Hot Isostatic Pressing of Metal Powders

J.J. Conway and F.J. Rizzo, Crucible Compaction Metals

Introduction

HOT ISOSTATIC PRESSING (HIP) is a process involving the use of high-pressure gas isostatically applied to a part or workpiece at an elevated temperature performed in a specially constructed pressure vessel. When consolidating metal powders in pressure-tight, sealed compacts, the HIP process plastically deforms the powder, which closes up porosity and achieves 100% theoretical density in the part. Thousands of fully dense HIP P/M compacts (equivalent to >10,000 tons) are commercially produced each year. This includes net shapes, near-net shapes, and a variety of mill forms for subsequent thermomechanical processing. Monolithic and bimetallic or clad parts are part of the near-net shapes that are supplied in many different conditions such as: HIP, HIP plus heat treated, HIP plus thermomechanically processed, and so forth. The majority of individual parts are made as near-net shapes. Other parts are made as net shapes, or with some net surfaces and other surfaces that need finishing to match mating parts or to meet some other critical parameters. The elevated temperature in HIP ranges from approximately 480 °C (896 °F) for aluminum alloy powders to 1700 °C (3092 °F) for tungsten powders. Most of the commercial HIP activity is with steel and nickel alloys, which are commonly hot isostatically pressed between 1100 °C (2012 °F) and 1205 °C (2201 °F). High-density argon gas is the most common pressure medium used in the process, although other gases such as helium or nitrogen can also be used. Pressures ranging from 20 to 300 MPa (3 to 45 ksi) are possible with 100 MPa (15 ksi) being the most common.

The HIP process was invented at Battelle Memorial Institute in 1955 as a method to diffusion bond dissimilar materials where Zircaloy was clad to uranium oxide nuclear fuel elements (Ref 1, 2). As the technology evolved, applications other than diffusion bonding were discovered, namely consolidating encapsulated powder, porosity healing inside castings, and densification of presintered components. In the 1960s, HIP technology (sometimes referred to as gas pressure forging) was used to consolidate beryllium metal powder, refractory metal powder, ceramic powder, and cemented carbides. Not until the 1970s, however, did the HIP process expand to an industrial scale. For example, high-volume hot isostatic compaction of high-speed tool steel was achieved in the United States (Ref 3) and Sweden by 1972. By the late 1970s, the United States Air Force Materials Laboratories funded work to develop HIP technology to manufacture titanium and superalloy components for aircraft engines. This application currently represents the highest technology level of the process. Powder metallurgy tool steels represent the highest production tonnage.

Some of the current applications of HIP P/M parts include:

- **Tool steel billets** (for mill processing) for hot and cold working tools and dies
- **Nickel and titanium alloys** for high-temperature components on aircraft and marine gas turbine systems
- **Nickel alloys** in the oil and gas and petrochemical industries for corrosion-resistant components on wellheads and piping systems
- **Nickel alloys** in the nuclear power industry for corrosion-resistant components in reactors
- **Titanium and cobalt alloys** in the biomedical industry for prosthetic implants
- **Refractory metal alloys** for high-temperature service
- **Cemented carbides** for superior wear-resistant parts
- **Composite aluminum materials** for lightweight structural components

Although HIP technology is commonly used for sealing porosity in castings and P/M sintered compacts, this article focuses on the consolidation of metal powder and the diffusion bonding of dissimilar materials.

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Process Selection

During the design phase of a particular component, the end user must select a material and process that will best suit the requirements of the application given the constraints of economics, availability, and time. There are several different processing routes available to designers, among them are ingot metallurgy (I/M) and powder metallurgy (P/M).

Ingot metallurgy processing involves casting molten metal into molds, solidifying at relatively slow cooling rates (0.1 °C/s), and subsequently converting to the final product form by thermomechanical processing. The as-cast ingot exhibits chemical compositional variations, porosity, and a nonuniform microstructure containing equiaxed and columnar grains. Postcasting processing (e.g., heat treatment, thermomechanical processing, etc.) helps to homogenize the materials and somewhat ameliorate these anomalies. However, in some cases, conventional ingot metallurgy restricts the material from attaining its fullest potential in terms of properties.

The P/M processing route enables materials to achieve higher alloyed compositions without encountering segregation. Once fully densified by HIP, the P/M material has a fine grain size with an equiaxed microstructure. This uniform microstructure with a consistent composition leads to isotropic mechanical and physical properties. Not only do the properties have less variation than those properties found in I/M products, but the mechanical properties are often enhanced due to the fine grain size and the fine dispersion of precipitates available for strengthening. Better properties extend product life cycles or allow lesser grade materials to be used in more stringent applications. In addition, machinability is enhanced due to the uniformity and fine microstructure of P/M materials.

One example where the P/M route was selected over ingot metallurgy is René 95, which initially started as a cast-and-wrought (C/W) nickel-base superalloy in 1972. This highly alloyed material used for rotating disks in jet engines is prone to microsegregation, which during forging (even under careful controls) ultimately causes severe cracking and an undesirable microstructure (Ref 4). Later, the process was modified to use HIP P/M (Ref 5) and more than 100,000 of these parts are in aerospace service today.

Another example of the beneficial use of P/M versus I/M is in highly alloyed tool steels (Ref 6, 7). Through the years, producers of these materials have increased the content of carbide-forming elements such as molybdenum, tungsten, and vanadium to achieve increased wear resistance. Increasing alloying additions has led to increased segregation, which has resulted in lower product yields and lack of toughness. Due to the fine nature of the carbides in the P/M product and their high volume fraction, the wear resistance and toughness are greatly improved over I/M-produced tool steels.

HIP versus Other Powder Metallurgy Methods. Once powder metallurgy is established as the preferred processing route, other factors influence proper selection of a compaction process, namely:

- Properties required to meet component service requirements
- Size, shape, thickness, and complexity of the part
- Tolerances and surface finish requirements
- Cost, availability, and lead time

Because mechanical performance improves with increasing density (Fig. 1) (Ref 8), applications requiring maximum properties generally employ HIP for larger-sized parts instead of pressing and sintering (Fig. 2). By utilizing the combination of high temperature and pressure, HIP can achieve a particular density at lower pressure when compared to cold isostatic pressing (CIP) or at lower temperature versus sintering. In addition to the processes described in Fig. 2, there are processes other than HIP that can be employed to achieve near-full or full density, each with their inherent advantages and disadvantages. Cold isostatic pressing combined with some subsequent thermal process (e.g., sintering, HIP, extrusion, etc.) can provide full density for most materials. The CIP approach can be used when it is metallurgically advantageous to begin densification prior to exposure to elevated temperature. Pressure-assisted sintering (PAS) involves the simultaneous application of pressure and heat to a powder mass typically created by hot pressing and pressurized sintering. Sinter plus HIP (containerless HIP) involves sintering a partially dense compact produced by die compaction or CIP and applying heat to sinter in an autoclave with subsequent pressure for the HIP phase. Sinter plus HIP has been used for tungsten carbide, silicon carbide, and silicon nitride for high-performance applications. Consolidation by atmospheric pressure (CAP) involves applying heat to a glass-enclosed, vacuum-tight compact to consolidate powder that has been chemically treated with a sintering activation agent. Rapid omnidirectional compaction (ROC), or fluid-die process, involves using a conventional forging press and a pot die to compact powder in a cavity of usually a thick-walled shaped "fluid" container. The Ceracon process (CERAmic CONsolidation) involves taking a heated preform and consolidating the material by pressure against a granular ceramic medium using a conventional forging press. Consolidation of metal powders via extrusion can be performed by using either a blocker die or extruding loose powder to full density (see the article "Extrusion of Metal Powders" in this Volume).
Fig. 1 Effect of density on mechanical properties of two P/M steels (Fe-1.5%Cu-0.6%C and Fe-2.0%Cu-2.5%Ni). (a) Strength, elongation, and impact toughness. (b) Fracture mechanic properties. (c) Nominal stress amplitude for 50% fatigue failure at $2 \times 10^6$ cycles. UTS, ultimate tensile strength; TYS, tensile yield strength; YS, yield strength. Source: Ref 8
Three variables that dictate the P/M approach are size, density, and performance (as a percentage of wrought). This behavior corresponds to ferrous-base P/M systems, but is representative of many P/M materials. P/S, press and sinter; reP, press, sinter and repress; P/S + F, press and sinter and forge; CIP + S, cold isostatically press and sinter; HIP, hot isostatic press; HIP + F, hot isostatic press plus forge. Source: Ref 9

Although these processes show promise, each has limitations. Some do not achieve full density (e.g., CIP and PAS). Others require subsequent processing (e.g., CIP and CAP), while others have not been scaled to production levels (e.g., Ceracon, ROC, and CAP). The HIP process is currently the most commercially viable method to produce fully dense P/M material. In excess of 10,000 tons of HIP P/M material is produced annually, far exceeding all of the other techniques.

When end users select the HIP P/M processing route to manufacture their parts, it is typically due to the following characteristics, which are desired for the final application:

- Full density of highly alloyed materials that usually cannot be produced by I/M processing
- Near-net-shapemaking capability
- Fine grain size
- Isotropic physical and mechanical properties
- Improved mechanical properties
- Extended life cycle
- Cost-effective components
- A range of possible part sizes from 1 to >10,000 lb
- Combinations of the above

Although cost of HIP P/M parts are often higher than the competing I/M or press-and-sinter P/M produced parts, HIP P/M can be an affordable method to increase part performance, reduce equipment weight (e.g., stronger material therefore thinner cross sections, etc.), maximize material usage by producing near-net shapes, or realize cost savings by increasing life cycles, eliminating downtime, and reducing maintenance and replacement costs. In general, the main cost elements for the HIP P/M process are the prealloyed, gas-atomized powder, the container (and container tooling if needed), HIP vessel efficiency (e.g., maximizing the number of parts during a single HIP run of the autoclave, Fig. 3), and postconsolidation processing. The combined considerations for customer requirements and the process cost elements determine if HIP P/M processing can be used for a given application. To properly assess each application, the end user should make a comparison with other production processes such as casting and forging. This should involve a total engineering evaluation that includes acquisition cost, finish part manufacturing cost, and component life-cycle cost.
**Fig. 3** HIP processing cost as a function of press size. The cost per unit work-zone volume to run a HIP cycle depends on the size of the HIP unit. Increasing the diameter of the work zone reduces the cost, but above 0.8 to 1 m diameter, further gains are small. Still larger HIP units are built to handle specific products. Source: Ref 10

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**HIP Technology**
Whether it is consolidating powder in an encapsulated compact, bonding two surfaces together, or healing a casting or sintered compact, HIP is a process to remove porosity. The following describes the mechanisms and stages of pore closure for metal powders and HIP diffusion bonding of two surfaces.

