The Evolution Of
HIP

Commemorating the First Hot and Cold Isostatic Processing Vessels

Designated an International Historic Mechanical Engineering
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The Evolution of HIP

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In 1955 a unique manufacturing technique was born.

Working only with a pressure vessel made from a length of high-pressure tubing, a few scientists at an independent research organization created an innovative bonding and fabrication process that today plays an important role in industry.

By some estimates the process will reach a $6-billion market by the 1990s and be a principal technique for manufacturing high-technology alloys and ceramics. And, it will be used to make complex-shaped parts and other products that previously were impossible to fabricate.

The development originated early in 1955 when the Atomic Energy Commission issued a challenge to researchers at Battelle Memorial Institute's Columbus Laboratories in Columbus, Ohio. The challenge was simple: develop a process to bond components of small Zircaloy-clad pin-type nuclear fuel elements while maintaining strict dimensional control.

Accomplishing the task was difficult. The scientists tried several conventional techniques, with unsatisfactory results.

The persistent researchers–led by Dr. Russell Dayton, Henry Saller, Stan Paprocki, and Edwin Hodge*–then decided to try a novel isostatic diffusion-bonding technique. At elevated temperatures they would apply gas pressure isostatically–applying equal pressure to the material from every side.

FIGURE 1. A replicated demonstration of the first hot-wall gas-pressure bonding system, as performed at Battelle in 1955.

Thus was born the technique first called gas-pressure bonding but now referred to as hot isostatic processing, more familiarly known by its acronym, HIP.

In need of a pressure vessel, the scientists obtained a three-foot-long piece of Type 304 stainless steel tubing. It had an outside diameter of 9/16 of an inch and an inside diameter of only 3/16 of an inch. They plugged one end and welded it closed. The other end was threaded to accept a high-pressure valve. The tube was now a small pressure vessel.

Next, they inserted a sample pin all the way to the closed end, then attached the valve. To the valve they attached a feeder line connected to a helium cylinder.

The experimental system was ready, as shown being simulated in Figure 1.

The researchers pressurized the vessel to approximately 2,000 pounds per square inch and inserted the closed end into a heat-treat furnace at a temperature of about 1,500 degrees Fahrenheit.

Several hot-wall experiments achieved excellent Zircaloy-to-Zircaloy bonds as well as Zircaloy-to-core bonds, with the desired dimensional control over the length of the fuel pin. But the technique took too long to be considered successful. Repeated experiments revealed bonding required from 24 to 36 hours.

With the principle proven, the scientists replaced the tube vessel with larger hot-wall laboratory vessels, conducting similar experiments at up to 5,000 psi. These experiments and discussions with equipment suppliers soon made it evident that hot-wall pressure vessels were limited in size, temperature, and pressure capabilities. Increasing the temperature decreased the strength of the pressure vessel.

This realization brought about the concept and development of the first cold-wall HIP system.

In late 1955. Battelle obtained a Type 410 stainless-steel forged pressure vessel and a 10,000 psi transfer compressor from Autoclave Engineers, Inc. Also acquired were two commercial single-zone

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*This idea was patented under these four inventors' names–U.S. Patent No. 687,842, and Canadian Patent No. 680,160.

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tube-type furnaces. The pressure vessel, shown in Figure 2, had an inside diameter of 9 inches and an inside length of 48 inches. The heating element for the two furnaces was wound on a grooved ceramic tube with lead wires extending from the top and bottom. The tube was insulated from the vessel wall with fire brick held together by three steel rods bolted at the top and bottom to two steel plates.

Later that year technicians began to install and assemble the components of the new system. Difficulties were encountered and the system sat idle until early 1956.

Early that February, Charles Boyer, a mechanical engineer who had been hired to design and supervise the operation and maintenance of equipment in Battelle's Fuel Element Development Division, was introduced to this new technology. He was directed to complete the assembly of the cold-wall pressure bonding system and make it operational.

With the assistance of a technician, James Stone, and encouragement by Stan Paprocki and Edwin Hodge, the first gas-pressure bonding system was assembled, as shown in Figures 3 and 4. The system's electrical power, supplied by a 25-kilowatt rotary transformer and controlled by a potentiometer controller, was fed into the furnace by two porcelain-coated steel electrodes in the upper vessel cover. The temperature was measured by inserting thermocouples into two thermocouple wells, one in the center of the lower cover and the other set off-center in the upper cover. These were both constructed of high-pressure tubing with an outside diameter of 3/8 inch and an inside diameter of 1/8 inch, with coned fittings attached inside the covers.

