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TECHNICAL NOTE

Reprinted from METAL POWDER REPORT
Vol. 37, No. 11, November 1982

Vacuum Hot Press Furnaces for Powder Compaction

The unique feature of vacuum hot pressing of powders is its ability to combine high pressure, temperature, and time simultaneously in a controlled environment. Kenelm W Doak of Vacuum Industries Inc. in Somerville, MA, describes developments in this field.

Hot pressing has been characterised as the more general example of materials fabrication technology, simultaneously utilising varying conditions of temperature, pressure and time to form the desired product (1). Processes involving time-temperature alone include sintering, fusing and casting. Pressure-temperature processes include hot forming operations such as forging and rolling. Pressure-time processes cover the cold forming operations such as pressing, pressure welding and cold drawing. High-rate and energy processes including explosive forming and impact forging, provide intense pressures in a minimum of time. The unique feature of hot pressing is its ability to combine high pressure, temperature and time simultaneously in a consolidation process (Fig. 1).

When a hot press is combined with a vacuum chamber, a fourth variable, the surrounding environment, can also be controlled. A high vacuum or a controlled atmosphere of a desired gas mixture can be provided. In Fig. 2 residual impurity levels are compared in commonly used units. A relatively easily attained vacuum level of 5×10^{-4} torr is equivalent to a residual impurity level less than 1 ppm. It was the special feature of vacuum environment which influenced one of the earliest applications for consolidating metal powders by this method (2). The need for classified shapes of beryllium for nuclear applications in the 1940's pointed the way to the PM route to obtain a usable fine grain material. Vacuum hot pressing was selected for final consolidation to minimise oxide formation.

INDUSTRIAL USE

Some of the largest vacuum hot pressed products produced today continue to be made from beryllium, part sizes range from 75 in. dia. x 24 in. thick to 10 in. dia. x 60 in. long with total weights approaching 5,000 lbs (3). The pressing temperature is around 1075C and densities of up to 99.7% of theoretical are attained. Using graphite moulds, parts up to 20 in. dia. can be pressed at 1200 psi. The outside diameter of graphite moulds is limited by the availability of the material. The present maximum size is 57 in. The technique of stacking alloy steel rings is also used and permits pressing at unit pressures up to 600 - 700 psi.