**Consolidation of Encapsulated Powder.** As will be described in more detail later, powder particles are randomly packed inside a container that encapsulates the powder during the HIP process. Powder used for HIP is typically spherical with a broad particle size distribution. This is better for achieving high packing density when compared to irregularly shaped particles and/or monosized particles. With monosized particles or powder with narrow particle size distributions, there are no small particles to fill the interstices, thus decreasing packing density. However, even with the ideal size distribution, there will always be some percentage of void space due to the spherical morphology (Fig. 4) of the powder and possibly the presence of hollow particles (Fig. 5) that may have formed during the powder-making process.

![Diagram of pore with varying radii of curvature](image1.png)

**Fig. 4** Pore with varying radii of curvature around the surface. The broken curve shows the pore surface after some spheroidization has taken place by redistribution of material from convex surfaces to concave. Source: Ref 1

![Image of pores in -80 mesh Astroloy powder particles](image2.png)

**Fig. 5** Pores in -80 mesh Astroloy powder particles produced by argon gas atomization. Courtesy of Industrial Materials Technology, Inc.

To describe the consolidation of metal powders and thus the removal of pores, it is generally believed that the HIP process is similar to sintering where three physical mechanisms (Ref 11) are present, namely:

- Powder consolidation
- Neck growth
• Final densification

Powder in compacts prior to HIP is packed at a relatively low density (e.g., 60 to 80% of theoretical density). During the powder consolidation phase, powder is considered a cohesionless granular material where particles can slide freely without particle deformation. Particle rearrangement occurs, and some macroscopic deformation is observed. Once a particular density is attained and particle rearrangement discontinues, necks grow at the contacts between particles. At this stage, particles are beginning to bond, but the porosity is still interconnected. Various mechanisms describe this phenomenon as follows:

• Plastic deformation of the particles by dislocation
• Power-law creep or dislocation creep
• Nabarro-Herring creep or volume diffusional creep
• Coble creep or grain boundary diffusion creep

During final densification, the material may be considered a solid containing isolated pores connected by grain boundaries. The mechanisms controlling the final densification stage is the same as those controlling neck growth (Ref 11). A more detailed description of the mathematics is given in the article "Principles and Process Modeling of Higher-Density Consolidation" in this Volume and elsewhere (Ref 11, 12, 13, 14).

Interface/Diffusion Bonding. Hot isostatic pressing technology was initially developed as a method to diffusion bond two dissimilar materials. Unlike fusion methods (e.g., welding, brazing, etc.), diffusion bonding depends on atomic transport across two mating surfaces to remove bond line pores. Fusion processes involve melting, which can cause segregation or solidification cracking. Unlike consolidation of a powder compact, components undergoing HIP diffusion bonding deform very little macroscopically. However, microscopically there is localized plastic flow across the bondline with the size of the initial pore being a function of the surface roughness (Fig. 6). Depending on the mismatch in physical and mechanical properties of the two materials, pores at the interface will spheroidize due to surface energy considerations. Afterward, previously described mechanisms take over until final densification is reached (Ref 1).
Fig. 6 (a) Magnified view of a region where two materials come into contact showing surface roughness. (b) Magnified view of the same region after microplastic deformation. (Material B is harder than material A.)

Source: Ref 1

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Process Description

The interaction of the HIP process and equipment must be understood to help explain the technology. There are two basic types of autoclave used for HIP. The first is known as a hot-loading cold-wall vessel where the parts to be densified are heated to the HIP temperature external to the high-pressure chamber. The second type is known as a cold-loading cold-wall vessel where the parts and furnace enter the high-pressure chamber at room temperature and are sealed in place. The heating and pressurizing of the parts and the vessel are then done according to a preprogrammed set of parameters.

A typical HIP cycle for a hot-loading cold-wall HIP unit is as follows:

1. The powder-filled compacts would be placed on fixturing designed to hold the load at elevated temperatures. Some compacts can be self-fixtured by attaching a lifting device to the container.
2. The compacts and fixtures are placed into a furnace and heated to the desired temperature at a controlled rate or progress through a series of preheat and high-temperature furnaces to a hold point. Because the thermal conductivity of loose powder is much less than that of fully dense material, long hold times are necessary to ensure the entire cross section of the largest compact in the load is thoroughly heated and ready for HIP.
3. Once heated, the compacts and fixture are lifted from the furnace and placed into the pressurizing vessel. The load may be protected with insulating material during the transfer to prevent cooling prior to placement in the HIP unit.
4. The lid is placed on the vessel and sealed.
5. A pressure valve is opened, and gas at high pressure is released from a reservoir into the densifying chamber until there is equalization in both tanks. The vessel is held at these conditions for a set period of time, which is usually short, and thus lends itself to high production rates.
6. The gas is pumped from the densification chamber back into the storage reservoir, and the connecting valve is closed.
7. The lid is removed from the HIP chamber, and the load is removed.
8. The load is cooled to room temperature either in air or in another chamber (e.g., a furnace) at a predetermined rate to protect the parts from undesirable reactions or microstructural phase transformations.
A typical HIP cycle for a cold-loading cold-wall autoclave is as follows:

1. The powder-filled compacts are placed on fixturing designed to withstand the load weight and elevated temperature to which it will be subjected. This is usually done at a station that can also accommodate the furnace. To maximize efficiency, the load is built in a duplicate base heater while the vessel is actively running a cycle.
2. The furnace is placed over/around the compacts and the base heater. The entire load is lifted and placed into the HIP vessel as a module. The base heater engages outlets in the bottom of the vessel, and the process is ready to start. The vessel is closed by putting the lid in place and sealing it properly via threads or a yoke.
3. The vessel is then evacuated and purged with inert pressurizing gas sufficient to remove all the traceable remnants of air and/or moisture from the vessel.
4. High-purity gas, usually argon, is discharged into the vessel from the gas storage tanks until the pressure is equalized.
5. The compressor is turned on, and vessel pressure is increased via pumping until the hold pressure is reached. For most cycles, heating takes place during high-pressure purging, contributing to the pressurization. In fact, gas expansion is responsible for the largest contribution to the high pressure.
6. The load is heated to the desired holding temperature previously determined and based on material properties and so forth at some predetermined rate. The duration of the hold is based on the nature and size of the load.
7. After the cycle completes the established hold conditions, the load can be cooled in three ways. First, the furnace can be turned off, and the entire system will cool naturally to a temperature low enough to protect the furnace elements and to enable the load to be handled safely. This rate will vary with vessel size and with the amount and configuration of the load. Second, the furnace can be programmed to cool slowly to prevent a material problem such as unwanted excess residual thermal stresses. Third, the furnace can be rapidly cooled by circulating new cooler gas to replace the high-temperature gas in the vessel (Ref 15). This process can be used to improve cycle turnaround time or affect a quench of the material in the HIP unit. Regardless of cooling cycle type, the gas can either be vented to the air or recycled for future use.
8. The HIP charge is removed from the vessel when the temperature is appropriately low. Another charge built during the cycling of the furnace charge replaces it in the vessel, and this process repeats itself.

The cycle times for a given vessel depend on the load and the size of the autoclave. However, a typical floor-to-floor time for medium- to large-size vessels is a matter of minutes in hot-loading units and 24 h or less in cold-loading units. The cycle time in a hot-loading unit is generally a matter of minutes. These run usually 30 min or less. However, to achieve maximum benefit from this type of system, the preheating furnace capacity must be extensive. The charge will generally require 12 to 24 h of heating before it is ready to be introduced into the HIP unit. Nearly all of the autoclaves used extensively in the HIP P/M industry are the cold-loading cold-wall style because they can achieve a more reproducible cycle, which is necessary for near-net shape processing. The hot-loading cold-wall wall vessels are used where high volume is needed and the product is a compact or preform for reprocessing.

Selection of Specific Cycle Parameters. Generally, HIP of P/M material produces fully dense material in the shortest process time, using pressures within the rating of equipment large enough to accommodate the parts being processed. Control of the fully densified microstructure may also be required for HIP P/M parts, especially if they will be subsequently hot worked. For most materials, the temperature of HIP is the primary controlling factor and is usually at least 70% of the melting point of the material. With most materials, the HIP temperature is specified by the end user because of its effect on the subsequent microstructure and properties of the product.

There are potentially an infinite number of HIP cycles available, and the rate of heating and pressurizing the vessel is of primary importance to the shape of the parts being produced. Work by Evans and Malley (Ref 16) showed that by varying how heat and pressure are applied, the shape of a simple cylinder can vary dramatically. All cycles can be classified into three basic categories: (1) heat up first and then pressurize, (2) pressurize first and then heat, or (3) heat and pressurize simultaneously. How the load of parts reaches the hold parameters determines the shape of the parts. This means that reproducibility of the HIP cycle is most important to the reproducibility of any given part and is a key variable in obtaining a desired shape.
Consolidation of Encapsulated Compacts

When metal powder is consolidated via HIP, encapsulation is required to act as a barrier between the high-pressure gas and the powder to provide the differential force required to create the plastic deformation of the powder. Powder is typically placed in a container or envelope made of metal or glass to a specified shape larger than final desired size. There are three main components to producing a HIP-encapsulated P/M part, namely: powder manufacturing and processing, container manufacturing, and compact manufacturing.

