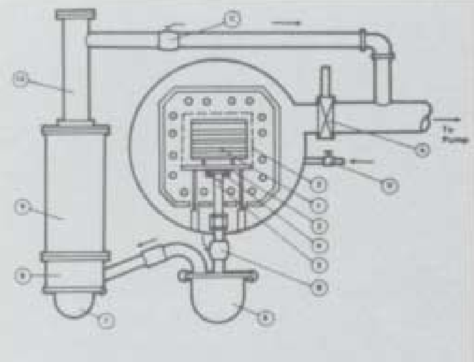
C/VI seeking to expand MIM materials base

In the late 1960's Centorr/Vacuum Industries of Nashua, New Hampshire, USA (then known as GCA Vacuum Industries) developed the first vacuum debind and sinter furnace for the production of tungsten carbide. That development has led to a close association with the powder metallurgy (PM) industry that now includes 10 years experience with metal injection moulding (MIM) processing. C/VI runs an extensive R&D programme dedicated to extending the materials available to the MIM industry. David Yoel of C/VI outlines the programme's activities.



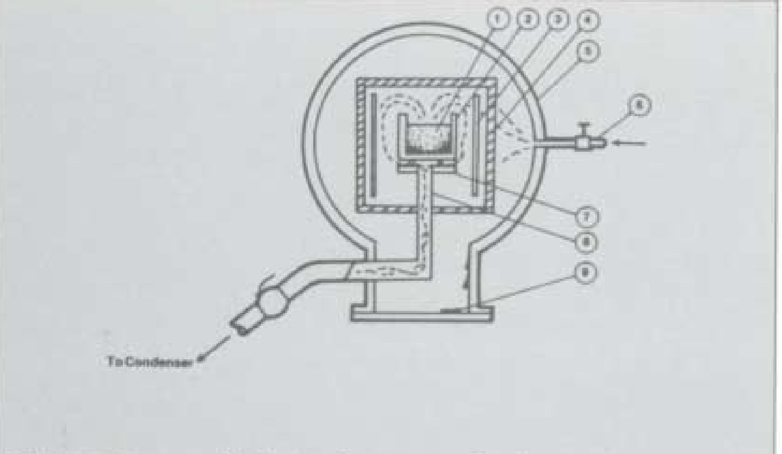
1 Work
2 Work box (alternative)
3 Hearth plate/work box bottom with pump out
4 Kragen - joins pump out tube to hearth plate opening
5 Pump out tube - distribution system
6 First stage collector - operates warm
7 Second stage collector - operates warm
8 Filter stage - operates warm
9 Expansion and centrifugal 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 Sweepgas valve. Open during delube with sweep gas

FIGURE 2: Sweepgas system for delubrication.

In the early days of vacuum debinding and sintering of tungsten carbide, parts containing 1-2% (by weight) paraffin wax binder were loaded into open graphite crucibles in a vacuum furnace. As heat was applied, the wax liquified, and moved to the surface of the part, where, because of its high vapour pressure, it vapourized (see Figure 1). The wax vapour then diffused through the graphite insulation pack, and deposited on the warm chamber walls where it ran down into a wax condensing system. Although the approach was effective, there were several problems. If the wax was heated above 400°C, it cracked, producing free carbon, adversely effecting the carbon levels of work. Also, heating rates were limited, as was hot zone life.

FIGURE 1: Setup for vacuum debinding.



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

Load: paraffin 800 grams
Gas: nitrogen 120 1/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

The company then developed the Sweepgas system shown in Figure 2. A carrier gas at sub-atmospheric pressure is used to sweep the lubricant away from the product. While maintaining a pressure differential between the retort and the furnace, the gas/paraffin mixture is pumped directly through openings in the retort into a new condensing system specifically designed for sweep gas. Unlike vacuum delubing where the wax vapour covered the entire hot zone and chamber, the Sweepgas system forces the vast majority of the wax to be swept into the condensing system, leaving little wax behind in the hot zone. The result is reduced carbon generation, improved hot zone life, and improved product consistency. This approach formed the basis for the industry standard Sintervac furnace. Over the years, the Sintervac has been refined and enhanced, new technology has been developed, and the process has been applied to additional powder metallurgy (PM) materials including mild steels, tool steels, high speed steels, and stainless steels.

