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Developments in Combination Debinder/ Sinter/Pressure Consolidation Furnaces

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TECHNICAL NOTE

Based on paper presented at:
ISO3
3rd INTERNATIONAL CONFERENCE ON ISOSTATIC PRESSING
London, November 10 - 12, 1986
Reprinted from METAL POWDER REPORT, May 1987.

Developments in combination furnaces used for binder removal, sintering and pressure consolidation have followed the requirements of clearly identifiable markets. At present, varying requirements are leading to detailed differences in the furnaces supplied to users in these developing markets. Two of the markets, hardmetals and ceramics, and their requirements are discussed in the following article.

When combination delube, sinter and pressure densification furnaces were first being developed, uniaxial vacuum hot press sintering furnaces were being used to densify samples of powder materials to near theoretical density. This process results in an excellent product, but is slow and mostly suitable for laboratory development work. It was not, and still is not, practical for low cost manufacture of a large quantity of parts from powder materials. At the same time, large capacity production furnaces were also being used for binder removal and sintering a wide range of ceramics, hardmetals and steels at low absolute pressure. It was logical, therefore, to think in terms of combining binder removal and sintering with pressing to increase part density. Isostatic pressing in the same chamber offered a convenient way to accomplish some of the densification that had been achieved by hot pressing. Moreover, it made possible densification of shaped articles because of the uniform pressure on all sides.

Work on the combination furnaces has therefore proceeded, and a number of furnaces are now in full scale operation filling market niches not otherwise satisfied.

HARDMETAL MARKET

A clear need for a combined furnace was first presented by a part of the hardmetal industry. Hardmetal or tungsten carbide nibs installed in the tri-cone bits used in oil drilling need to be both very hard and very strong to satisfy the requirements of the very deep holes being drilled for oil in these times. If one nib breaks off, the extra load is transferred to its neighbours, and this also results in failure, and finally, the premature failure of the entire drill.

Providing uniformly high density and strength was initially accomplished by the use of hot isostatic presses which densified the nibs as a separate operation following the normal vacuum delube and sinter process. Later, it was found that a lower isostatic pressure would suffice, provided it was applied when the matrix was at or near its liquidus. The use of a densification pressure of only 27 bar resulted in TRS values nearly equal to the values for samples processed at usual HIP pressures (Table 1).

This knowledge led directly to the construction and use of a number of combination furnaces in sizes typical of normal tungsten carbide part production (Figs 1 and 2). Fig. 2 photograph shows a recent production furnace with 127 liters working volume. Closures are provided at both ends for convenience in loading (Fig. 3).

For tungsten carbide cutting tools, the use of a pressure densification step following normal vacuum sintering is not generally regarded to be absolutely necessary. Careful attention to the preparation of the powder and the press feedstock, coupled with careful control of the delube and sintering steps, has resulted in the production of uniformly high-quality cutting tools by many manufacturers. Some manufacturers of the highest grade cutting tools, wear parts, and forming punches and dies have found, however, that the pressure densification step following sintering virtually guarantees the absence of pits and flaws and hence provides a uniform higher quality than they have been able to achieve with normal vacuum sintering.

Freedom from flaws is, of course, of prime importance in such products as punches and dies, slitter knives, and similar specialty products which rely upon very fine and extremely sharp cutting edges, as well as super smooth finishes. This finding appears to apply to almost all grades of carbide except those with the very least quantity of cobalt binder. Coupled with the close carbon control and furnace cleanliness resulting from the use of a Sweepgas™ delube system, at least one manufacturer has achieved sufficient improvement in efficiency to more than justify the higher cost of combination furnaces for future furnace procurements.

Computer control is the latest innovation for combination furnaces (Fig. 4). The computer allows unattended operation because of its decision-making capability. While it provides maximum flexibility in programming the pressure and temperature to secure optimum cycles, it also supplies data in a convenient format for rapid analysis. Thus the combination delube, sinter and pressure consolidation furnace has moved from the laboratory to the production floor in tungsten carbide or hardmetal production. Since the results have been established, the questions presented to the equipment maker now, are those relating to production concerns such as cost effectiveness, reliability, justification and the like.