**Powder Processing**

**Powder Manufacture.** Although crushed/irregular, mechanically alloyed, and hydride/dehydride powders can be used for HIP processing, prealloyed, spherical powder produced via gas atomization is most commonly used throughout the HIP industry. The primary advantage of spherical powders is their high pre-HIP packing density (e.g., 60 to 80% or greater of theoretical density) and relatively low variation in packing, which provides repeatable packing density in production. As the initial packing density increases, the probability of predicting final fully dense shape is higher, thus the more reproducible the HIP P/M process becomes.

For HIP P/M parts, the most commonly used powder-making process is inert gas atomization, which entails creation of powder by impinging high-pressure inert gas on a molten metal stream to disintegrate the metal into tiny liquid droplets. Due to surface tension considerations, the liquid droplets form spheres that rapidly solidify into powder particles at a rate of $10^4$ to $10^8$ °C/s (Ref 17). This rapid solidification provides a powder particle with superior chemical homogeneity not possible with conventional ingot metallurgy. In general, several alloy systems can be gas atomized successfully, namely those based on iron, nickel, cobalt, niobium, titanium, aluminum, copper, silver, and so forth with the molten metal containment (i.e., refractory or skull melt) being the limiting factor.

In addition to gas atomization, spherical powder can be produced by other methods that utilize centrifugal force as the energy mechanism to break up liquid metal. The rotating electrode process (REP) and the plasma rotating electrode process (PREP) are two such methods. During REP, the face of a rotating bar is melted by a direct-current electric arc maintained between the electrically negative tungsten tip and the positive alloy bar (Ref 18). The liquid metal is centrifugally ejected from the outer edge of the rotating bar and solidifies in flight as spherical particles, which are collected in a sealed chamber. While REP is normally used to produce iron-base powders, applications requiring superior cleanliness (e.g., titanium, nickel alloys, etc.) can be produced by PREP. During PREP, the plasma torch uses a dc arc to melt the rotating bar with a water-cooled tungsten cathode that is protected by helium gas to minimize erosion (Ref 18). Another mechanical atomizing technique is the rotating disk process where a molten metal stream is impinged on a rapidly spinning disk. As with the first two processes, the liquid metal is spun off the disk and centrifugally atomized into spherical droplets, which solidify in flight. Particle cooling can be enhanced by blasting the emerging particles with a stream of helium (Ref 19). Centrifugally produced powder typically has a narrower particle size distribution and lower packing density than gas atomized powder.

Another process that is commonly used to make titanium powder is the hydride/dehydride process. Titanium sponge is charged with hydrogen and thus embrittled. The particles are mechanically crushed and dehydried to yield fine powder particles. This powder is not as pure as that made by the PREP or inert gas atomization, nor is it spherical. However, it
can be used to make HIP components. Powder of other refractory metals such as molybdenum and niobium can also be made in this way. It is also possible to make alloys of these elements using this process.

Powders can also be made by crushing and grinding ingot material. This process is usually used for highly alloyed materials and is not ideal for HIP P/M because of the inherently low packing density associated with this manufacturing method. These powders are often used with the HIP cladding process because they can be highly alloyed to achieve a specific property such as wear and/or corrosion resistance. These powders have a tendency to be extremely nonhomogeneous.

**Powder Particle Size Classification.** Classification may involve separating powder by particle size, typically via vibratory screening. Air classification through specially designed cyclones has also been employed. Bulk screening processes involve feeding raw powder onto the vibratory screener. Two exit ports from the screener are connected to separate collection bins to segregate the oversize and undersize fractions. The prime powder (usually the undersize) is used to manufacture HIP P/M parts, and the other fraction (usually the oversize) is reprocessed through melting.

**Powder blending** is performed to homogenize the particle size distribution. Hot vacuum blending is used to remove adsorbed gases from the surface of powder particles. Uniform particle size distribution is essential to HIP P/M near-net shape parts to minimize distortion during the consolidation step of the process. If a near-net shape is not being produced, then blending of the powder is not an essential step in the process and is often omitted. For some applications, blending can be used to achieve the desired chemistry of the end product. This can be as drastic as blending of elemental powders to make the entire composition or blending prealloyed powders that are close in chemistry as a refinement to meet a requirement for a specification. Blending for chemistry reasons is not considered a high-quality process and would not be utilized for technically demanding applications. After blending, powder is ready for loading into containers for HIP.

**Container Manufacture**

**Container Design.** There are a number of methods used to make both simple and complex shapes by the HIP P/M technology. The HIP component designer usually works with the end user to establish dimensional parameters that will meet the requirements of the application and still be compatible with the capability of the HIP process. The designer of the HIP container considers all the factors and then makes his decision on container type and material to give the best chance of successfully meeting the needs of the user. Figure 7 (Ref 20) displays a typical algorithm that designers of cylindrical HIP cans will employ. There may also be some consideration for metallurgical factors as well. As an example, section size of a part may be critical for cooling from heat treatment. These factors must be included in the part/container design process.
Fig. 7 Container design methodology for cylindrical shapes. Source: Ref 20
The basic requirements of the container are that it must:

- Be strong enough to maintain shape and dimensional control prior to and during processing
- Maintain leak tightness against the low and high pressures (typically up to 100 MPa, or 15 ksi) during evacuation, sealing, and densification
- Remain inert and plastic under applied temperature and pressure conditions
- Not contaminate the powder
- Be compatible with the material to be compacted and exhibit minimal integration with the powder by diffusion processes
- Be readily removable by machining, grinding, chemical dissolution, and so forth.

Currently, metal encapsulation is the most widely used containerization method for both experimental and production scale HIP P/M parts. In most cases, metal containers in the form of sheet, tubing, pipe, and so forth are used. Container components machined from heavy mill products (e.g., plate or bar) is not advised because pinhole piping originating from steel ingot solidification occurs frequently and persists through mill operations. This can result in a defective container that is likely to leak during the HIP cycle. Nickel and copper can be used, but thermodynamic reduction of sheet metal grain boundaries and weld metal oxides can occur in high-pressure argon during processing. Sheet materials commonly used for metal powder encapsulation include 1010, 1018, and 1020 low-carbon steel, extra-low-carbon-killed steel, type 304 austenitic stainless steel, commercially pure titanium, and Ti-6Al-4V alloy. Thicknesses less than 0.75 mm (0.030 in.) are impractical because of limited tensile ductility, whereas appreciable plasticity without through fracture may be required in certain container configurations that stretch or bend during the shrinkage that occurs during HIP. Special tooling (e.g., shear spinning tools) can be necessary to form the desired container shape. Large solid shapes, such as shown in Fig. 8, usually are produced with sheet metal containment. For shapes with internal cavities, sheet metal encapsulation with internal inserts that can be removed by machining or chemical etching is used (Fig. 9). Figure 10 illustrates the magnitude of the shrinkage a cylindrical compact experiences during HIP.

![Fig. 8 Large as-HIP shapes. Source: Ref 21](image-url)
The fill, evacuation, and seal tube attachment to the powder container is designed to prevent can buckling and tearing in the transition area. Thick-walled fill tubes are preferred, typically 19 mm outside diameter by 3 mm (\(\frac{3}{4}\) in. outside diameter by 11 gage) for compacts weighing 5 to 200 kg (11 to 440 lb) and 9.5 mm outside diameter by 2.3 mm (\(\frac{3}{8}\) in. outside diameter by 13 gage) for smaller compacts. A thick-walled tube prevents buckling under the applied HIP pressure and facilitates metal flow during the forge welding crimping step.

A successful metal container weld design is the "matched-lip" style (Fig. 11), which accommodates severe deformation during consolidation and minimizes potential leaks. Applied process pressure creates a self-sealing action and eliminates shear loads on the weld. The small volume of accessible weld metal also permits repair by grinding and rewelding. When the matched-lip design is impractical, suitable fillet and butt welds can be employed so long as the welds are capable of passing radiographic inspection per ASME Section VIII (Ref 20).
Fig. 11 Typical can corner designs. (a) Recommended design. (b) Poor design. Source: Ref 22

Low-carbon steel parts made from plate or bar (free-machining grades are not recommended) may be used for can interior shapes or cavity-forming components. As compact weight increases, container sheet gage should be increased to provide structural support during pre-HIP operations. Usually 14 gage (1.9 mm, or 0.07 in.) sheet is adequate for parts weighing approximately 200 kg (440 lb) and 12 gage (2.7 mm, or 0.1 in.) sheet is suitable for parts weighing approximately 1000 kg (2200 lb).

The presence of "continuous path" oxides in the metal container walls or welds can lead to subsequent leakage during densification and result in a nonusable product. Powder/container metal combinations must be evaluated or tested for detrimental interactions at processing temperatures. Binary phase diagrams can be consulted for potential low-temperature melting reactions. Commercially pure titanium encapsulation of refractory metals (molybdenum, niobium, tantalum, and tungsten) permits processing of these alloy systems between 1400 and 1650 °C (2250 and 3000 °F) without problems. However, any iron-containing capsules can only be used with titanium powders if the HIP temperature is kept below approximately 1000 °C (1830 °F). Solid-state reactions between can and powder include interdiffusion alloying and carbide formation.

In some boron- and carbon-containing materials (e.g., cemented carbide) thermodynamic activity of these elements may approach unity, leading to powder/container system melting points approximated by binary phase diagram eutectics. For example, the iron boron eutectic melts at 1149 °C (2100 °F). The latter case precludes steel canning of tungsten carbide/cobalt powder and HIP above approximately 1100 °C (2010 °F), because tungsten carbide/cobalt contains approximately 6 wt% C.