As MIM began to emerge in the early 1980s C/VI immediately recognized the significant potential of this new technology and began to apply its experience in PM to develop a combined debind and sinter cycle for the technique. Although quite analogous to the tungsten carbide process, the standard Sintervac system was not capable of full MIM debind and sinter cycle for several reasons. These factors include MIM's significantly higher binder content, and the use of a wide variety of different binders. The modified Sweepgas cycle developed formed the basis for the Injectavac Furnace.

In the modified MIM Sweepgas system, the carrier gas is brought directly into the graphite retort (Figure 3). The graphite shelves are staggered so that the gas flows over the shelves sequentially. The binders are carried away to the condensing system through a port at the bottom of the retort. The binder removal rate is dependent on the gas pressure and the surface to volume ratio of the parts being processed. The lower the pressure, the higher the rate of binder removal. Therefore, during certain phases of the debind cycle, a high vacuum diffusion pumping system was found to substantially increase the binder removal rate. The paraffin wax binders are first sublimated from the parts, followed by the polymer binder which decompose to lower molecular weight species upon evaporation. The pumping system uses a 'once-through-oil' design. On each cycle of the pump, the sealing surface is wetted with a small amount of fresh oil. The rotary vane then traps the pumping gas stream. As the vane completes its rotation, the contaminated oil and gas effluents are pushed into a reservoir. Since the binder effluents are not able to collect on the sealing surfaces, the pump is not damaged by the binders. The oil is an industrial grade (10 W-30). The pump uses approximately the same amount of oil as a standard pump. The Injectavac system has been used to pump a wide variety of binder systems. The MIM Sweepgas system forms the basis for the Injectavac furnace.

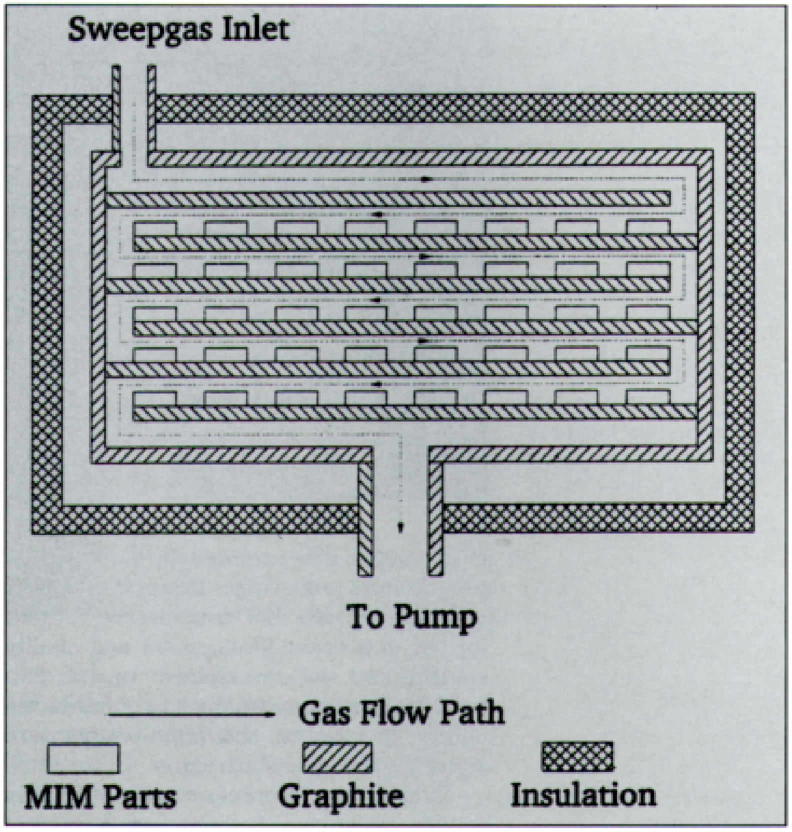
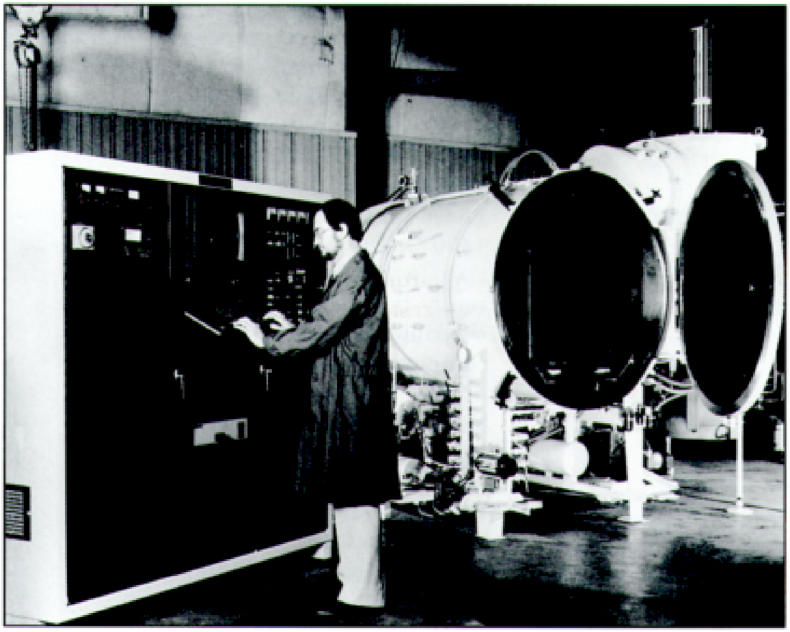