	Vacuum Sintered	Vacuum Sintered and HIP'D	Pressure Sintered
6% Cobalt			
Density	14.87	14.90	14.89
Hardness	91.6A	91.9A	92.0A
TRS	316,000 psi	384,000 psi	360,000 psi
Porosity	A_1	A_0	A_0
9% Cobalt			
Density	14.59	14.58	14.63
Hardness	91.2A	91.2A	91.2A
TRS	315,100 psi	345,000 psi	412,400 psi
Porosity	A_1	A_0	A_0
12% Cobalt			
Density	14.09	14.07	14.11
Hardness	89.9A	90.0A	90.8A
TRS	310,000 psi	365,000 psi	372,000 psi
Porosity	A_0	A_0	A_0

TABLE 1 Comparison of transverse rupture strength - sintered, ISO pressed and pressure densified tungsten carbie

The world of new engineering ceramics is entirely different, however. It is not simply a question of substituting a higher quality part for the original and justifying the increased cost by better performance. The very nature of ceramic materials usually dictates significant design changes in the equipment in which they are introduced and part designs differ from designs used by the more forgiving metal parts that are being replaced. The question of justification for the use of ceramics therefore becomes much more far reaching and much more complex.

For these reasons acceptance of ceramics has been slow and has been accomplished by both extensive and expensive development effort, usually on a case-by-case basis. To date, very few structural ceramic parts have reached the production stage except those that are made by pressureless sintering or reaction bonding. These include such new entries as chip carriers in the electronics industry, seals and heat exchangers made from sintered silicon carbide and reaction bonded silicon carbide, Sialon cutting tools, and evaporation boats made from titanium diboride to mention a few. Pressure densification has not been required, and the sintering furnaces have been designed with an extension of existing, proven, reliable technology.

The combination of physical characteristics embodied in sintered silicon nitride recommends it for a wide range of structural, wear resistant and heat tolerant parts in which other ceramic materials have not been found to be suitable. Consequently, extensive effort is underway to develop the formulations required to produce silicon nitride parts, as well as the production techniques which will ultimately lead to large scale production at low cost.

While some parts made from silicon nitride - turbine and turbo-charger rotors for example - are heavily stressed and require the maximum strength, uniformity and density imparted by hot isostatic pressing, many more applications can tolerate parts which are not called upon to operate under such extreme conditions. They may, therefore, be formulated with sintering aids such as yttria, silica, calcia and the like. These glassy materials not only promote densification at lower sintering temperatures, they also allow some densification at pressures lower than are normal in hot isostatic presses.

Fig. 5 shows a typical combination binder removal, sinter and pressure densification furnace that is being used in the development of structural and wear parts which are based upon silicon nitride. The work pieces may be prepared by cold isostatic pressing, injection moulding and even slip casting to suit the part geometry. In the furnace, they are heated under close temperature control while the binder materials are evaporated at sub-atmospheric pressure and are directed toward the condenser which protects the pump. Heating may be done in vacuum to outgas the work at temperatures lower than sinter temperature, and final sintering is done in nitrogen gas at pressure. In this case, a pressure of 27 bar was deemed adequate for