Glass that softens at the HIP temperature can be used as a container (e.g., Vycor glass for nickel alloys) (Ref 1). Glass containers can be fabricated by glass blowing or casting a slurry of ground glass to the required shape, drying, and then firing the shape. Typically, glass is used for high-temperature HIP cycles (e.g., refractory alloys, ceramics, etc.) where metal containers do not have the required properties. Currently, glass molds are not used extensively in production of HIP P/M components. Glass mold technology is described in detail elsewhere (Ref 23).

Another choice for container material is porous ceramic. The ceramic mold process (Ref 24) can produce solid net shapes with a complex geometry. The ceramic container is produced via the same principles as the investment-casting lost-wax process. A wax pattern is produced either by machining (usually experimental quantities) from wax billets or by wax injection molding in precision-machined tooling. A series of ceramic coatings are applied to the wax patterns and air hardened. The mold is then dewaxed and fired to leave the shape of the desired finished part expanded appropriately to accommodate the HIP shrinkage. Because the ceramic container is porous, it cannot be used directly as a container for HIP. The mold is filled with powder and placed into a secondary container and surrounded with a pressure transmitting medium (e.g., sand) and sealed except for an evacuation stem for outgassing. Once the entire assembly is outgassed, the secondary container stem is crimped and the compact is densified by HIP (Fig. 12). The ceramic mold process is particularly advantageous for production of complex precision shapes with compound curved surfaces and reentrant configurations.
**Design Methodology.** At first glance, the design of the HIP container would seem to be one of isotropic expansion of the finish shape given a particular packing density (Ref 25). However, in practice, it is rarely observed that a photographic reduction of the original shape is obtained (Fig. 10). Factors that can influence the dimensional distortion during HIP are:

- Stiffness of the container
- Thickness and variations in thickness and geometry
- Local variations in powder density
- Types and positions of welds
- Heating and pressurization rates during HIP consolidation
- Formation of densification fronts due to nonisothermal conditions of the powder (Ref 11, 26)

This dimensional anisotropy can be difficult to predict given the complex interrelationships between the properties of the powder and container materials as a function of temperature, density, and part geometry.

The designer of HIP cans starts with an end user drawing of the required part and a bill of material listing specifications to be met. The next step is to design a net or near-net shape to protect the dimensions of the part with considerations of machining allowance, powder/container interaction, bowing (particularly in thin sections), end effects, integral sample material allowance, and other secondary processing as described in Fig. 7 (e.g., nondestructive testing, machinability, ability to straighten, heat treatment, etc.). Once this is established, an aim configuration is determined with material allowances as required.

The container design may include several components fabricated from various techniques (e.g., shear spinning, stamping, hydroforming, etc.) and eventually put together to yield a capsule for loading. Because powder occupies only about 70%
of the available volume inside the container, a considerable amount of shrinkage occurs. The science/art of designing a container must account for the packing density and the symmetry or lack thereof to achieve an acceptable part. During the initial production of a particular component, preproduction trials and/or iterations of the full-size shape may be necessary to determine the shrinkages empirically. However, this iterative approach is frequently costly and time consuming.

Through the years, HIP P/M part manufacturers have employed engineering intuition and previous experience to develop the starting can design. At this time, other approaches are being developed and used, namely, empirical and continuum mechanics/finite element modeling. Some of these are briefly described here.

**Empirical Models.** A large percentage of the HIP P/M compacts produced are either simple or hollow cylinders. An empirical model for these shapes was developed several years ago (Ref 26) by analyzing dimensional data from before and after HIP for a variety of cylindrical shapes. To eliminate the effects of the HIP cycle, alloy systems, and container thickness, the analysis focuses on cylinders made from nickel-base alloys consolidated in similar HIP cycles with a certain can thickness range. Best fit curves were generated for axial and radial shrinkage as a function of aspect ratio (length to diameter) and surface area ratio (area of cylindrical component to area of the lateral ends) as shown in Fig. 13. Based on these data, a computer program was generated to provide either the starting container dimensions to make a finished near-net shape or predict post-HIP dimensions given a specified starting container (Ref 26).

![Image](image.png)

**Fig. 13** Normalized shrinkage on solid cylinders (normalized shrinkage = actual shrinkage/isotropic shrinkage). Source: Ref 20

**Engineering Models.** One promising approach to perform a direct process simulation via the use of computer models is to combine a constitutive model of continuum mechanics equations solved by a finite element computational method. Several approaches ranging from simple plastic to compressible, viscoplastic constitutive models have been investigated as described in the article "Principles and Process Modeling of Higher-Density Consolidation" in this Volume. Even though some of these mathematical models are utilized in production, none has matured into a reliable modeling system for arbitrary geometries and HIP cycles.

**Container Fabrication**

**Tooling and Container Component Fabrication.** Once the design has been established, the metal container is fabricated. This is not a trivial step because the container must be producible in an economical fashion or the finished part cannot be manufactured. The most economical and easily formed container material is low-carbon steel; however, other materials (e.g., stainless steel, nickel alloys, titanium, etc.) can also be used. The process is constrained by existing metalforming techniques (e.g., metal spinning, hydroforming, stamping, hand forming, casting, machining, etc.) with each having its inherent advantages and limitations.

Tooling for the HIP P/M process refers to that which may be used to fabricate the container components. Usually, the quantity of parts to be made dictates the precision and cost committed to the tooling for HIP P/M containers. Large numbers of parts (1000 or more) would employ stamped containers. Anything less than this would be determined on a case-by-case basis. Cost of tooling must be amortized over the quantity of parts produced, so more expensive and more
precise tooling can be cost effective only for sizable production runs. Because HIP P/M is most often used as a near-net shape process with small quantities of parts, container tooling is not made to be as precise as other processes where net shape is important.

**Cleaning.** Contamination of encapsulated powders will result, unless dirt, oxides, metalworking lubricants, and rust preventatives used to fabricate container components are removed. See *Surface Engineering, Volume 5 of the ASM Handbook* for cleaning procedures applicable to various metals. Proper cleaning, storage, and handling procedures immediately prior to any welding operation are necessary to prevent dirt entrapment or contamination on can surfaces.

Powder metallurgy alloys that are particularly sensitive to contamination (titanium, nickel-base alloys, and refractory metals) require controlled humidity and stringent cleanliness for final can preparation, assembly, welding, and filling. Electropolishing of stainless steel container components and nonchlorinated solvent cleaning (usually acetone, methylethylketone, or methanol) of titanium container components represent typical cleaning processes for specialized applications. Carbon sheet metal containers should be supported carefully to prevent distortion during welding. Similar precautions are recommended for the outer sheet metal container used in a ceramic mold process.

**Welding.** A matched-weld-lip container configuration is designed to promote directional plane front solidification with good liquid metal feeding in the solidifying weld metal. Certain oxides (iron, nickel, and copper) can be reduced at high temperature in a high-pressure argon environment. This process may produce leaky containers during HIP if oxides extend through weld metal or container wall materials. Use of stainless steel filler metal for carbon steel container repairs is recommended because chromium oxides essentially are stable under processing conditions up to 1200 °C (2200 °F) in argon. Gas tungsten arc welding of nickel containers with stainless steel filler metal is also advised.

Containers for loose powder are assembled, welded, leak tested, and filled in sequence. Containers with interior spacers (mandrels), powder/solid composites (e.g., clad components), or precompacted and sintered P/M compacts can be filled with at least one cover removed. This procedure results in an extensive assembly weld area that cannot be leak tested in the vacuum mode because of the slow response time of helium through the interior of the filled container. Consequently, careful removal of loose powder from the weld area is necessary and use of precision and reproducible (preferably machine) weld techniques is required to prevent leakage. Leak tightness of HIP containers is a major process consideration.

Electron beam, gas tungsten arc welding, and stick welding are used for final container assembly. Argon dry box and electron beam welding are used for titanium alloy containers because nonoxidizing conditions are required. Gas tungsten arc welding with and without filler is used for carbon steel and stainless steel containers. Carbon steel containers may require a final reduction anneal after welding assembly, and some clad parts may need to be preheated prior to welding because of substrate material considerations or section size differences. Because weld metal is essentially a solidified casting, shrinkage and gas porosity are the fundamental causes of leakage at welds.

**Leak Testing.** Containerized HIP of metal powders can only be achieved successfully with leak-free containers. Location of leaks in a fully assembled container by use of valid leak-testing procedures and subsequent repair are fundamental requirements of HIP P/M technology. Leak detection is based on characteristics of helium and argon flow through small capillaries when compared at 1 atm (0.1 MPa) and 1000 atm (100 MPa) total pressure. Flow characteristics of a cylindrical capillary have been described by Guthrie and Wakerling (Ref 27):

\[
Q = \frac{1}{L} \left[ \frac{C_1 P^2 + C_2 P + C_3 \ln \left( 1 + C_4 P \right)}{P_2} \right]^{P_1}
\]  

(Eq 1)

where \( Q \) is the flow rate, cgs units; \( P_1 \) and \( P_2 \) are the exterior and interior pressure, cgs units; \( C_1, C_2, C_3, \) and \( C_4 \) are constants; and \( L \) is the capillary length, cm. For \( P_2 = 0 \) (evacuated container interior) and \( P_1 \) large:

\[
Q = C_1 \frac{P_2^2}{L} \pi D^4 P_1^2 / 256 \eta L
\]  

(Eq 2)

where \( \eta \) is the gas viscosity and \( D \) is capillary diameter, both in cgs units. This applies strictly in the viscous flow region, when Reynolds number (Re) is <1200:
Re = $\frac{D\nu \rho}{\eta} < 1200$  \hfill (Eq 3)

where $V$ is the gas velocity, cgs units; and $\rho$ is the gas density, cgs units.