Having assembled and tested the pressure components of the system, researchers tested one of the two furnaces to 1,300 F in air. The outer furnace container was cold to the touch. Then they placed the furnace into the vessel for a test in helium. After they secured the vessel and made all connections, they pressurized the vessel to 500 psi and turned on the furnace. With each 100 F rise of the furnace temperature, as measured by the lower thermocouple, a thermocouple monitoring the outer wall of the vessel showed an almost equal rise. The pressure was increased to 1,000 psi while continuing to heat. After approximately an hour and a half the temperature of the furnace and outer wall of the vessel were both at 600 F, unsuitable for a successful cold-wall system.

It was realized, for the first time, that operating a furnace in a high-pressure gas atmosphere was going to be an arduous problem. Unable to obtain any outside assistance to overcome this obstacle, Boyer approached the problem on his own. Using slotted half-shell ceramics and coiled chromel wire, he assembled a 24-inch-long, 10-ohm furnace with an inside diameter of 3 inches. This furnace

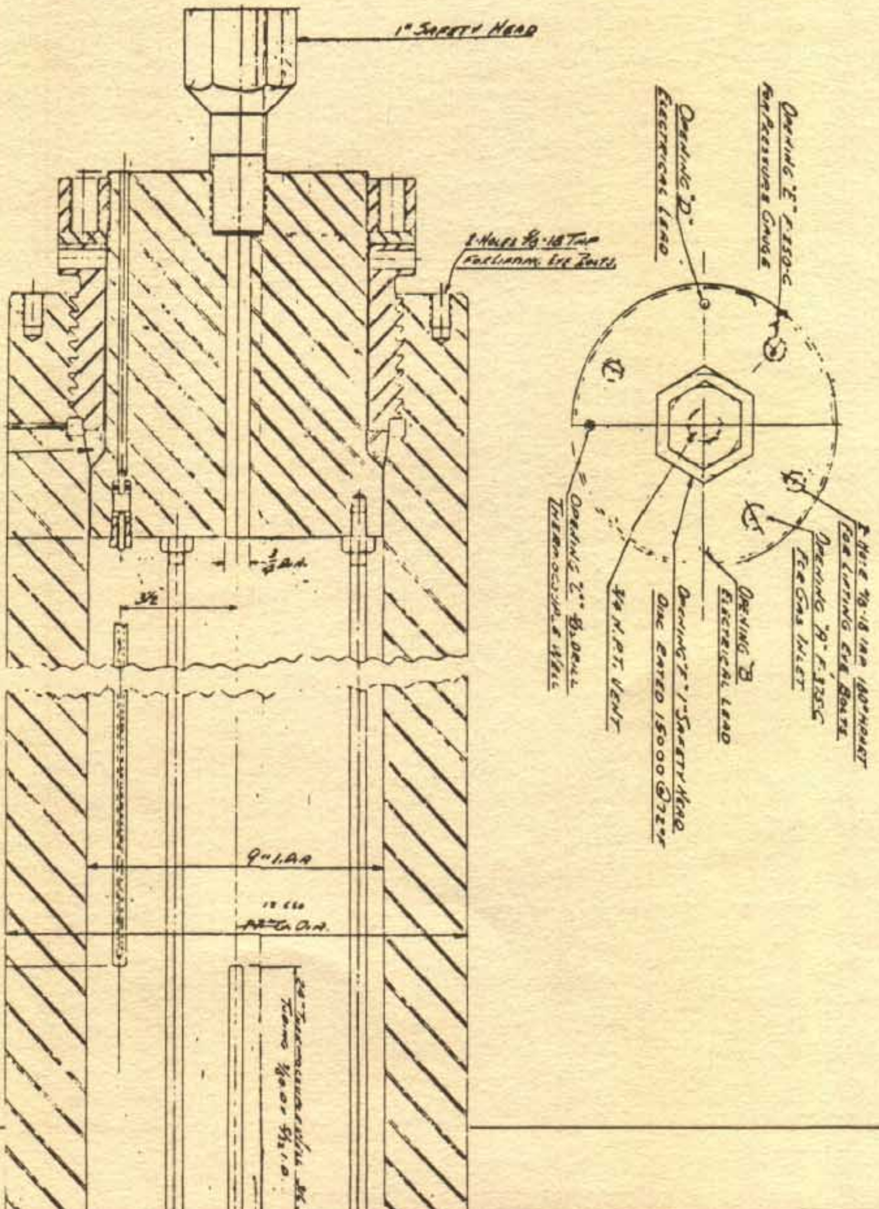

FIGURE 2. Drawing (dated 12/31/54) of the reactor assembly used by Autoclave Engineers, Inc. to manufacture the first cold-wall gas-pressure bonding vessel for Battelle.

was centered inside a stainless-steel can that conformed to the inside dimensions of the pressure vessel. The intervening space between the outside diameter of the ceramic shells and the inside of the stainless-steel can was tightly packed with a fibrous insulation. With this heater, researchers obtained a temperature of 1,525 F at 10,000 psi for a short period of time—with a wall temperature of only 80 F. This was an improvement and a step in the right direction in the development of a workable cold-wall system.