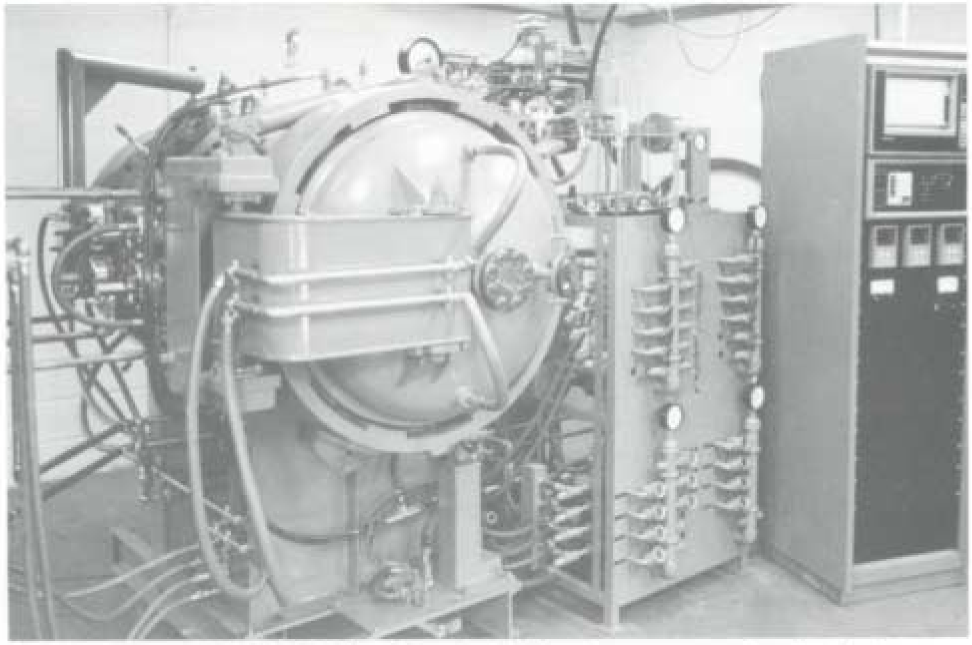

FIG 1 Early design SINTERBAR™ combination delube, sinter, pressure densification furnace

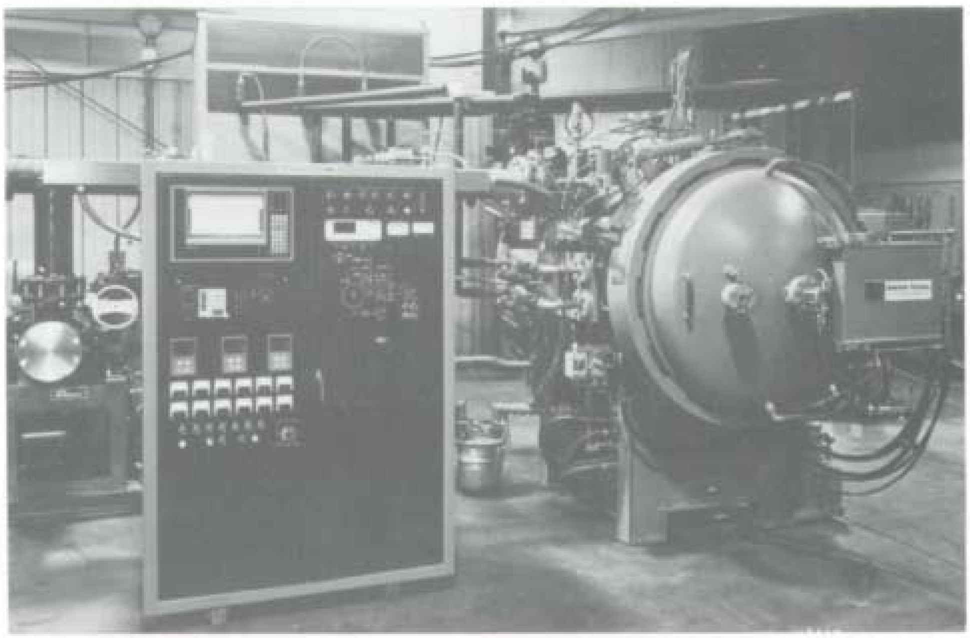

FIG 2 Typical tungsten carbide combination delube, sinter, pressure densification furnace