FIGURE 3: Injectavac retort for MIM parts.

The Injectavac process cycle has several advantages. The number of separate process steps required to produce MIM parts is reduced. For typical MIM parts, complete debind and sinter cycles require 12-20 hours, depending on part thickness, and other factors. The process is very repeatable, resulting in a high degree of run-to-run consistency. The system is quite flexible in that a variety of parts, made of different alloys, can be processed in the same system, with the changeover from one process to another occurring in a matter of minutes. Parts handling is minimized, saving labour and reducing damage caused by handling of partially debound parts. Floorspace requirements are minimized, since separate debinding and sintering equipment is not required. Utility requirements are minimized, since power is only used when parts are in process, and large amounts of gas are not required for the process. Environmental concerns are elimi-

FIGURE 4: This Injectavac furnace is capable of processing 500 kg of green parts in a single 12-15 hour cycle.



nated, since the spent oil is recycled. Figure 4 shows an Injectavac furnace capable of processing 500 kg of green parts. This is equivalent to 12 600 parts with a cross-section of 25 mm × 25 mm × 12.5 mm. Injectavacs are currently in use manufacturing MIM parts in over a dozen plants around the world.

Technology development

Early Injectavac furnaces were used for processing nickel-iron parts. As the industry evolved, the requirement to process additional materials became evident, and a program was initiated to develop process cycles for a series of stainless steels. A key factor in C/VI's ability to develop these systems is its applied technology centre (ATC). Along with Injectavac and sintering furnace capabilities, the ATC also includes a carbon analyzer, metallographic preparation capability, optical microscope, microhardness tester, residual gas analyzer, and oxygen monitor. The experienced in-house personnel are complemented with collaborative efforts with universities, the use of outside consultants, and outside laboratory service providers (e.g., SEM, EDS, Auger, etc.).

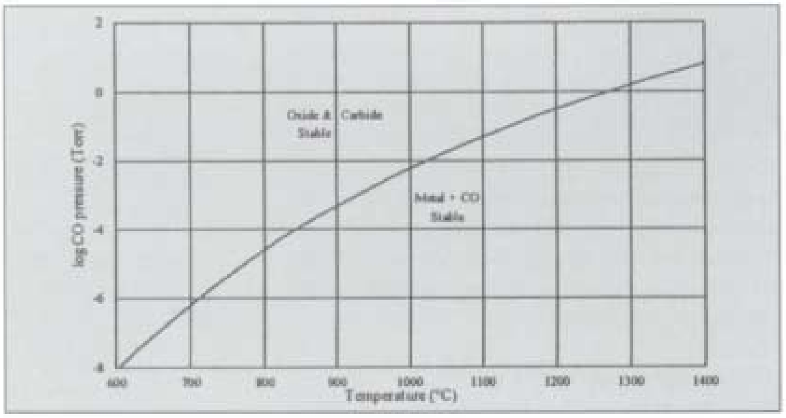


FIGURE 5: The chromium vs chromium oxide/chromium carbide equilibrium CO pressure curve.

The first stainless steel process to be developed was for 316L. Because the Injectavac uses a graphite hot zone, concerns arose over the ability to control carbon levels. A second concern was the ease with which chromium forms oxides, carbides, and nitrides. This later concern led to the use of argon gas to eliminate nitride formation. In the presinter and sinter stages, 5% hydrogen is added to the argon Sweepgas, to reduce oxides. As shown in Figure 5, if the partial pressure is sufficiently low, the metal is stable, and oxides and carbides do not form. Indeed, under certain circumstances, carbon levels in the parts can actually be reduced. A typical process cycle is shown in Figure 6. It has now been conclusively demonstrated that high quality 316L stainless steel can be completely debound and sintered in a graphite hot zone using the Injectavac process cycle.