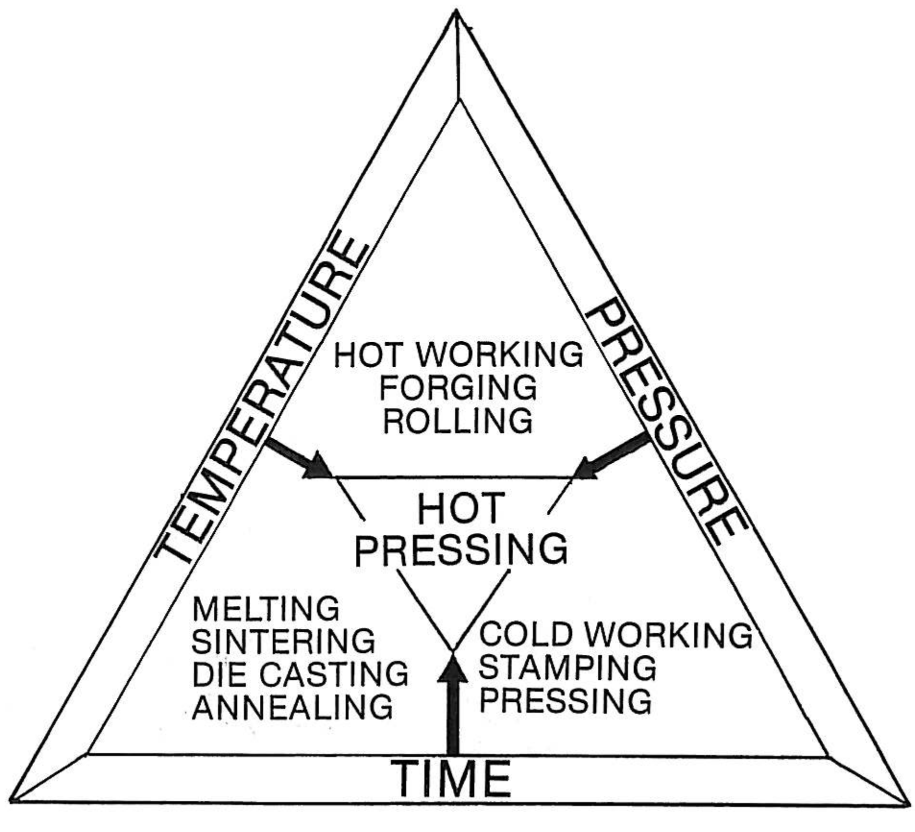

FIG. 1 Relationship of hot pressing to other forming and consolidation processes

Whilst beryllium may be an isolated example of a special purpose material being processed almost exclusively by vacuum hot presses, vacuum hot presses have found their way into inumerable research and development operations in industry, governmental and private institutions. Hot pressing as such was the process of choice of compacting practically all refractory carbides, borides, nitrides, silicides, oxides and numerous combinations thereof (1). Hot pressing techniques for moulding silicon and boron carbide wear resistant shapes were well established by the mid 60's. Almost every hot press in use at that time was made by the user and employed either resistance or induction heating. Graphite tooling was most common. Temperatures range from 1300 to 2800C and compacting pressures varied from 1000 to 10,000 psi.

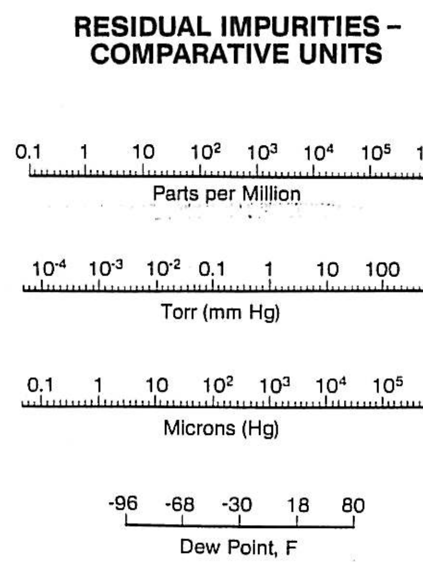

FIG. 2 Residual impurities in comparative units

VACUUM HOT PRESS FEATURES

Spearheaded by the early work on beryllium in the late 40's and reinforced by the push for developing new and improved materials in the 50's, vacuum furnaces of all kinds were developed and put into service for thermal processing of advanced materials. Vacuum hot presses have been introduced in a number of standard models since then and a typical laboratory size press is shown in Fig. 3. A common vacuum press design provides a front opening vacuum chamber for easy access to the die and plunger area. The furnace is mounted in a fixed position on the press frame and does not require movement in order to load and unload.

The furnace assembly is mounted within the vacuum chamber with powder feed throughs for the resistance or induction heated hot zones. Penetrations in the top and bottom of the vacuum chamber equipped with vacuum seals are fitted with water cooled rams connected to hydraulic cylinders on the press frame (Fig. 4). Hot tooling, most frequently graphite, is mounted on a high temperature graphite die support, and surrounded with insulation on the top, sides and bottom. In a high frequency induction furnace, the coupling can be direct to the die or to an intermediate susceptor. The die support pedestal is mounted directly on an external support frame to avoid stressing the vacuum chamber.

Most laboratory-scale vacuum hot presses can accommodate dies up to 5½ in. OD in the smaller sizes and 8 in. dia. in the larger sizes (Fig. 6). Custom vacuum hot presses can be produced in practically any size. Size limits for PM production scale presses have been mentioned above. A related group of vacuum hot presses, comprising a complete family of diffusion bonding units, are used to produce aircraft propulsion unit components and airframe members. A large number of different press configurations have been produced and press capacities range up to several hundred tons.