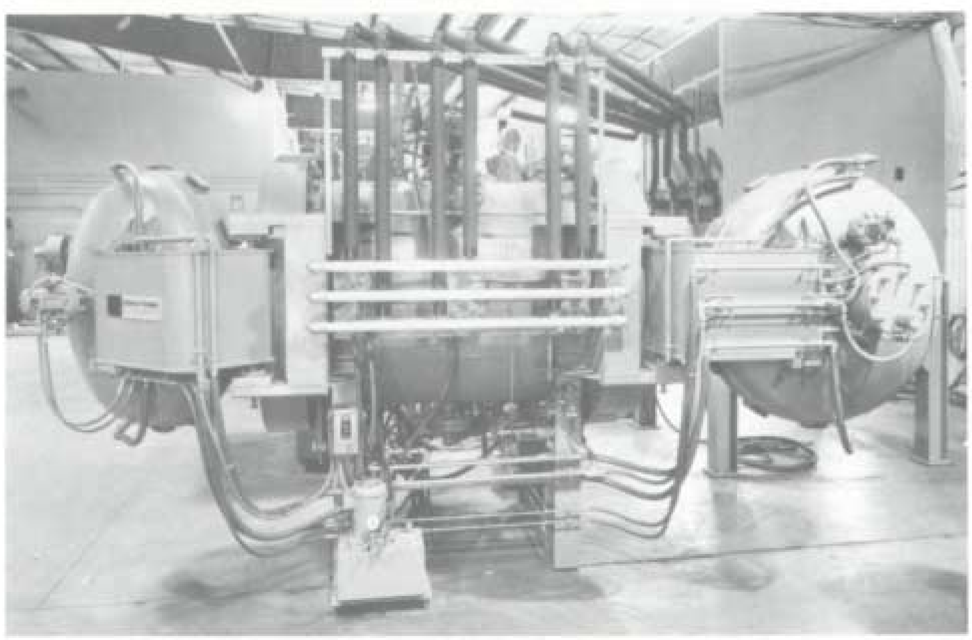

FIG 3 Typical combination furnace - side view showing both loading doors

the work to be performed. The heating elements in this furnace are graphite which can be tolerated because the parts are embedded in silicon nitride setter powder which has been shown to positively prevent any carbon from reaching the work and which also supplies extra nitrogen to the part during sintering.

Fig. 6 illustrates three similar pilot plant size furnaces that are being used for binder removal and sintering silicon parts designed for a static structural application while subjected to the stresses created by thermal cycling at extreme temperatures. Again, the materials have been selected to allow sintering at only modest nitrogen overpressure in the expectation that success in part development will quickly lead to practical low cost production including a single step binder removal and sintering operation.

In order to densify with the least quantity of additives which reduce its strength and temperature tolerance, silicon nitride must be