Further improvement in the furnace and system was made, and on April 20, 1956 Boyer and Stone assembled a sample and performed a cycle at the same pressure and temperature conditions on a flat-plate fuel assembly specimen 1-inch square by 6 inches long. After 6 hours at these conditions, the outer vessel wall had reached a temperature of 234 F. Upon removal of the specimen, they found the upper end had melted and the lower end was unbonded. However, a 1-inch-long section in the middle revealed very good Zircaloy bonds, and with the exception of one channel that had shifted, excellent dimensional control was achieved.

With this limited success, project support was obtained to continue Battelle's gas-pressure bonding efforts and for the next 18 months Boyer and Stone improved the system.

During this period, while work continued on flat-plate fuel assemblies, other researchers—Paul Gripshover, Don Carmichael, and Hugh Hanes—began making significant contributions to the development of the gas-pressure bonding process. They launched work on a program to bond Zircaloy-clad flat-plate uranium-dioxide fuel elements. They also began studies to compact magnesium and alumina oxides and to clad delta-phase Zirconium hydride with Types 304 and 347 stainless steel.

In the spring of 1958, Battelle obtained two new and improved 10,000 psi pressure vessels with internal cooling liners. With the acquisition of these new vessels, a new laboratory facility (shown in Figure 5) was designed and constructed at Battelle's West Jefferson Site, 15 miles west of Columbus. An early view of one of the new autoclaves within one of the four pits is shown in Figure 6.

It was at this time that Dean Orcutt joined Boyer to aid with the installation, operation, and improvement of the gas pressure bonding furnaces and system. His contributions in the area of equipment and systems and operations were significant.

To handle larger fuel elements, there was increasing project demand for furnaces with increased size and temperature ranges

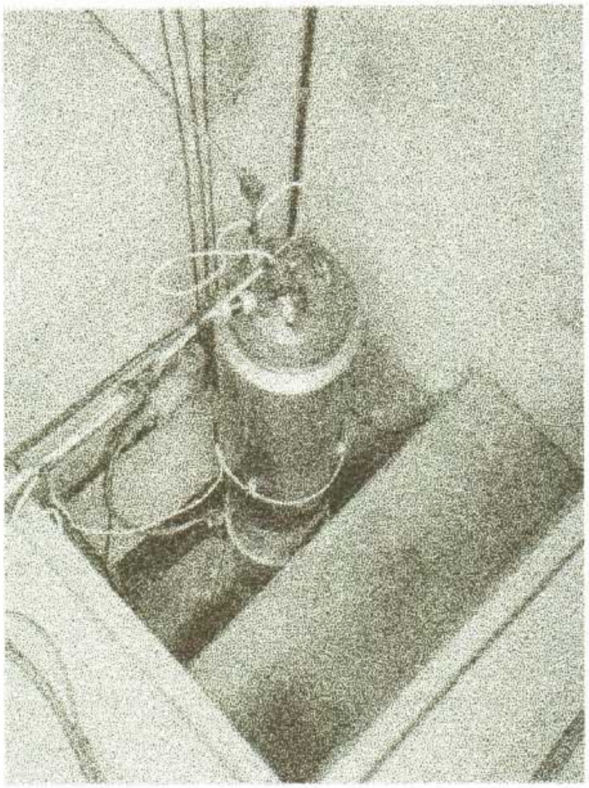

FIGURE 3.
The first cold-wall gas-pressure bonding vessel, as installed at Battelle in 1955.

