improve mechanical properties.

Binder Types

Many compounds have been used or proposed as binders for MIM. These have been divided in five categories by German¹, and are listed in Table I.

*Formerly Director of Research, Vacuum Industries, Inc. Now: Associate Professor, Materials Division, Department of Mechanical Engineering, Northeastern University, 360 Huntington Avenue, Boston, Massachusetts 02115, USA.

The most common binder type used for MIM is a thermoplastic, usually a combination of waxes and polymers, with a small amount of additives such as lubricants and surfactants. All the binder types, with the exception of the inorganics, can be removed by thermal treatment. All binders share one common trait; once the molding operation is complete they are no longer necessary or even desirable.

Vacuum Binder Removal

Early experience with the vacuum sintering of tungsten carbide cutting tools which contained 1-2w/o paraffin wax as a lubricant demonstrated that vacuum delubing, as it was called, was very efficient. This is based on the fact that paraffin waxes have high vapor pressures at temperatures low enough that thermal decomposition or "cracking" does not occur. In Figure 1, it is seen that the various paraffin hydrocarbons evaporate readily at temperatures below 200°C and at a pressure below 100 μm of Hg.* Conversely, Figure 2 shows that temperatures in excess of 400°C are required to reach the boiling point at atmospheric pressure of the higher molecular weight paraffins. At these temperatures there is a real danger of "cracking" or decomposition of the paraffins, adversely effecting the carbon content of the parts. The original Sintervac™ furnaces were developed for vacuum delubing and sintering of WC cutting tools and rapidly became the industry standard. However, direct vacuum delubing allowed the evaporated paraffins to condense on the water-cooled furnace walls and in the graphite

*100 μm of Hg = 0.1 torr = 0.133 mbar = 13.33 Pa.

TABLE I. Five Binder Types

Type	Examples
Thermosetting Plastics	Phenolics, Epoxies
Thermoplastics	Waxes, Polyethylene
Water Based Systems	Ice
Gellation Systems	Methylcellulose, Agar
Inorganic Systems	Sodium Silicate-CO ₂

felt insulation from where it could back diffuse into the parts during the sintering cycle.

This led to the development of the Sweepgas™ system, in which an inert gas, such as argon or nitrogen, was allowed to flow over the WC parts which were contained in a solid graphite retort. A pressure differential was maintained between the furnace chamber and the inside of the retort and the carrier gas directed the paraffin vapors directly out of the system.

The Sweepgas™ system was modified for debinding of MIM parts, as shown in Figure 3. A graphite box containing coated graphite shelves is used to hold the parts. The carrier gas is directed onto the top shelf and serpentine over the parts, carrying the evaporated binders to the pump port at the bottom of the retort. The rate of binder removal was found to be a function of pressure and shape factor of the parts.² Lowering

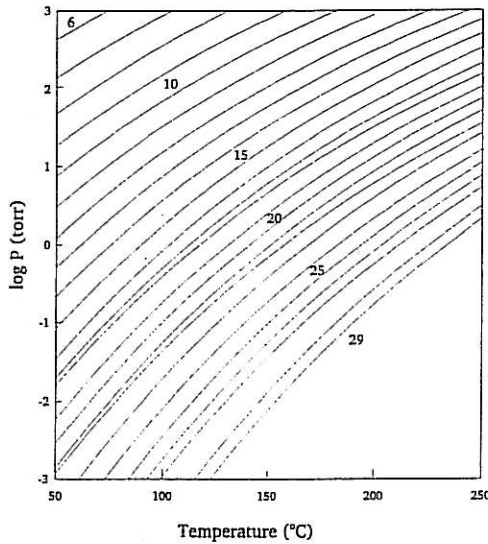


Figure 1. Vapor pressure of normal paraffins (C6 through C29).