Equation 2 indicates the relationship of container design, manufacturing, and leak testing. Leakage flow is proportional to the exterior pressure squared. Whereas leak testing is conducted with pressure differences across the container wall of one to several atmospheres, HIP typically uses 1000 atm (100 MPa). Thus, a leak occurring just below the detectability limit of a selected method permits leakage flow rates $10^6$ times greater during HIP. Consequently, sensitivity of the leak-detection method is of utmost importance.

Capillary length ($L$) can be identified with container wall thickness, and with all other variables being constant, a capillary leaks ten times faster through a 0.25 mm (0.01 in.) wall than a 2.5 mm (0.1 in.) wall. The self-sealing, matched-weld-lip design is advantageous because capillary path length through the weld increases rapidly as weld flanges deform and solid-state bonding occurs. The fourth power dependence of leakage flow rate on capillary diameter indicates the necessity for procedures to eliminate weld porosity.

Leakage flow rates for helium at 1 and 10 atm and for argon at 1000 atm (100 MPa) for a set of capillary leak sizes and $D/L$ parameters that could occur in practice are given in Table 1. This illustrates the major problem in leak testing of HIP containers: the leakage flow rate of argon through a capillary hole at 1000 atm (100 MPa) process pressure is approximately $10^5$ times greater than the flow rate during a 1 atm (0.1 MPa) leak-testing procedure such as use of the helium mass spectrometer in the vacuum mode (i.e., evacuated container and/or atmospheric helium surrounding container exterior). This flow-rate difference defines a requirement for maximum sensitivity of the leak-testing method that is satisfied only by use of the helium mass spectrometer method in the vacuum mode.

### Table 1 Leakage rate as determined by capillary (hole) diameter, gas type, and pressure

| Capillary diameter (D), $\mu$m | Leakage rate, cm$^3$/s, for: |  |
|-------------------------------|------------------------------|  |
|                               | Helium at 0.1 MPa (1 atm)    | Helium at 1 MPa (10 atm) | Argon at 100 MPa (1000 atm) |
| 0.001                         | $3.8 \times 10^{-16}$        | $3.8 \times 10^{-14}$    | $3.3 \times 10^{-11}$      |
| 0.01                          | $3.8 \times 10^{-12}$        | $3.8 \times 10^{-10}$    | $3.3 \times 10^{-7}$       |
| 0.1                           | $3.8 \times 10^{-8}$         | $3.8 \times 10^{-6}$     | $3.3 \times 10^{-3}$       |
| 1.0                           | $3.8 \times 10^{-4}$         | $3.8 \times 10^{-2}$     | $3.3 \times 10^{-1}$       |

Note: Leakage rate is inversely proportional to the capillary length; that is, if the capillary length is twice as long, the leakage rate will be half as much. For example: if $D = 0.1 \mu$m helium pressure = 1 MPa, and $L = 0.3$ cm, then leakage rate = $(3.8 \times 10^{-7}/3)$ cm$^3$/s = $1.3 \times 10^{-4}$ cm$^3$/s. Source: Ref 22

Figure 14 illustrates a typical setup for a HIP container that is connected to a commercial helium mass spectrometer for leak testing in the vacuum mode. The effect of argon in powder compacts is estimated here to emphasize the importance of using only leak-free containers for powder encapsulation. Total leakage (in argon at standard temperature and pressure, assuming constant leak conditions and no appreciable pressure rise inside the container) for 1000 and 10,000 s flow times is given in Table 2. Distribution of this "leaked" argon within compacts of various sizes permits estimates of argon contamination in parts per million (ppm) by weight, as shown in Fig. 15.
Table 2 Total argon leakage flow at standard temperature and pressure

<table>
<thead>
<tr>
<th>Capillary diameter (D), μm</th>
<th>Leakage flow, cm³ at a leakage time of:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1000 s</td>
</tr>
<tr>
<td>0.001</td>
<td>3.8 × 10⁻⁸</td>
</tr>
<tr>
<td>0.01</td>
<td>3.8 × 10⁻⁴</td>
</tr>
<tr>
<td>0.1</td>
<td>3.8 × 10⁻⁰</td>
</tr>
</tbody>
</table>

Note: Leakage flow is inversely proportional to capillary length; that is, if the capillary length is twice as long, the leakage flow will be half as much. For example, if $D = 0.1 \text{ μm}$, argon pressure = 100 MPa (1000 atm), leakage time = 1000 s, and $L = 0.3 \text{ cm}$, then leakage flow = $3.8 \times 10^0/3 \text{ cm}^3 = 1.3 \times 10^0 \text{ cm}^3$. Source: Ref 22

Fig. 14 Leak testing setup. Acceptance criterion: $Q$ (flow rate) < $10^{-9}$ standard cm³/s. (a) Test piece evacuated and hooded with helium atmosphere to determine overall leakage rate. (b) Test piece evacuated; helium jet probe used to locate leak. Source: Ref 22

Fig. 15 Argon contamination level versus total leakage flow for various compact sizes. Source: Ref 22

Contained argon, although compressed during early powder densification stages in the HIP cycle, can limit end-point densification by "pressure balance" within small remaining pores. Regrowth of pores in subsequent heat treating operations, with related adverse effects on properties, can occur at levels as low as 0.1 mL/m³ (0.1 ppm) for tool steels and 1 to 5 mL/m³ (1 to 5 ppm) for superalloys. Leaks representing argon contamination at the 10 to 100 mL/m³ (10 to 100 ppm) level generally prevent full densification, and larger leaks usually result in partial or no HIP densification.

Compact Manufacture
Loading. Filling of powder into the hermetically sealed, preshaped metal container can be performed in air, under inert gas, or under vacuum conditions, with the latter to aid in the removal of adsorbed gases. In some cases, powder is still loaded in open air as it was 25 years ago; however, most processing today is containerized to protect the product and prevent inhalation of the metal powder by operators (Fig. 16). Advanced filling systems have been developed to ensure clean, dry handling of powder for critical aerospace applications. Magnetic particle separation, screening, outgassing, and settling have been incorporated into these systems. In a production operation, there is a need for more sophisticated load stations that are automated to achieve maximum productivity. These are usually enclosed systems capable of operating with inert gas or vacuum conditions inside the container and the system. Some loading facilities are also capable of hot dynamic outgassing during the filling operation. If effective, this type of load station will prevent the need for subsequent outgassing once the compact is filled. Figure 17 illustrates a commercial degassing and capsule filling station.

![Figure 16 Illustration of Modeen loading station](image-url)
The loading process must be performed in a manner to meet the desired packing density of the powder to obtain the proper post-HIP shape. Vibration to settle powder (i.e., packing) is usually employed to get the uniform distribution of powder throughout the inside volume of the container. This is important for good shape definition and for reproducibility among parts of the same configuration. It is also possible to load powder into the container first, and then by use of a large-amplitude low-cycle vibration pack the powder in the compact. This process is sometimes called "thumping." Organic materials such as rubber tubing are not recommended in the powder flow path as they are an obvious contamination source.

Fig. 17 Commercial degassing and capsule filling station

Particular attention should be paid to completion of loading. Figure 18 illustrates recommended and poor container filling practice. An incompletely filled container results in loss of shape control and may result in collapse and tearing of the container under external process pressure. Compacted powder in the fill tube provides integral contiguous material for testing and evaluation of the part.

![Incomplete fill](image)

(a) Incomplete fill

(b) Tube partially filled ensures geometry control and provides integral sample material

Fig. 18 Container filling practices. (a) Poor practice. (b) Recommended practice. Source: Ref 22

Outgassing. The functional requirement of encapsulated powder vacuum/hot outgassing is to remove the atmosphere and water vapor (free and absorbed) from the packed powder bed to prevent formation of particle surface oxide and nitride films, which reduce workability and/or mechanical properties of the subsequent consolidated product. Behavior of powder in a heating/vacuum cycle (at temperatures up to approximately 400 °C, or 750 °F), for the purpose of defining process specifications, can be determined by thermogravimetry, combined with limited range mass spectrometry techniques. Vacuum outgassing does not remove gas entrapped in hollow powder particles originating from inert gas atmosphere atomization operations. Evacuation time for a packed powder bed can be estimated using viscous and molecular flow concepts. Elevated temperature is used to raise gas pressure within a bed and to promote desorption of water vapor. Packed metal powder beds are poor thermal conductors; therefore, an excessively high heating rate and temperature gradient in the compact during outgassing can result in redistribution of gas by chemisorption and reaction in the outer zone before all the gas is pumped out of the bed. This can occur because the center of the bed evolves gas at "low" temperature, which diffuses and reacts with the outer "high"-temperature portion of the bed before it leaves the compact.

The required practical end point for degassing a powder-filled hot isostatic pressing container can be estimated from the residual bed pressure (assuming air composition), which contributes oxygen and nitrogen levels ten times less than the
base level of the powder. Based on powder packing density, temperature, bed pressure, and ideal gas laws, parts per million by weight is given by:

\[
\text{ppm} = 1.32 \frac{P}{(1 - \rho_f/\rho_p)\rho_p' RT} M \quad \text{(Eq 4)}
\]

where \( P \) is bed pressure, \( \mu \text{m Hg} \); \( \rho_f \) is full density of metal, \( g/\text{cm}^3 \); \( \rho_p \) is apparent (tap) density of powder, \( g/\text{cm}^3 \); \( f \) is fractional composition of gas, oxygen = 0.21 (air); \( M \) is molecular weight, \( g/\text{mole} \); \( R \) is gas constant, 82.06 \( \text{cm}^3\text{-atm}/\text{K} \); \( T \) is absolute temperature, K.