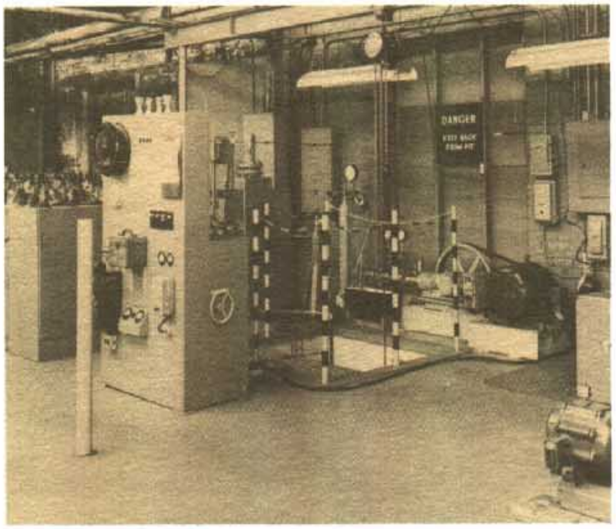

FIGURE 4.
The initial cold-wall pressure bonding system, assembled in 1956.

having reliability and controllable hot zones. In response to these demands, Battelle designed and installed a molybdenum wire-ceramic tube furnace with a hot zone of 8 inches long by 2 inches inside diameter.

During this period, Battelle demonstrated gas-pressure bonding of full-scale flat plate and developed the process for manufacturing Zircaloy-clad flat-plate uranium dioxide fuel elements. The latter brought about the first production use of gas-pressure bonding at Westinghouse for the flat-plate fuel elements for Core 2 of the Shippingport Pressurized Water Reactor.

Led by Edwin Hodge, the Battelle team also conducted considerable research on using gas-pressure bonding for compacting and cladding ceramic, cermet, and dispersion fuel materials. In these studies they demonstrated that by applying pressure they could achieve full density at temperatures significantly lower than those required by sintering, thus improving the properties of most materials.

To continue work on the emerging technology, between 1961 and 1965 Battelle purchased three more autoclaves and associated compressors of varying sizes. Researchers and technicians, as shown in Figure 7, also improved the designs of the ceramic resistance-type furnaces used in autoclaves. They controlled the electrical power for the larger 27-inch inside diameter autoclave furnace by silicon controlled rectifiers. Maximum temperatures in the hot zones ranged from 1,800 to 3,000 F, a capability allowing research on a wide range of materials.

During this time period, Battelle also purchased its first autoclave package system, a unit capable of delivering 50,000 psi. Using the system, researchers performed cycles on new graphite materials and composites on a fairly routine basis at 5,000 F and 20,000 psi for periods of up 2 hours, in a hot zone of 2-½ inches diameter by 4 inches long. It was later replaced with a 3,000 F furnace having a larger hot zone. This work was conducted to determine whether operations at high pressures would allow lowering the temperature in the hope of improving properties. For some materials this proved to be the case.

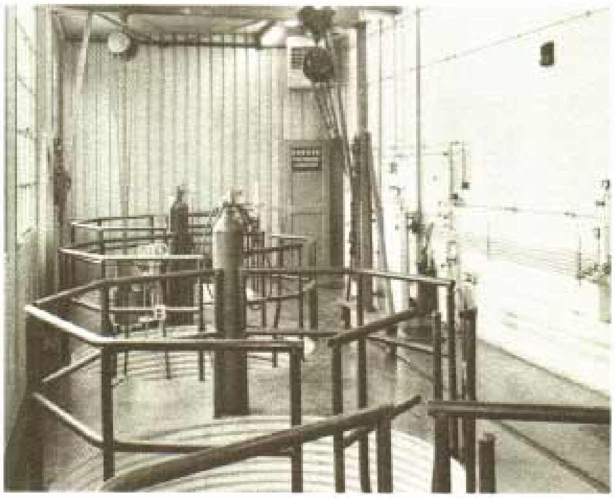

FIGURE 5.
View of Battelle's new gas-pressure bonding pit bay in 1958.