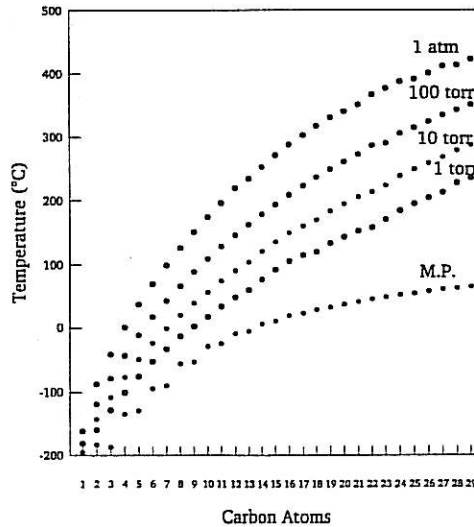


Figure 2. Vapor pressure and melting point of the C1 to C29 normal paraffins.

the pressure from 10 torr to 0.1 torr caused a dramatic improvement in the rate of paraffin removal as shown in Figure 4. A further reduction by a factor of 3 in binder removal time was achieved by further reducing the pressure to the submicron level as shown in Figure

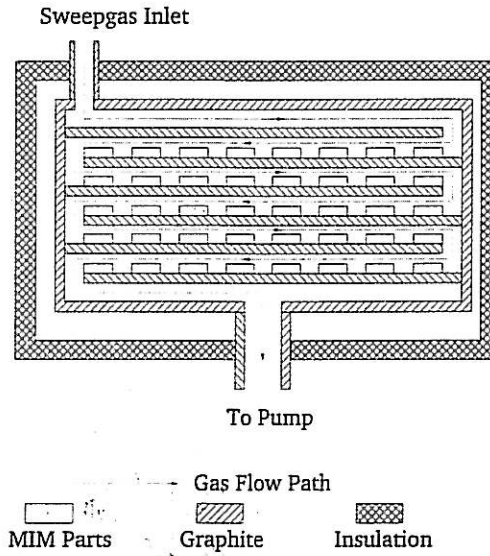


Figure 3. Injectavac retort for MIM parts.

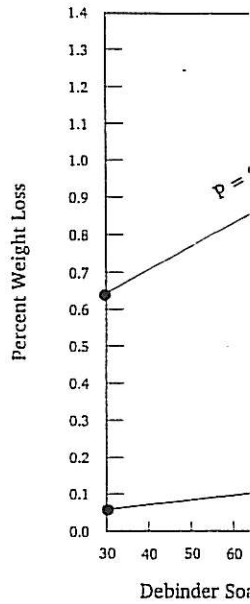


Figure 4. Effect of pressure on binder removal.

5. This required the use of a high vacuum system. The advantage of a high vacuum is also clear from Figure 5. The binder was removed, higher temperatures were used to remove the polyethylene from the parts, and higher molecular weight binders were removed by direct evaporation. Polyethylene depolymerizes to lower molecular weight binders. These binders are then removed by the oil in the diffusion pump. Sweepgas is thus introduced at a rate of several liters per minute during the binder removal and sintering cycle.

Binder C

All the binder breakdown products must be removed from the pump port and must be collected by the mechanical pumping system. In the early Sintervac™ container system, called a 'sweepgas' system, a line and served to collect the binder vapors, which were cooled and condensed. Only relatively small amounts of binder were removed. However, some problems