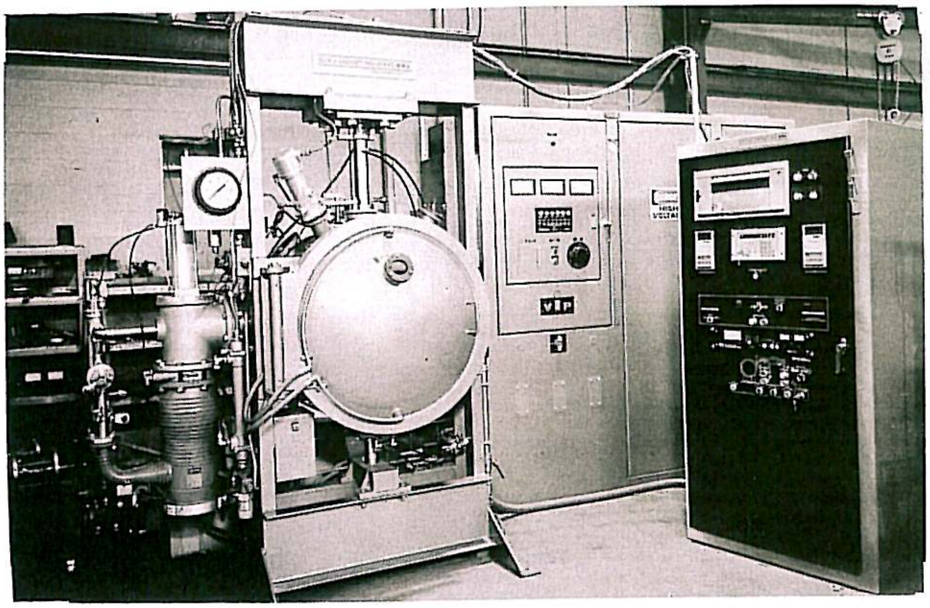

FIG. 3 High vacuum hot press, 30 ton capacity, 3 in. OD compact size, 2300C, H.F. induction heated. Top ram active, bottom fixed. Dual channel programming: Temperature pressure vs. time. LVDT extensometer with digital readout and analog recording.

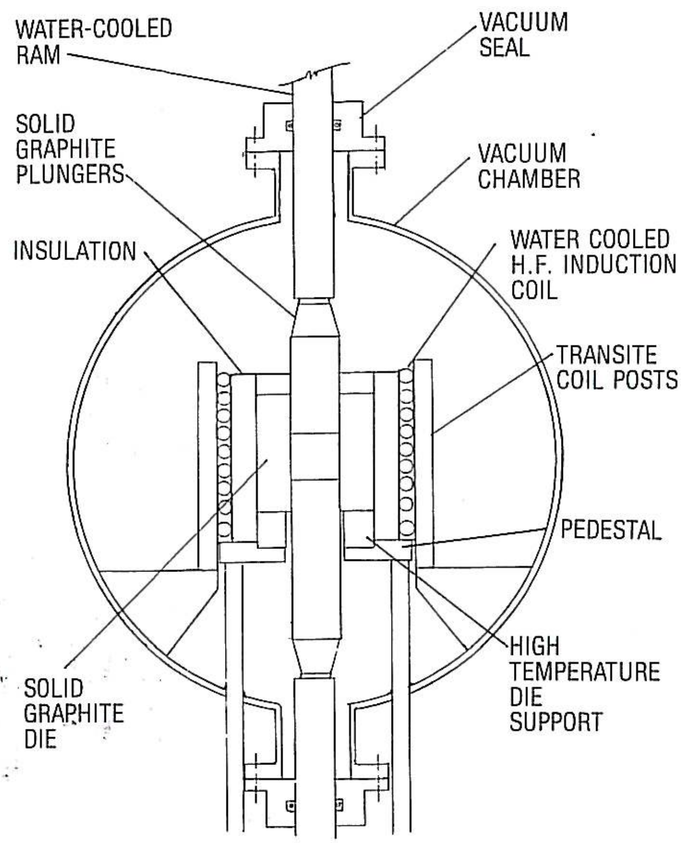

FIG. 4 Schematic diagram of typical vacuum hot press with graphite tooling; press frame and hydraulic cylinders not shown