For \( T = 600 \text{ K} \), oxygen in air \( (f = 0.2) \rho_f = 8.0 \text{ g/cm}^3 \), and \( \rho_p = 5.2 \text{ g/cm}^3 \) (65% packing density):

\[
\text{ppm}_0 = 1.8 \times 10^{-4} P \quad \text{(Eq 5)}
\]

and for \( \text{ppm}_0 = 1 \):

\[
P \cong 5.5 \times 10^3 \mu \text{m Hg} = 5.5 \text{ mm Hg} \quad \text{(Eq 6)}
\]

The normal oxygen level of commercial superalloy powders ranges from 10 to 50 mL/m\(^3\) (10 to 50 ppm) by weight. Thus, relatively high finishing evacuation pressures are acceptable in some cases. Other alloys, particularly refractory metals, may be more sensitive to residual gas. Carbon steel containers can be through oxidized in an air bake-out furnace with prolonged exposure. Container wall thickness, therefore, should be increased with increasing size and weight. For steel containers, grit-blast descaling is recommended after powder degassing. Stainless steel containers offer greater oxidation resistance during powder degassing and do not require descaling.

Loss of part dimensional control in large compacts (greater than about 25 kg, or 55 lb) also can occur during degassing, because the sheet metal container heats faster than the contained packed powder. New empty space is created inside the can at the bottom, into which powder flows from top areas. An oversize diameter at the bottom and uneven top geometry result from this type of powder movement during can heating without applied pressure. This particular problem also can occur in hot-loading HIP operations.

At the completion of the outgassing cycle, the fill/outgas stem is torch heated to approximately 982 °C (1800 °F) and sealed by use of a crimping tool. This leaves the powder-filled container sealed under vacuum and ready for consolidation by HIP. Loss of powder during evacuation and degassing (after can filling) can be prevented by inserting a stainless steel wool plug or a metal plug and partial crimping (Fig. 19).

![Fig. 19 Insertion of plugs to prevent loss of powder during evacuation. Source: Ref 22](image-url)
Consolidation by HIP. The HIP of powder compacts is performed to consolidate the powder metal by plastic deformation to 100% of theoretical density. Details of the various HIP systems and cycles are discussed in previous sections of this article. In a cold-loading system (most commonly used for near-net-shape work), powder-filled compacts are fixtured and loaded into the autoclave (HIP) vessel. Pressure and temperature are increased at preprogrammed rates until the desired HIP hold parameters are reached. At the completion of the hold, the vessel is depressurized and the furnace is turned off. The fully dense parts are removed from the fixturing and sent to the next step in the process.

The upper size limitation for densification of encapsulated parts is governed primarily by the processing unit uniform temperature working zone diameter and length. Tool steel billets approximately 60 cm (24 in.) in diameter by 300 cm (120 in.) long and larger have been fully densified by HIP. Nickel-base superalloy P/M turbine disks greater than 1 m (3.3 ft) in diameter have been successfully densified. For sheet metal encapsulation of P/M parts weighing more than approximately 20 kg (44 lb), attachment of handling lugs is recommended. For large-diameter parts (greater than 0.5 m, or 1.6 ft, in diameter) and weights greater than 100 kg (220 lb), sheet metal bending stresses due to enclosed powder weight must be considered. Careful consideration must be given to the support of large parts in HIP tooling to prevent distortion during heating prior to complete densification. For small net-shape parts (1 to 1000 g, or 0.03 to 35 oz), particularly with thin sections, tooling that permits separate setting of each part is required.

Production of small (less than 10 kg, or 22 lb) net-shape parts by the HIP P/M process is not generally economical because it is labor intensive and the container fabrication costs are high. This applies particularly to the manufacturing of individual tools from P/M tool steels. Exceptions include experimental parts and manufacture of specialty parts in P/M refractory metals, composites, and precious metals, where metal cost is a controlling factor. Small net-shape parts (less than 0.5 kg or 1 lb) are best manufactured by containerless HIP, particularly for tool materials, provided satisfactory process procedures can be developed.

Postconsolidation Processing. After HIP consolidation, the compact fill stems are removed, and the components are dimensioned. The material in the fill stem is checked for density and microstructure. Failure to meet set criteria for any of these characteristics is cause for rejection of the compact. Many parts are further processed through heat treatment, container removal via chemical dissolution or machining, NDT, and mechanical testing prior to certification and shipment to the end user.

Because the HIP P/M process generally provides a near-net shape, the powder part manufacturer usually supplies a semifinished product. This could include material in any one or a combination of the following conditions:

- As-HIP
- HIP plus heat treated
- Rough machined
- NDT qualified
- A preform for thermomechanical processing
- HIP plus thermomechanically processed

The end users determine in what condition they want to receive their parts and specify all requirements such as dimensions, surface, thermomechanical history, properties, and so forth.

References cited in this section

The ability of HIP to produce near-net shapes has been a primary impetus behind the development of HIP P/M parts. Conventional manufacturing methods for materials with high alloy content have low process yields and typically utilize only 10 to 30% of the material purchased in the final product; the remainder becomes scrap during machining. Hot isostatic pressing to near-net shape improves material utilization significantly during part manufacturing and finish machining. A hot isostatically pressed near-net shape part normally loses only 10 to 20% during final machining. The inability to provide nondestructive inspection of complex near-net-shape parts for certification has somewhat inhibited application of this technology, particularly for turbine engine applications.

**High-Speed Tool Steels.** The development of gas-atomized prealloyed steel powders in the 1960s (Ref 3) led to HIP P/M tool steels. This represented the first production application of HIP for a relatively low-cost material. Hot isostatic pressing improves the microstructure of tool steels by preserving the fine grain size and carbide distribution present in the atomized powder through the consolidation process. Increased homogeneity of the fine carbides throughout the material is an added benefit. Superior tool properties result from the improved microstructure. Shape stability during subsequent heat treatment is superior in HIP material. Grindability, wear resistance, and uniformity of hardness also are improved. Additionally, cutting performance of high-speed tool steels is improved by this processing treatment, due to the increased toughness related to fine austenite grain size. New high-alloy-content steels with enhanced material properties can be produced. High-speed tool steels are generally consolidated in billet form. A HIP high-speed steel compact is shown in Fig. 20.
Nickel-Based Superalloys. Starting with development in the early 1970s, nickel-base superalloys have evolved into one of the best applications for the P/M HIP technology. More than 5000 tons (4545 metric tons) of superalloy components are currently operating in commercial and military aircraft turbine engines. Hot isostatic pressing of forging preforms represents a significant portion of the current production, but there are approximately 100,000 as-HIP parts in service as well. The use of HIP/P/M consolidation for superalloys is economically attractive because of its near-net-shape capabilities. High-alloy-content superalloys can be produced with attractive properties. Superalloys strengthened by a large volume fraction of second-phase $\gamma'$ undergo severe segregation during ingot solidification. Such ingots would be virtually unworkable by conventional hot-working techniques for large-size parts. The division of the melt into small powder particles during atomization eliminates macrosegregation, and microsegregation is reduced because of high cooling rates during particle solidification. Hot isostatic pressing of these powders produces a homogeneous microstructure that improves mechanical properties and hot workability.

Superalloy powders are typically made by inert gas atomization or REP. Care must be taken in processing to avoid the presence of stable nonmetallic compounds on the surface of the powder particles because they can be detrimental to the properties of consolidated products. The article "Powder Metallurgy Superalloys" in this Volume discusses the properties of many nickel-base superalloys made via the HIP P/M process. A comparison of HIP properties with other forms is given in Table 3.

Table 3 Heat treatments, grain size, and tensile properties of René 95 forms

<table>
<thead>
<tr>
<th>Heat treatment/property</th>
<th>Extruded and forged&lt;sup&gt;(a)&lt;/sup&gt;</th>
<th>Hot isostatic pressing&lt;sup&gt;(b)&lt;/sup&gt;</th>
<th>Cast and wrought&lt;sup&gt;(c)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat treatment</td>
<td>1120 °C (2050 °F)/1 h AC + 760 °C (1400 °F)/8 h AC</td>
<td>1120 °C (2050 °F)/1 h AC + 760 °C (1400 °F)/8 AC</td>
<td>1220 °C (2230 °F)/1 h AC + 1120 °C (2050 °F)/8 h AC</td>
</tr>
<tr>
<td>Grain size, μm (mils)</td>
<td>5 (0.2) (ASTM No. 11)</td>
<td>8 (0.3) (ASTM No. 8)</td>
<td>150 (6) (ASTM No. 3-6)</td>
</tr>
<tr>
<td>40 °C (100 °F) tensile properties</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.2% yield strength, MPa (ksi)</td>
<td>1140 (165.4)</td>
<td>1120 (162.4)</td>
<td>940 (136.4)</td>
</tr>
<tr>
<td>Ultimate tensile strength, MPa (ksi)</td>
<td>1560 (226.3)</td>
<td>1560 (226.3)</td>
<td>1210 (175.5)</td>
</tr>
<tr>
<td>Elongation, %</td>
<td>8.6</td>
<td>16.6</td>
<td>8.6</td>
</tr>
<tr>
<td>Reduction in area, %</td>
<td>19.6</td>
<td>19.1</td>
<td>14.3</td>
</tr>
<tr>
<td>650 °C (1200 °F) tensile properties</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.2% yield strength, MPa (ksi)</td>
<td>1140 (165.4)</td>
<td>1100 (159.5)</td>
<td>930 (134.7)</td>
</tr>
<tr>
<td>Ultimate tensile strength, MPa (ksi)</td>
<td>1500 (217.6)</td>
<td>1500 (217.6)</td>
<td>1250 (181.3)</td>
</tr>
<tr>
<td>Elongation, %</td>
<td>12.4</td>
<td>13.8</td>
<td>9.0</td>
</tr>
<tr>
<td>Reduction in area, %</td>
<td>16.2</td>
<td>13.4</td>
<td>13.0</td>
</tr>
</tbody>
</table>

Source: Ref 28, 29

<sup>(a)</sup> AC, air cooled. Processing: -150 mesh powder, extruded at 1070 °C (1900 °F) to a reduction of 7 to 1 in area, isothermally forged at 1100 °C (2012 °F) to 80% height reduction.