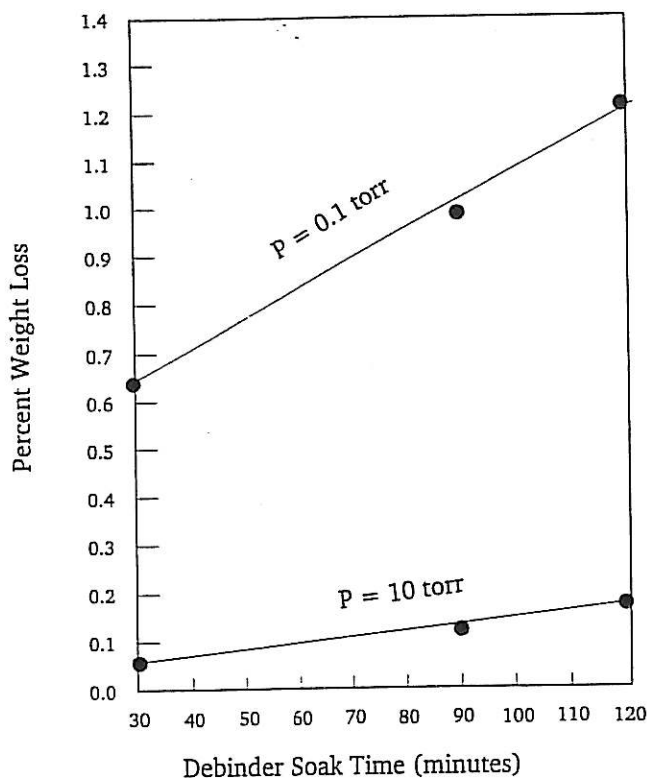


Figure 4. Effect of pressure on binder removal time.

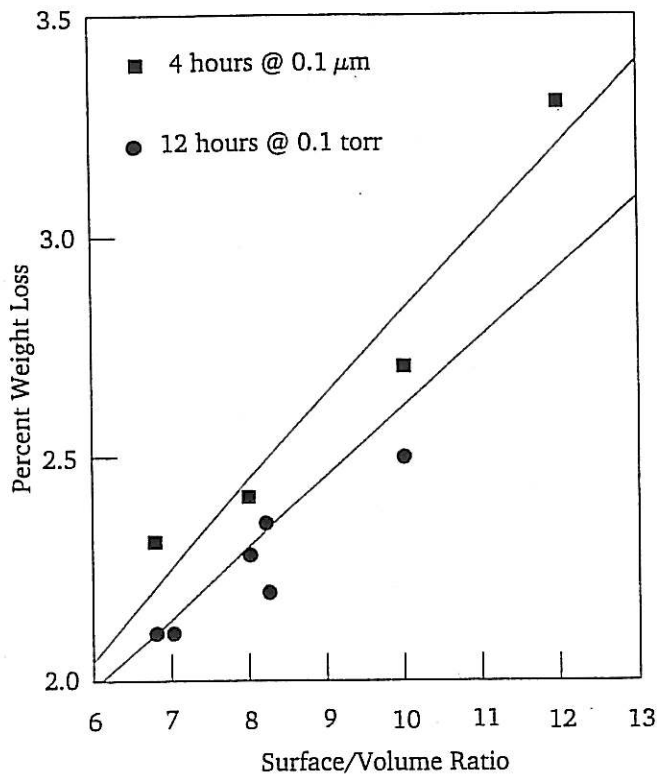


Figure 5. Effect of shape factor and pressure on binder removal.

5. This required the use of a diffusion pumping system. The advantage of a high surface to volume ratio is also clear from Figure 5. Once the paraffin fraction was removed, higher temperatures were required to remove the polyethylene fraction. Unlike paraffin, the higher molecular weight of polyethylene precludes direct evaporation. Polyethylene decomposes or depolymerizes to lower molecule weight species during binder removal. These breakdown products react with the oil in the diffusion pump, precluding its use. Sweepgas is thus introduced at 1 to 100 torr pressure at a rate of several liters per minute. A typical binder removal and sintering cycle is shown in Figure 6.

Binder Collection

All the binder breakdown products leave through the pump port and must be trapped before reaching the mechanical pumping system.

In the early Sintervac™ furnace design, a simple container system, called a "wax pot," was placed in-line and served to collect the paraffin vapors as they cooled and condensed. This worked well for the relatively small amounts of lubricant in the WC parts. However, some problems were encountered with

paraffin wax reaching the mechanical pumps used by the WC industry, leading to pump breakdown.

In 1985, Lutts and Anderson patented the Sweepgas condenser, shown in Figure 7, to capture the paraffin lubricants. Hot paraffin-laden carrier gas was directed to a water jacketed condenser where they were collected. Figure 8 shows that the collection efficiency is in excess of 98%, with > 95% of the paraffin collected in the wax pot.