HOT EJECTION

The earliest laboratory vacuum hot presses consisted of a simple press frame with a manually operated hydraulic cylinder on top. The bottom ram was actually fixed and the die support pedestal was spring-mounted to provide for die movement during compaction. Later presses included motor-driven hydraulic systems to replace the manually operated pump.

As an aid in compaction and to provide for hot ejection of a pressed compact after consolidation, presses with active rams at the top and bottom are now often supplied. In this design, either the top or bottom ram can be locked into position and force applied on the opposing ram. This application of force can be alternated to achieve desired compaction characteristics. At the end of the pressing cycle, the two rams can be moved downward in concert to eject the compact from the bottom of the die cavity. Certain ceramic compounds expand during cooling and the hot ejection feature is necessary to avoid sticking within the die.

In addition, hot ejection permits a cooling gas medium to be circulated within the working area of the furnace to assist in cooling. Vacuum hot presses have been equipped with gas recirculation loops incorporating blowers and heat exchangers which direct cooled gas into the working area of the furnace. Production vacuum hot presses utilise such systems to improve throughput.

AUTOMATIC CONTROLS

Digital programmable controllers, widely used on vacuum furnaces of all types, have found particular application for vacuum hot press equipment. Two- and even three-channel programmers can be used for vacuum hot presses to achieve pre-

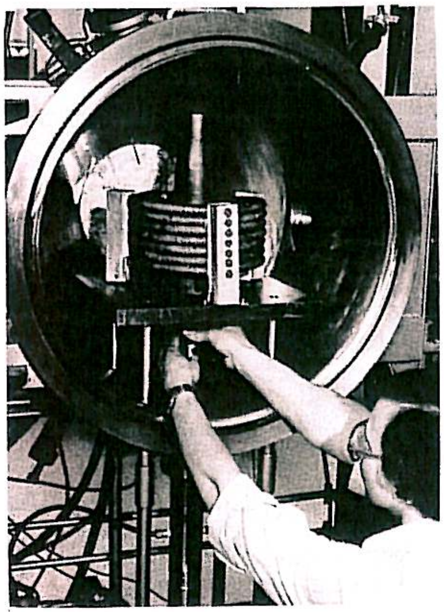

FIG. 5 Tooling shown being installed in an induction heated vacuum hot press used for synthesising new ceramic and metal matrix materials (photo courtesy of Bendix Advanced Technology Center, Columbia, Md.)

determined temperature and pressure ramps with respect to time. Temperature is controlled by either thermocouple or optical temperature sensors. Hydraulic pressure is programmed through pressure sensors in a servo-controlled loop in the hydraulic system. The force applied on the compact is calculated from the pressure applied on the hydraulic cylinder piston. Presses have been instrumented with load cells in the load train to measure direct force in pounds or kilograms.

Special purpose vacuum hot presses have been equipped with a third channel of control connected to the atmosphere and vacuum system. It is therefore possible to adjust the vacuum level and even introduce partial pressures of various gases during a pressing cycle as required for the material being processed.

Additional instrumentation on hot presses can include extensometers to measure the displacement of the active ram (or rams). With the use of linear variable differential transformers (LVDT's) the motion of either ram or net motion of both rams can be measured, displayed digitally, and recorded. Sophisticated press designs include provisions for using ram displacement as a controlling parameter for hydraulic pressure. With the programmable controllers, preset values of process variables can be used to activate interlocks or initiate other events in the overall cycle. In this way, the vacuum hot press has become more versatile and its operation has become simpler. Pre-programmed cycles can easily be repeated in order to duplicate pressing conditions on subsequent runs.