<sup>(b)</sup> Processing: -150 mesh powder, HIP processed at 1120 °C (2050 °F) at 100 MPa (15 ksi) for 3 h.

<sup>(c)</sup> Processing: cross-rolled plate, heat treated at 1218 °C (2225 °F) for 1 h.
Heat treatment after HIP can have significant effects on material properties as shown in Table 4. Material response to post-HIP treatment depends on the processing conditions. Near-net-shape parts also may be subject to distortion during post-HIP heat treatment. If complex shapes are required, the ceramic mold process is suitable, particularly for static parts. If a carbon or stainless steel container is used for powder consolidation, a 0.5 mm (0.02 in.) diffusion zone may surround the part. This does not cause a problem in the final part because HIP envelopes usually exceed this dimension. Hot isostatic pressing conditions are alloy dependent. Processing temperatures may be keyed to the $\gamma'$ solvus temperatures for purposes of grain size control in nickel-base superalloys.
### Table 4 Mechanical properties of hot isostatically pressed plus conventionally forged Nimonic alloy AP1

<table>
<thead>
<tr>
<th>Processing temperature</th>
<th>Solution treatment</th>
<th>Size of sample disk</th>
<th>Tensile properties (a)</th>
<th>Stress rupture (b)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>°C</td>
<td>°F</td>
<td>mm</td>
</tr>
<tr>
<td>1150</td>
<td>4 h at 1110 °C (2030 °F), air cool</td>
<td>150</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>1150</td>
<td>4 h at 1080 °C (1980 °F), oil quench</td>
<td>150</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>1150</td>
<td>4 h at 1110 °C (2030 °F), quenched and aged (d)</td>
<td>150</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>1220</td>
<td>4 h at 1110 °C (2030 °F), air cool</td>
<td>150</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>1220</td>
<td>4 h at 1080 °C (1980 °F), oil quench</td>
<td>150</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>1220</td>
<td>4 h at 1110 °C (2030 °F), quenched and aged (d)</td>
<td>150</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>1150</td>
<td>4 h at 1110 °C (2030 °F), air cool</td>
<td>475</td>
<td>19</td>
<td></td>
</tr>
<tr>
<td>1150</td>
<td>4 h at 1080 °C (1980 °F), oil quench</td>
<td>475</td>
<td>19</td>
<td></td>
</tr>
</tbody>
</table>

All material aged 24 h, 650 °C (1200 °F); air cooled; 8 h, 760 °C (1400 °F); air cooled.

(a) 650 °C (1200 °F).
(b) 760 MPa (110 ksi) at 705 °C (1300 °F).
(c) 1080 MPa (157 ksi) at 600 °C (1110 °F).
(d) 50% water-soluble polymeric compound, 50% water.
Oxide-dispersion-strengthened superalloys also can be consolidated by HIP. Prior to processing, alloy powders, additives, and oxide dispersoids are put in a high-attrition ball mill and mechanically alloyed. This ensures fine grain size and uniform oxide distribution throughout the powder. Hot isostatic pressing produces fully dense material with these microstructural features maintained.

**Titanium-Base Alloys.** Powder production for titanium and titanium alloys requires special setups because of the reactivity of titanium. The hydride/dehydride process is the most common way to make titanium powders, but the particles resulting from this process are not spherical and thus do not work well for near-net-shape processing. The early method used to make spherical titanium powder was the REP. This was later supplanted by PREP to reduce contamination. Either of these processes depends on the ability to manufacture bar product of the alloy being made into powder. In the late 1980s, an inert-gas-atomizing technique was developed for titanium and its alloys (Ref 30). By the use of inert atmosphere or vacuum induction skull melting, the titanium alloy is brought to the molten state. The liquid is then poured through a metallic nozzle into a high-pressure gas stream. The metal breaks up and resolidifies as spherical titanium particles. The powder is collected in a cyclone system designed to cool the powder to prevent sintering.

There are any number of applications for titanium and titanium alloy powders. In the late 1970s and through the 1980s, the Air Force Materials Laboratory supported many programs to develop near-net shapes for military uses (Ref 31). For many reasons, this work never resulted in an ongoing production process, even though there is still some experimental work being performed currently. All of the meaningful earlier work was conducted with PREP powder. When the gas-atomized powder became available, it was used for all subsequent activities. At that time, the emphasis changed to applications needing titanium aluminide powders. Because these can be easily made by the skull-melting/gas-atomization process, the bulk of the experimental work is currently being performed in this area. The powders are now being used to manufacture metal-matrix composites and intermetallic-matrix composites. The advantages of these products are their light weight, high strength, oxidation resistance, and creep resistance at high temperatures.

**Cemented Carbides.** Tungsten-carbide/cobalt tools are the premier example of containerless HIP to achieve full density by removing residual porosity. Superior transverse rupture strength results from HIP. The wear performance of cutting tools at high speeds is not significantly improved, however, because this behavior is governed by the hardness of the material rather than by its fracture properties. Low cobalt content (3%) alloys can be produced by HIP to give enough toughness for use in drawing dies.

Fully dense cemented carbide can be finished to give a perfectly smooth surface, which is required for high-quality rolls, dies, mandrels, and extrusion tools. Generally tungsten-carbide/cobalt tool materials are manufactured by CIP and sintering of blended powders, followed by HIP. Typical conditions for HIP are 1290 °C (2350 °F) at 100 MPa (15 ksi) for 1 h. Cemented carbide parts produced using HIP are shown in Fig. 21.

**Refractory Metals.** Consolidation of refractory metals by HIP is a two-step process. Processing these materials to net and near-net shape promotes conservation of these critical resources. Niobium alloy C-103 (Nb-10Hf-1Ti-5Zr) has been successfully hot isostatically pressed using a duplex cycle. Hydride/dehydride and PREP powders are consolidated in a plain carbon steel container filled with powder at 1260 °C (2300 °F) at 100 MPa (15 ksi) for 3 h. The container is then removed in a nitric acid solution and further chemically milled in a nitric-hydrofluoric acid solution to remove the alloy/container interaction layer. The material is finished in a HIP step at 1590 °C (2900 °F) at 100 MPa (15 ksi) for 3 h to a final density in excess of 99% of theoretical. Room-temperature and high-temperature (1650 °C, or 3000 °F) tensile strength and ductility properties compare favorably to wrought alloy properties. The ductile/brittle transition temperature is higher (-18 °C versus 160 °C, or 0 °F versus 320 °F, for standard products) in the HIP material due to increased oxygen content. Gas content of the hydride/dehydride material results in poorer weldability than the PREP powder. Hydrogen embrittlement also occurs in the hydride/dehydride alloy C-103. Vacuum baking at 870 °C (1600 °F) for 2 h eliminates embrittlement, and the alloy will fail in a ductile manner in tensile and Charpy tests.

Near-net shape forward bowls manufactured by consolidation of C-103 in the duplex HIP cycle are shown in Fig. 22. The diameter of the bowls was within 0.13 mm (0.005 in.) of final dimensions. The P/M net shape weighed 0.8 kg (1.8 lb). This, compared with rough forging weighing 1.7 kg (3.8 lb) and a final part weighing 0.6 kg (1.4 lb), illustrates the material savings achieved by HIP to near-net shape.
Included is a provision for parts to be low-temperature HIP to a closed porosity condition, decanned, and re-HIP usually at higher temperatures. This option can be employed when the powder/container integration (melting, alloying, contamination, etc.) is unacceptable at the preferred higher HIP temperature. This technique has been used, for example, for niobium alloys that are initially hot isostatically pressed at 1205 °C (2200 °F) in low-carbon-steel containers, decanned, and re-HIP at 1595 °C (2900 °F) to circumvent an iron-niobium eutectic reaction at 1360 °C (2480 °F).

**Stainless Steels.** One of the most prominent applications of the HIP P/M technology is in the area of stainless steels. Both duplex and austenitic steels have been used extensively as P/M near-net shapes in the oil and gas and petrochemical industries. For example, valve bodies, fittings, and large complex manifolds for piping systems have been successfully produced in a cost-effective manner via HIP processing. Figure 23 (Ref 32) shows some of the typical fittings that have been made from 254 SMO material. Figure 24 (Ref 32) is a valve body that weighs more than 2 tons and was made from an austenitic stainless steel. Large manifolds with integral outlets hot isostatically pressed from a superduplex stainless steel have also been put in service in an offshore oil rig in the North Sea (Ref 32). In addition to the other benefits of a HIP P/M approach, the manifold can be fabricated in far less time and avoid costly welding processes. An analysis of the cost factors showed a greater than 20% savings over a similar manifold produced from fabricated cast and wrought components (Ref 32).

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**References cited in this section**


Not only can HIP be used to consolidate loose powder, it can also be used to create a component of multiple bonded materials. Diffusion bonding by HIP can be performed on solid-to-solid, powder-to-solid, and in some cases, powder-to-powder surfaces. As with powder/metal container combinations, material compatibility must be evaluated to ensure no low-temperature melting reactions occur at the HIP temperature. If this does occur, interlayers can be used to alleviate this problem.

**HIP Diffusion Bonding versus Other Joining Processes.** As stated previously, HIP technology was initially developed as a method to diffusion bond two materials together. Table 5 shows various attributes of joining two materials when comparing diffusion bonding with fusion methods (i.e., welding and brazing). The major advantages of diffusion bonding are no melting of the parent metal and therefore no segregation or cracking problems, very little dimensional distortion, and stronger bonds due to the elimination of a low-melting-point filler.