When development work began on the Injectovac™ furnace for the debinding and sintering of MIM parts, the earlier binder collection techniques were used. This required re-design of the condenser to handle the larger amounts of paraffin as well as the products of polymer breakdown. Direct observation through the sight port at the top of the condenser revealed the presence of "smoke-like" particles during the polymer removal phase. Capturing these ultrafine particles presented a real engineering challenge. Small quantities of these products soon destroyed the pumping system.

As noted earlier, there are numerous binder systems used for MIM. Each combination required a different condenser, or series of condensers. One commercial system, available in 1989, had 3-5 condensers in series. This was clearly a clever chemical engineering solution

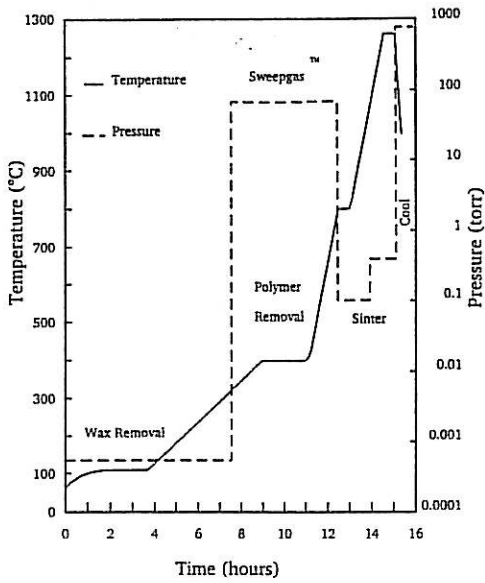


Figure 6. Typical binder removal and sinter cycle.

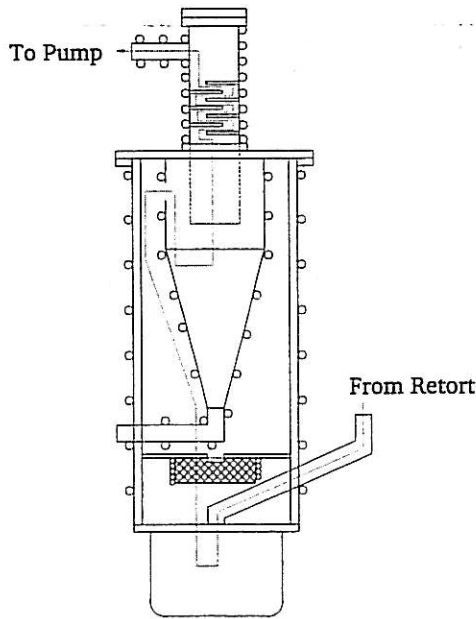


Figure 7. Sweepgas condenser (after Lutts and Anderson).

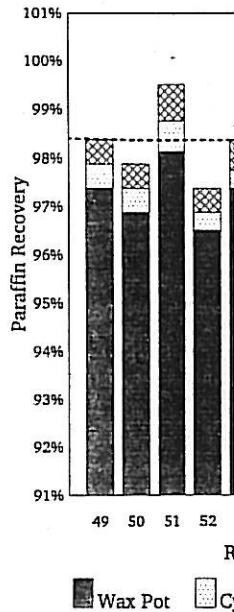


Figure 8. Sweepgas condenser in 25 minutes, 800 grams of Pa

but at what cost? Each condenser costs money to design, build and maintain. However, a larger economic problem occurs. Flow in a vacuum is controlled by molecular diffusion. Conductance (i.e. the inverse of resistance to flow) can be calculated for simple geometries. For example, for a straight long pipe the conductance, in liters/second, is given by Van Atta³ as:

$$C = \frac{8}{3} \left(\frac{2kT}{m\pi} \right)^{1/2} \frac{A^2}{sL} \quad (1)$$

where:

- k = Boltzmann's constant
- T = Absolute temperature (K)
- m = number of molecules per unit volume
- A = cross section of the tube (cm²)
- s = perimeter of the tube (cm)
- L = length of the tube (cm)

For a round tube pumping air at 20°C, this equation reduces to:

$$C_{air} = 12.12 \frac{D^3}{L} \quad (2)$$

where:

- C = conductance (l/s)
- D = tube diameter (cm)
- L = tube length (cm)

For example, for a 6 foot long straight tube, 4 inches in diameter, the conductance (C) is 70 liters per second. This can be converted to a pumping speed using the relationship:

$$S = \frac{S_p C}{S_p + C} \quad (3)$$

where:

- S = pumping speed (l/s)
- S_p = speed of the pump at its inlet (l/s)
- C = conductance of the pipe (l/s).