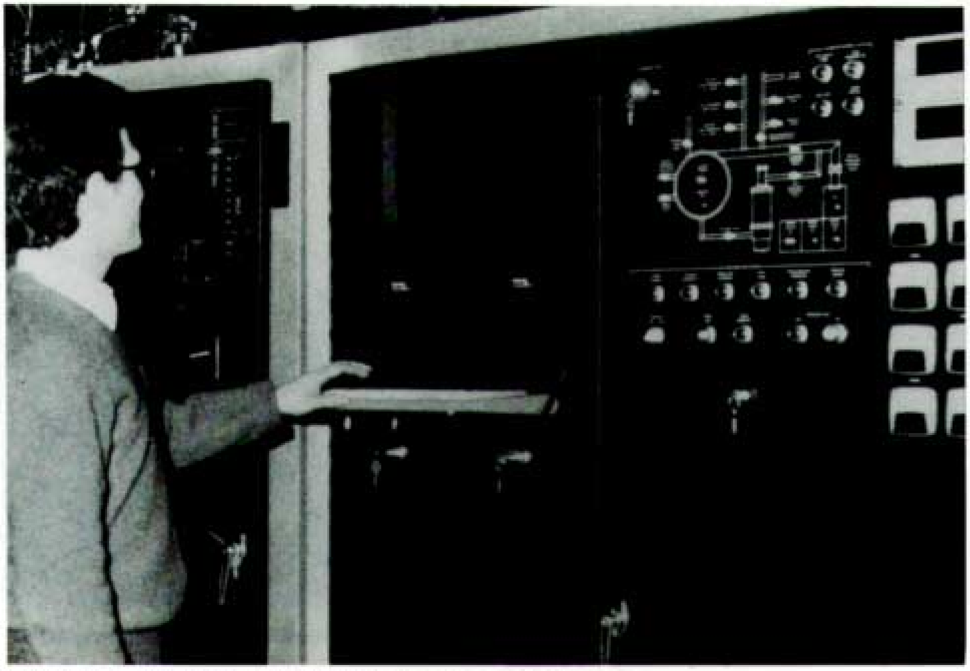

FIG 4 Computer control system applied to combination furnace

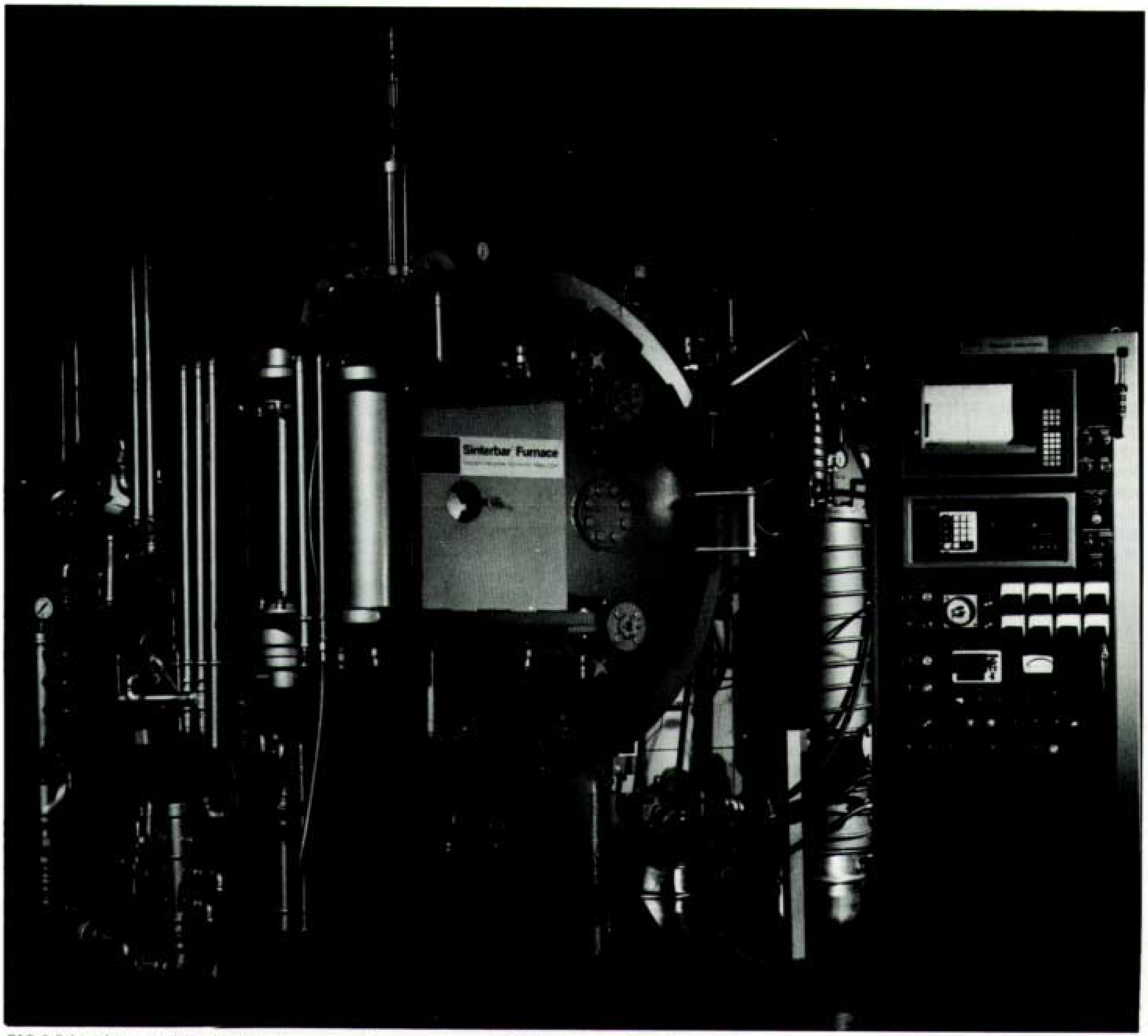

FIG 5 Pilot plant combination binder removal, pressure consolidation furnace for silicon nitride articles

FIG 6 Three pilot plant pressure furnaces for binder removal, reaction bonding, sintering silicon nitride