**Table 5 Diffusion bonding in comparison with other joining processes**

<table>
<thead>
<tr>
<th></th>
<th>Fusion welding</th>
<th>Diffusion bonding</th>
<th>Brazing</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Contacting method</strong></td>
<td>Autogenous fusion, autogenous</td>
<td>Pressure (no fusion)</td>
<td>Contact fusion, contact</td>
</tr>
<tr>
<td></td>
<td>fusion and pressure, pressure</td>
<td></td>
<td>fusion and pressure,</td>
</tr>
<tr>
<td></td>
<td>and autogenous fusion</td>
<td></td>
<td>pressure and contact</td>
</tr>
<tr>
<td><strong>Bonding</strong></td>
<td>Cohesive</td>
<td>Adhesive, diffusion</td>
<td>Cohesive, adhesive</td>
</tr>
<tr>
<td><strong>Heating</strong></td>
<td>Local</td>
<td>Local, total</td>
<td>Local, total</td>
</tr>
<tr>
<td><strong>Temperature</strong></td>
<td>Melting point of parent metal</td>
<td>0.5-0.7 of melting point of</td>
<td>Somewhat above melting</td>
</tr>
<tr>
<td></td>
<td></td>
<td>parent metal</td>
<td>point of braze</td>
</tr>
<tr>
<td><strong>Surface preparation</strong></td>
<td>Less exacting</td>
<td>Careful</td>
<td>Less exacting</td>
</tr>
<tr>
<td><strong>Fit-up</strong></td>
<td>Lenient</td>
<td>Precise</td>
<td>With capillary gap</td>
</tr>
<tr>
<td><strong>Materials</strong></td>
<td>Metals, alloys</td>
<td>Metals, alloys, nonmetals</td>
<td>Metals, alloys, nonmetals</td>
</tr>
<tr>
<td><strong>Joint formation</strong></td>
<td>Gradual</td>
<td>Simultaneous</td>
<td>Simultaneous, gradual</td>
</tr>
<tr>
<td><strong>Edge preparation</strong></td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Joining of dissimilar materials</strong></td>
<td>Limited</td>
<td>Unlimited</td>
<td>Unlimited</td>
</tr>
<tr>
<td><strong>Stepwise conduct of process</strong></td>
<td>Limited</td>
<td>Unlimited</td>
<td>Unlimited</td>
</tr>
<tr>
<td><strong>Susceptibility to solidification cracking</strong></td>
<td>Strong</td>
<td>None</td>
<td>Weak</td>
</tr>
<tr>
<td><strong>Porosity</strong></td>
<td>Shrinkage, blowholes</td>
<td>None</td>
<td>Blowholes, shrinkage,</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>diffusion</td>
</tr>
<tr>
<td><strong>Overlapping with heat treatment</strong></td>
<td>No</td>
<td>Unlimited</td>
<td>Limited</td>
</tr>
<tr>
<td><strong>Warpage</strong></td>
<td>Heavy</td>
<td>None</td>
<td>Light</td>
</tr>
<tr>
<td><strong>Principle types of</strong></td>
<td>Butt, lap</td>
<td>Flat (butt, lap, tapered plug in)</td>
<td>Butt, lap</td>
</tr>
</tbody>
</table>
When HIP diffusion bonding is compared with conventional diffusion bonding, there are several advantages, namely (Ref 1):

- Conventional diffusion bonding uses uniaxially applied pressure, which limits the geometry of the joint. For HIP, complex, shaped surfaces can be bonded together.
- Applied pressure must be low to prevent macroscopic plastic deformation with conventional diffusion bonding. For HIP, the plastic deformation is on the microscopic scale and therefore can be performed at a higher temperature.
- Powder and porous bodies can be simultaneously densified to a substrate with HIP diffusion bonding.

**Encapsulation Methods.** As with consolidating P/M compacts, components for HIP diffusion bonding must be encapsulated to ensure a differential pressure exists to create the driving force for bonding. One method is to simply weld the contact area between the two parts. Another is to seal only the contact area with a container component. Yet another is to encapsulate part or all of the substrate. Figure 25 shows the steps used to HIP diffusion bond a powder material with a solid substrate material (Ref 21).

![Figure 25 HIP diffusion bonding of powder to solid. Source: Ref 21](image-url)
**HIP Parameters.** The choice of HIP parameters is usually based on metallurgical and economic consideration. Diffusion bonding is typically enhanced by increasing temperature and pressure. The temperature will generally be 50 to 70% of the melting point of the lowest-temperature material in the system. The pressure shall be sufficient to close up all pores along the bond line as well as internal pores and pores created by interdiffusional pores (i.e., Kirkendall effect). The time at temperature should be kept to a minimum to decrease cost and potentially avoid any deleterious effects from formation of brittle intermetallics, excessive grain growth, and secondary recrystallization.

**Use of Interlayers.** An interlayer is sometimes used between surfaces to prevent the formation of deleterious brittle compounds and/or alleviate stresses due to thermal expansion mismatch. As described previously, interlayers must be compatible with each material that it contacts. The thickness must be sufficient enough to accommodate cooling stress and not so thick that the bond strength is decreased by the presence of a thick ductile interlayer. A 100 μm thick Ni-Cu-Ni interlayer was successfully used as a carbon diffusion barrier between BG42 tool steel and 17-4 stainless steel and a cobalt-base alloy and 17-4 stainless steel, thus maintaining a martensitic structure up to the interface (Ref 33). Another interlayer application was the use of refractory metal and ceramic interlayers during the fabrication of as-HIP foil of highly alloyed material (e.g., titanium, nickel, and niobium alloys) (Ref 34).

**Applications.** There have been several applications of bimetallic components that have utilized the HIP diffusion bonding process. Examples include:

- Corrosion-resistant alloy 625 clad to the interior of F22 steel (Fig. 26)
- Wear/corrosion resistant alloy (MPL-1) clad to 4140 steel (Ref 21)
- Alloy CPM 9V clad on the exterior of 4140 cylinders (Ref 20)
- Twin extrusion barrel internally clad with CPM 10V against 4140 steel (Ref 21)
- CPM 10V clad to low-carbon steel for segmented screws used inside the plastic extrusion barrel (Fig. 27)

![Fig. 26 Low-alloy steel HIP clad with alloy 625 for corrosion resistance](image)

![Fig. 27 Bimetallic wear-resistant screw segments for the plastic extrusion industry](image)
Future Developments

Applications using HIP technology have evolved from diffusion bonding of dissimilar materials to consolidating encapsulated powder and sealing microporosity in castings. Hot isostatic pressing technology is continuing to grow with diversification into new areas. These areas include equipment improvements, mechanistic modeling of material undergoing HIP, and new applications of HIP.

**Refinements of Batch Processing.** One equipment refinement that is generating interest is "quick cool" or "HIP quenching." After the HIP cycle hold, furnace cooling on a cold-walled vessel can take several hours with cooling rates of about 100 °C to 200 °C/h depending on the vessel and size of the load. By utilizing a flow device (Ref 15) and the introduction of cold gas into the hot gas, the convective cooling is dramatically increased. One portion of the gas is forced to the outside of the thermal barrier for cooling while the other portion is circulating inside. To achieve the desired cooling rate, the proportion of the hot and cold gas can be computer controlled. The major driver of this technological improvement is to increase productivity, which ultimately increases capacity and decreases costs. In addition, there may be metallurgical enhancements of some materials, thus potentially eliminating some downstream processing steps.

**HIP Modeling and Microstructure Prediction.** As described in the article "Principles and Process Modeling of Higher Density Consolidation" in this Volume, there has been much work devoted in the 1990s (Ref 13) to predicting dimensional changes during hip via continuum mechanics/finite element modeling. To predict shrinkage changes, an understanding is needed of the anisotropy of consolidation brought about by the complex interrelationships between the properties of the P/M and container materials as a function of temperature, density, and part geometry. With the development of the constitutive equations for the particles and powder aggregates to predict shrinkage, the underlying mathematics now exist to also predict microstructure of the HIP product (Ref 11, 12, 35). With computational power continually increasing at an affordable rate and material property characterization available from hot triaxial compaction tests (Ref 36), the ability to predict grain size (Ref 37) and other microstructural features (Ref 12, 38) may soon be possible.

**HIP Modeling and Closing Porosity in Spray Formed Billets.** To compete with ring-rolled products, there has been some interest in producing large nickel-base superalloy rings via spray forming followed by HIP (Ref 39, 40). For this process, metal is nitrogen-gas-atomized onto a low-carbon steel substrate to form a partially dense preform (typically, >90%). The resulting microstructure is determined by amount of liquid in the spray before impact and amount of liquid on the top surface of the deposit. As the amount of liquid is increased, an increase in deposit yield is observed (i.e., atomizing into a swamp); however, these slower solidification rates typically lead to a coarser grain size. If a finer grain
size is required, the amount of liquid is decreased, but this typically increases the amount of unusable overspray that cannot be recycled due to increased nitrogen content concerns. Hot isostatic pressing of the preform increases the density to nearly 100% density with some interconnected surface porosity present.

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Hot Isostatic Pressing of Metal Powders

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Extrusion of Metal Powders*

B.L. Ferguson, Deformation Control Technology, Inc.; P.R. Roberts, American Superconductor Corporation

Introduction

EXTRUSION is a relatively recent addition to metalworking as noted in a historical survey of extrusion and the development of the process (Ref 1). Notably, the inventive genius of Alexander Dick and the increasing availability of steels that could withstand higher working temperatures opened the way for the hot extrusion of copper alloys and laid the foundation for modern extrusion. Pearson and Parkins (Ref 1) and Lange and Stenger (Ref 2) have written comprehensively on the history, development, application, and mechanics of extrusion; these are recommended texts that provide an excellent background for understanding the process.

There are two main types of extrusion mechanisms, (a) direct and (b) indirect or inverted, as shown in Fig. 1. In direct extrusion, the ram pushes a workpiece forward through a die, causing a reduction in cross-sectional area of the workpiece. Conversely, in indirect extrusion, the workpiece remains stationary relative to the container and there is no friction between the workpiece and container. Both methods may be used to extrude metal powders, although direct extrusion is more widely practiced.