After the completion of the 316L programme, C/VI launched an effort to develop a process cycle for 17-4 PH stainless steel. Early experiments showed a loss of copper in the processed parts, resulting in inadequate hardness. 17-4 PH stainless

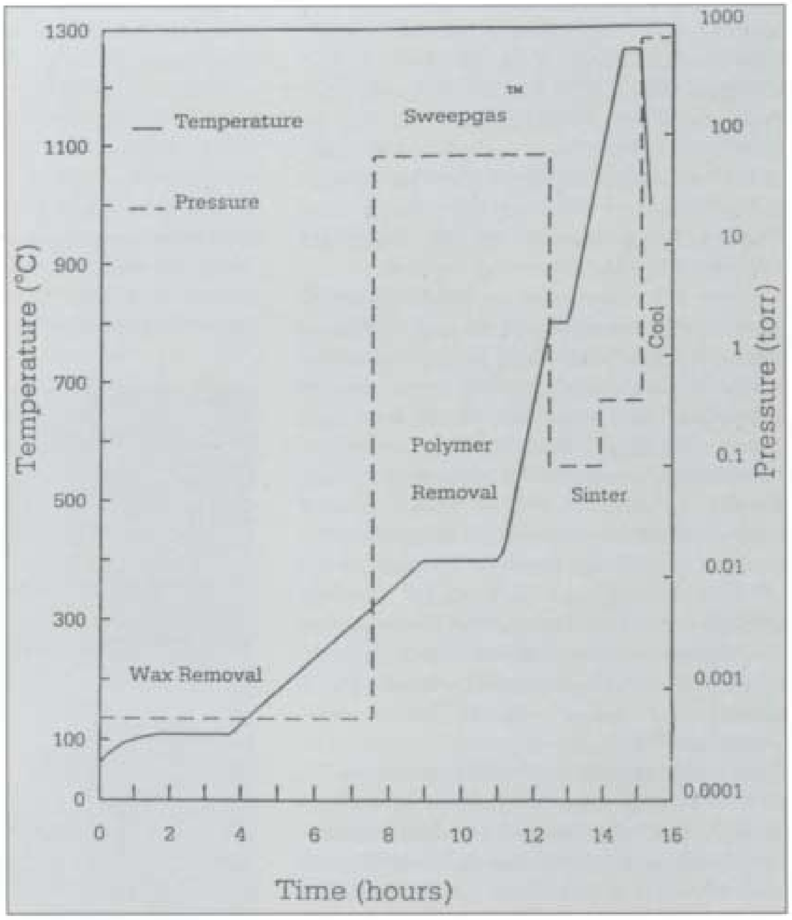


FIGURE 6: A typical binder removal and sinter cycle for an Injectavac furnace.

steel has a copper content of approximately 4%. Copper has a much higher partial pressure at lower temperatures than the constituents of 316L. The challenge, then, was to develop a process cycle in which the vacuum levels were sufficiently high to completely debind the parts, and yet low enough to suppress copper vapourization. A process cycle has recently been developed that accomplishes these goals. 17-4 PH parts have now been processed to 99% densities (per MPIF Specification 35), with excellent uniformity of the alloy constituents, round, uniformly distributed pores, and a hardness of over 40 on the Rockwell C scale (after aging). Cycle duration is comparable to the other materials discussed above.

Current efforts are focused on optimizing the process cycle for 304L stainless steel MIM. This effort has been underway for several months and is nearing completion. Future plans call for developing cycles for tungsten carbide and titanium.

In recent experiments on 17-4 PH stainless steel, photomicrographs of etched and polished sections revealed trace amounts of silica precipitate near the surface of the parts (see Figure 7). The silica precipitates were thought to result in slightly higher overall porosity, and a less-than-uniform porosity distribution near the surface. A detailed analysis found that trace amounts of silica were present in the alumina fibre paper used to isolate the parts from the graphite trays. As can be seen in Figure 8, this source of contaminants has been eliminated, and the Injectavac process has been further refined. This finding is of benefit to all materials processed by the Injectavac process.