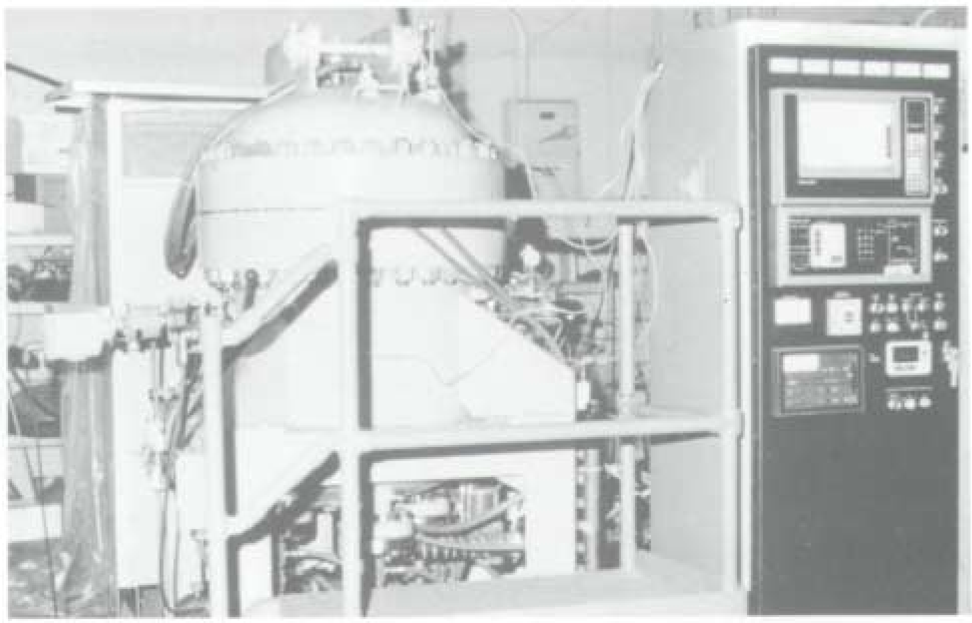

FIG 7 Front view of 2200C, 100 bar, 15 liter carbon-free furnace for silicon nitride article development

sintered at temperatures in the order of 2200C. As this temperature is approached, the pressure of the nitrogen gas surrounding the work piece must be increased to 100 bar to prevent dissociation and the resulting melting of the free silicon. This phenomenon is succinctly described in Prochazka U.S. Patent 4,374,792 as well as in several other citations in the literature. In order to provide the process development engineer with the greatest possible flexibility in his work, the furnace illustrated in Fig. 7 was developed and is in operation. It provides approximately 15 liters work volume with a maximum temperature of 2200C and 100 bar isostatic pressure in a totally carbon-free hot zone. The use of setter powder is thereby eliminated and another step is taken along the road to production.

From the foregoing, it may be seen that the pressure furnaces delivered for use with structural ceramiçs are relatively small pilot plant units suitable for development work. The technology, however, is directly transferrable to production furnaces and, indeed, larger furnaces are now in the design stages.

BINDER REMOVAL

One of the major problems affecting high production of engineering ceramics is the need for more rapid removal of the binders used when parts are injection molded or slip-cast. Binder removal cycles ranging from many hours to days are reported for air burn-out even when oxygen-assisted.

The Sweepgas^{TM} system developed by Vacuum Industries offers one means of removing and condensing binder breakdown products in combination furnaces, but it is somewhat limited as to types of binders, most suitable pressures, etc. Although it appears to meet some needs, much further development work is needed.

At present, the state of the art in injection molded metal powders of 1 to 5 micron size allows complete removal of two part binders in as little as 8 hours depending upon section thickness. This new technology development has made practical the use of combination furnaces for binder removal and sintering in this field. Extension of this new technology to fine ceramics could well be a first step on the way to low priced ceramic parts.

SUMMARY

In summary, combination debinder, sinter, pressure consolidation furnaces are now viable production units for the hardmetal industry and are becoming important processing plants for the new engineering ceramics as they move from the development stage to production. Their effectiveness has been established and refinements in operation range, larger sizes, and associated gas control and binder handling system will further increase their application and use.

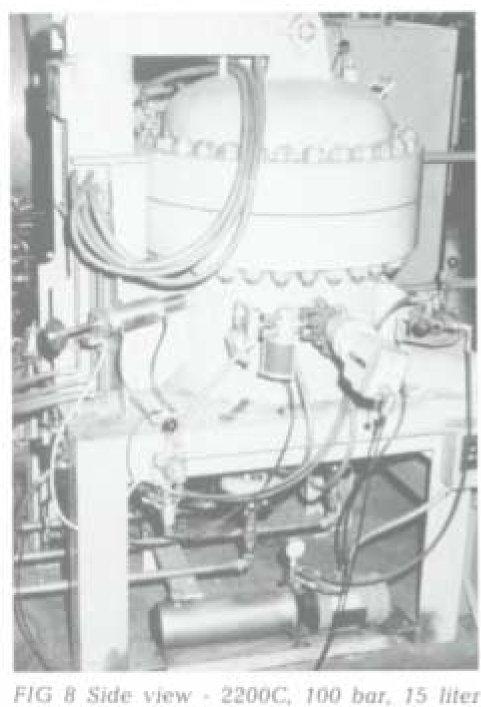

FIG 8 Side view - 2200C, 100 bar, 15 liter carbon-free furnace