Using a Stokes 149MB pump at 50 μm, its listed inlet speed (S_p) is 52 liters/second. Solving equation (3),

the pump rate (S) is 30 l/s. As with electrical conductances in series added for convenience of the plus two 2 foot lengths of 4 in. Each two foot length of 209 liters/second while each of 350³:

$$\frac{1}{C} = \frac{1}{70} + \frac{1}{21}$$

Thus, the conductance has increased to 21 liters/second. What if the system has an inlet speed of 21 l/s to 2149MB HC pump having an inlet speed of 21 l/s gives 49 l/s and 28 l/s, 'obstacles' that are in the vacuum system, and buying a larger pump?

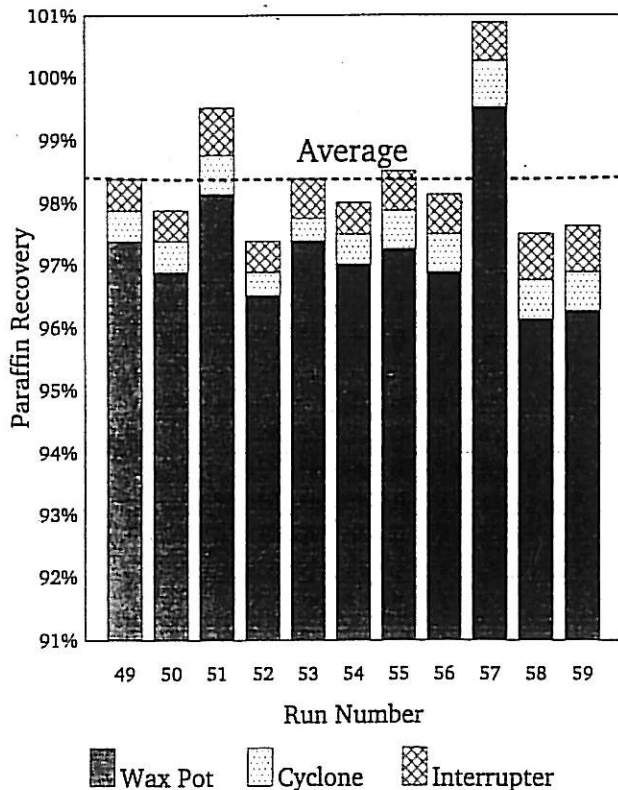


Figure 8. Sweepgas condenser efficiency. Ambient to 600°C in 25 minutes, 800 grams of Paraffin, 19 LPM Nitrogen, 10 torr.