One key advantage to the Injectavac process is the relatively short period of time required for the debinding phase. Indeed, in small furnaces, high quality parts can be produced with debinding times half that required in larger systems. For example, a furnace processing 50 kg of parts may only require 5 hours to debind MIM parts of typical cross-section, yet a large furnace processing 500 kg, requires approximately 10 hours to debind. The binder removal rate is, therefore, (in part) dependent on the design of the furnace itself. C/VI is actively developing two new technologies to increase the binder removal rate in large Injectavac furnaces.

A combination of careful experiments and analytical thermal models are being used to further improve the temperature uniformity of the Injectavac systems. This is one of the key areas of focus of C/VI's programme. It is important to understand that uniformity is necessary at both low and high temperatures. Typical temperature span across the hot zone is currently in the order of ± 10°C depending on furnace size, across the entire temperature range. Uniformities of ± 5°C are achievable at high temperatures.

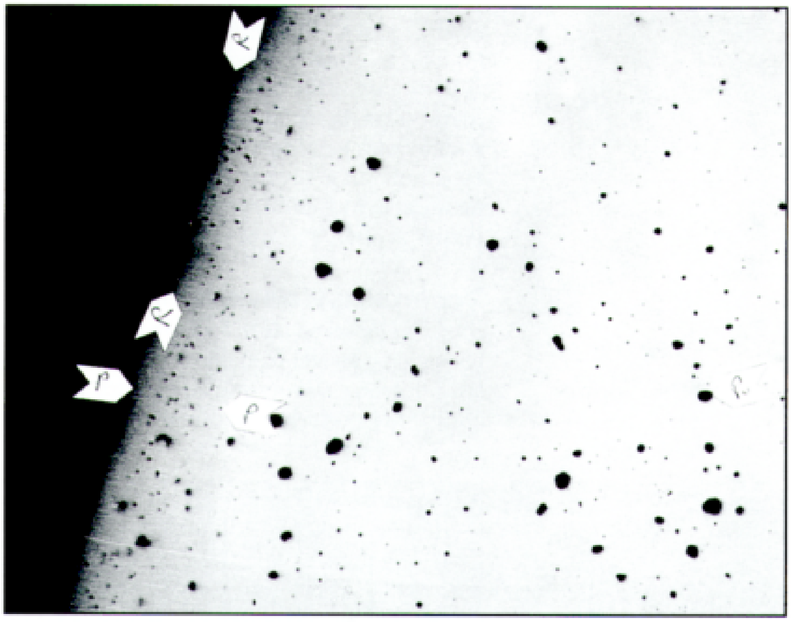


FIGURE 7: Unetched 17-4PH stainless steel (early experiments 180X). Arrows P-P surround a region 1 mm deep that contains a fine silica precipitate. (Micrograph by Powder-Tech Associates, Inc.).

Reducing system cost

Manufacturers constantly work to reduce the cost of their products, C/VI is no exception. Because the Injectavac is a combination debind and sinter system, the initial cost appears high compared to either a separate debind oven or a sintering furnace. In any case, an aggressive effort to 'valve engineer' the entire system has resulted in a reduction in system cost of over 10% in the last year. This effort is still underway, and is continuing to yield results.

Along with the two new technologies discussed above to reduce the debinding times of large Injectavacs by a factor of two,

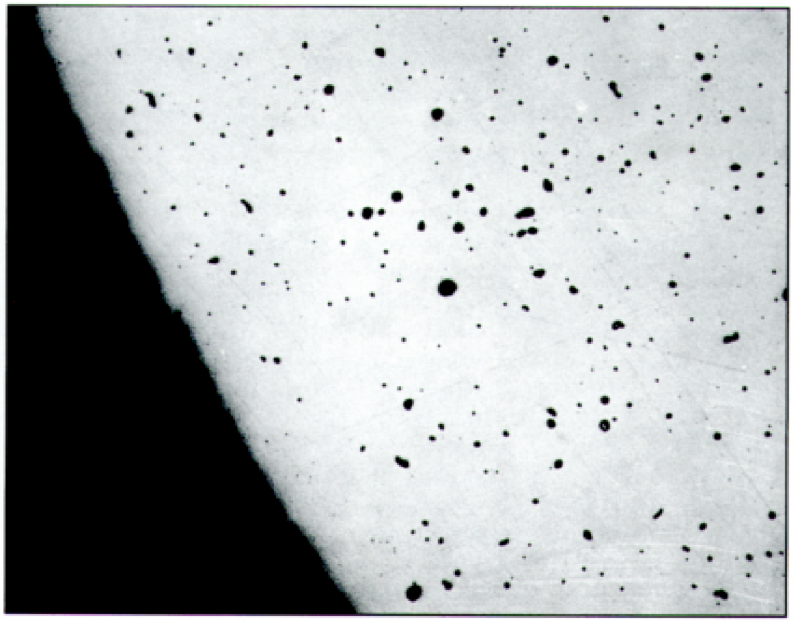


FIGURE 8: Unetched, repolished 17-4PH stainless steel (180X). Silica precipitate eliminated. Shows well rounded porosity and good sinter. (Micrograph by Powder-Tech Associates, Inc.).