Vacuum hot presses for powder compaction can be supplied with hydraulic systems that actually exceed the capabilities of most tooling. Standard laboratory vacuum hot pressing systems are supplied with load frames and hydraulic systems designed for applied forces of 30 tons. Using high purity graphite tooling (such as ATJ), the maximum compressive force that can be applied on a 1 inch compact in a 5½ in. OD die ranges from 1 ton at 1700C to 1.4 tons at 2300C. This loading corresponds to a die hoop tensile strength of about 3100 psi at the lower temperature and 3750 psi at the higher temperature.

As the compact size is increased, the maximum total force can be increased because it is distributed over a greater surface area. For a three inch compact in the same size die, the maximum force at 1700C is 6 tons and, at 2300C, slightly over 7 tons. The advantage of graphite tooling is its availability and relatively low cost. Moulded graphite is an attractive material because of its high compressive strength and the fact that its strength does increase with temperature.

Since many researchers want to apply higher forces to achieve greater unit loadings, an alternative which can be considered is filament wound graphite composite structures. The hoop tensile strength for some commercially available filament wound graphite tooling materials is reported as being over 100,000 psi (4). Using a value of 70,000 psi for calculation, the allowable press loading on a 1 in. compact in a 5½ in. OD die could theoretically be 25 tons and the corresponding press load on a 3 in. compact could be 132 tons. The disadvantage of this type of tooling is its cost which is 10 to 20 times the cost of machined moulded graphite. As this tooling is custom fabricated, lead times can be exceedingly long. Suppliers of this type of tooling recommend the use of die liners of machined moulded graphite as a sacrificial component. In case of damage during pressing, only the die liner made from machined graphite would have to be replaced.

Other materials which have been used for the load train in vacuum hot presses include aluminum oxide, silicon carbide, silicon nitride, refractory metals and their alloys.

APPLICATIONS

Vacuum hot pressing, because of its versatility, has been used to consolidate practically all types of engineering materials