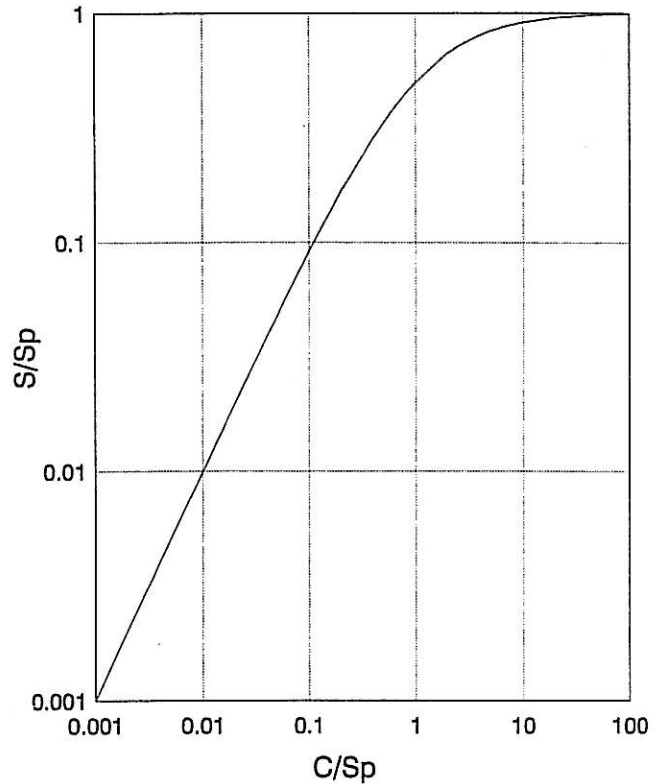


Figure 9. Effect of conductance on pumping speed.

the pump rate (S) is 30 liters/second at the inlet of the tube. As with electrical conductance, pumping conductances in series add as the inverse. Suppose that for convenience of the plumbing, it is necessary to add two 2 foot lengths of 4 inch pipe and two 90° elbows. Each two foot length of pipe has a conductance of 209 liters/second while each elbow has a conductance of 350³:

$$\frac{1}{C} = \frac{1}{70} + \frac{2}{209} + \frac{2}{350} = \frac{1}{34} \quad (4)$$

Thus, the conductance has dropped to 34 liters/second and the pumping speed at the pipe inlet has dropped to 21 liters/second. What happens if one uses a Stokes 129MBX which has an inlet speed (S_p) of 94 liters/second? With the 6 foot system the inlet speed (S) increases from 30 l/s to 40 l/s and with the 10 foot system from 21 l/s to 25 l/s. With a larger Stokes 149MB HC pump having an inlet speed (S_p) of 165 l/s gives 49 l/s and 28 l/s, respectively. Thus, the more 'obstacles' that are in the path between the pump inlet and the vacuum system, the lower the pumping speed. And buying a larger pump does not help significantly.

One can only guess at the effect of a condenser (or several condensers in series) on the overall pumping speed.

An interesting way of illustrating the effect of conductance on pumping speed is through the use of dimensionless numbers. Consider equation (3) which can be re-arranged by dividing through by S_p to give:

$$\frac{S}{S_p} = \frac{c/S_p}{1 + c/S_p} \quad (5)$$

Figure 9 shows the dimensionless pumping speed, S/S_p , versus the dimensionless conductance, C/S_p , on a log-log scale. At low conductances, the effect is essentially linear.

A Solution to the Problem

The key to the solution to the problem is lateral thinking. First, state the problem: **Binder breakdown products damage the pumping system.** Then state the current solution: **Trap the binders in a series of condensers.** Then state what is wrong with the solution:

Expensive to design, build, maintain and operate, with a different design needed for each use. Finally, think of an alternate, asking "What if?" types of questions. What if better pump oil can tolerate binder contamination? Or what if changing the oil were cheap enough that contamination does not matter? What if a better pump can tolerate contaminated oil or what if the oil does not stay in the pump long enough to cause the problem.

A research effort to design a better pump was undertaken but it was found that such a pump already existed! Originally designed for alternate applications, a "Once through oiling" (OTO) pump is commercially available. It is a rotary vane pump with an oil injector. Each rotation of the pump ejects the oil to a sump where it can be purified for re-use or discarded, depending on prevailing economics. The oil, common S.A.E. 40 non-detergent motor oil, does not remain in the pump long enough for the contamination to be a problem. Numerous tests over the last 18 months at Vacuum Industries have shown that the OTO pump can handle any binder system currently used in MIM.

Conclusions

Vacuum thermal processing can significantly improve binder removal rate, resulting in reduced cycle time. Application of the OTO pump eliminates the need for a condenser, improving pumping capacity. A new system (Vacuum Industries Binder Removal System (BRS)) provides a simple but effective solution for the debinding, sintering and heat treatment of MIM parts in a single unit, reducing overall process cost.

References

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