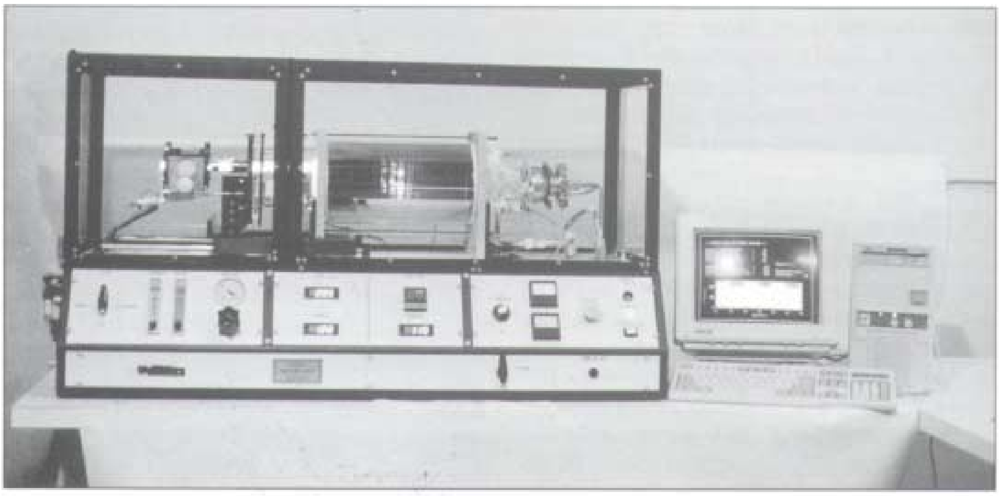


FIGURE 9: C/VI's transparent furnaces allow visual monitoring of the process cycle.

C/VI has two other technology development programmes underway. These involve the development of a new 'transparent' furnace for use in process development and quality control, and the development of new "in-situ" monitoring techniques to enhance the ability to monitor the high temperature sintering process as it occurs.

'Transparent' furnaces consist of spirally wound resistance heaters inside several concentric clear glass tubes (Figure 9). The outer tube has a thin film coating which is highly reflective to heat, and mostly transparent to visible light. This makes it an effective insulator and yet provides a clear view of the processes occurring within. Visual monitoring, coupled with the ability to adjust the process cycle, provides a powerful new tool for quickly and efficiently optimizing process cycles. These systems have also been used in quality control applications to verify the melting ranges of materials, and to troubleshoot production systems by providing the means to duplicate and observe problems. They are also useful in education and training, allowing the study of the effects of varying environments on processes. Currently, transparent furnaces are limited in operation to approximately 1000°C.

The C/VI programme is aimed at creating a 1400°C transparent furnace system. The program has two phases. The first phase targets a 1200°C rating, the second, 1400°C. The first phase is nearing completion, with demonstration hardware currently in fabrication. The second phase is scheduled to begin later this year and to be complete within one year.

The concept of making quantitative visual measurements of the debinding and sintering phases of the MIM process is a new one. On the other hand, it is common practice to make physical measurements of part dimensions before and after debinding and sintering as a means of characterizing part quality. A digital image acquisition and analysis system is under development that acquires images during debinding and sintering, enhances the images, compares the dimensions of the part as the cycle progresses to measure the shrinkage (and rate of shrinkage), and to correlate the changes in part dimension to process parameters including temperature, pressure, gas dimension etc. Flaws can be observed during their formation including flash, inconsistent surfaces, incomplete filling, blistering, cracking, and sagging. Initial experiments demonstrating this technique have been completed. The next stage will explore the utility of microscopic observations during debinding and sintering. The long range goals of this program are to develop a laboratory system capable of accelerating process development, and to evaluate the feasibility of developing in-process quality control techniques for production systems.

Sweepgas and Injectavac are registered trade marks of Centorr/Vacuum Industries.

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