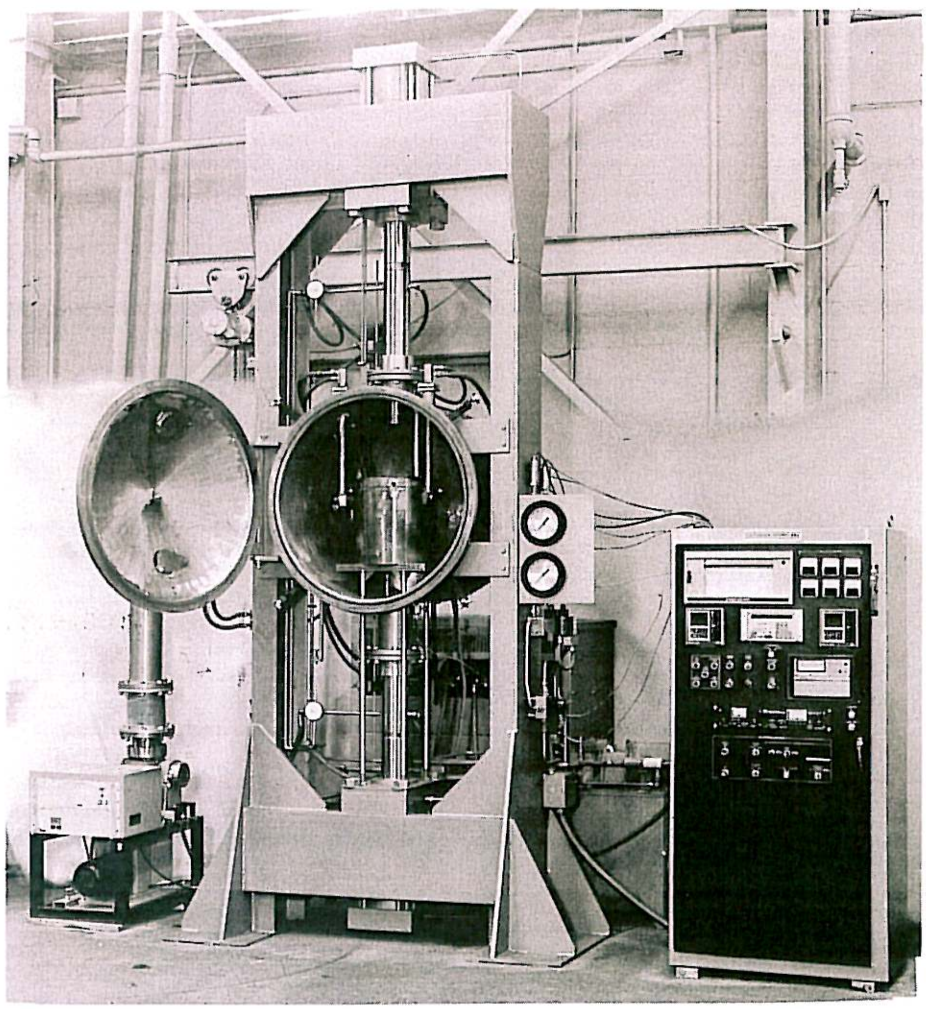

FIG. 6 Vacuum hot press for ceramics research; 100 ton capacity, 5 in. OD compacts, 2300C resistance graphite heated. Two active rams. Turbomolecular vacuum pumping system. Dual temperature and hydraulic pressure programming. (Photo courtesy of International Harvester, Hinsdale, Illinois)

including metals, ceramics, and composites. This practice is particularly applicable to high temperature engineering ceramics and especially the silicon-based carbides and nitrides (5).

While most schemes for actually producing high temperature engineered ceramics in sufficient quantities to make them economically feasible involve other processes, vacuum and controlled atmosphere hot pressing remains the basic process for attaining maximum mechanical properties. The properties of materials produced by this process can be used as a benchmark for comparing properties of these materials produced by other purposes. For specialised applications requiring maximum properties, hot pressing remains the preferred route.

The broad range of materials which have been vacuum hot pressed indicates the wide acceptance of the technique. In one instance, to study absorbed layer effects, fine ceramic spray-dried powders were hot pressed in vacuum and used as a standard of comparison to determine the effect of such additions on such properties as bulk density, elastic recoil and behaviour in multiple pressing (6). Studies involving hot pressing single pellets have indicated that transparent magnesia can be produced, the ideal conditions being 1000C and 1,000 psi for a ten-minute press cycle under high vacuum. A continuous hot press referred to in this work reportedly incorporated silicon nitride as a die-set material (7).

In the field of metal-matrix composites, aluminum-coated carbon fibres were used to manufacture a reinforced aluminum composite by vacuum hot pressing (8). A recent news announcement describes a fibre reinforced metal called 'ceramic fibre alloy' for automotive applications in fuel-efficient parts, such as piston ring grooves in diesel engines (9).

The range of materials being processed in vacuum hot presses ranges from the familiar to the most exotic. Not surprisingly, studies on the consolidation of ceramic radioactive waste have also included the use of vacuum hot pressing (10). The ability to process reactive and refractory materials without the need for encapsulation in protective cans and the ability to carefully adjust and vary applied pressure and temperature as well as environment makes the vacuum hot press a very versatile tool.

FUTURE TRENDS

Although semi-continuous vacuum hot press equipment has been available for many years for diffusion bonding operations, production vacuum presses for powder consolidation have primarily used the hot ejection and forced cooling with recirculated gas technique to increase throughput. If vacuum hot pressing is selected as a production technique for any emerging engineered material, semi-continuous presses with provisions for faster recycling can and will be developed.

Although isostatic hot presses have now become available in laboratory sizes at very competitive prices, there seems to be continuing interest in both vacuum hot pressing and in hot isostatic pressing in materials development. Both types of pressing equipment have unique capabilities which make them ideal for certain applications. The continued development of even more versatile vacuum hot presses with the inherent capability of controlled atmosphere processing is clearly indicated in order to meet the requirements of the advanced materials development community.

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