FIGURE 6.
View of a 9-inch inside diameter cold-wall autoclave with an interior cooling liner.

Helium gas was still being used for pressurization, though other gases were used for experimental cycles. Experience indicated that helium presented less severe control problems with the hot zone than did argon. Heat-flow studies were undertaken in a water analog system to better understand the flow patterns in a pressurized gas system.

From these studies, researchers concluded that convective flows within the furnace and vessel had to be stopped or minimized if an acceptable hot zone was to be achieved. They developed and built an inverted-container, three-zone furnace using a multiplicity of molybdenum and stainless-steel cans and molybdenum wire wrapped on a high-purity alumina tube. With this improvement they were able to stop the tornado-like convection currents within the vessel and decrease the loss of heat to the outside vessel wall. They were able to use this furnace for long periods of time at 3,000 F and 30,000 psi with a hot zone of 16 inches. This was three times longer than any previous hot zone and turned out to be a major breakthrough in bringing HIP to commercial realization.

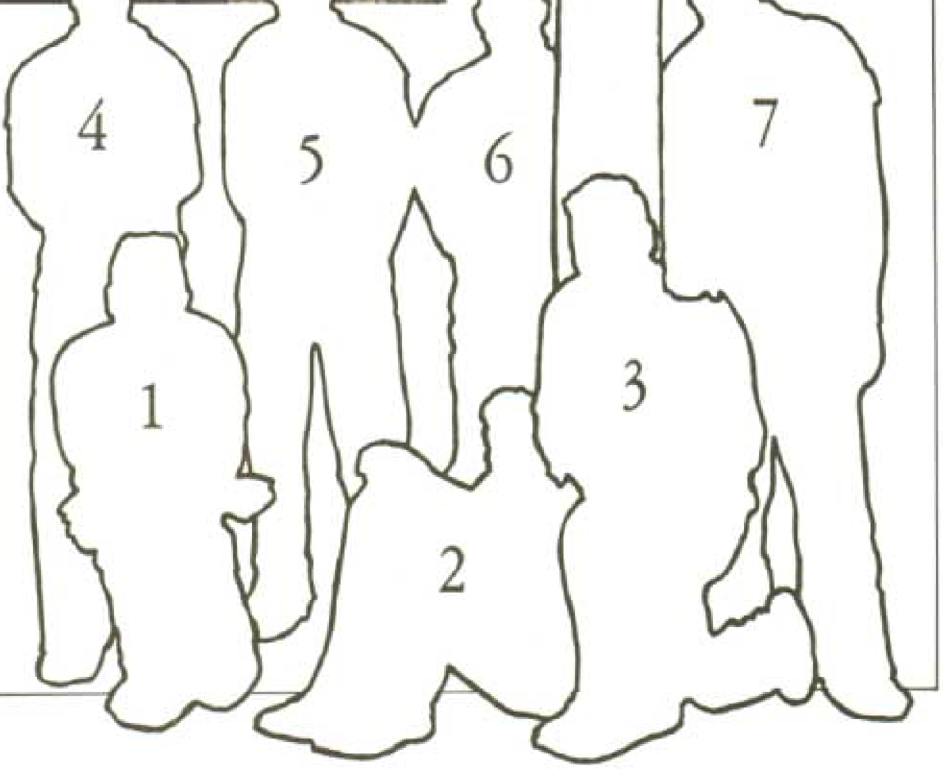

FIGURE 7.
Battelle's HIP technicians in the spring of 1963 are, left to right, in the front row, (1) Dean Orcutt, (2) Keith Newton, (3) Robert Palmer, and in the back row, (4) Robert Shaw, (5) Amos Tapp, (6) Richard Yates, and (7) Nathan Guttman.

Also from 1961 to 1965 researchers at Battelle and elsewhere began to realize the commercial uses for gas-pressure bonding. The developmental studies for nuclear applications began to fade and in their place came the commercial development of hot-isostatic compaction of powders. These powders were used, for example, in tool steel for machine tools, super alloys for jet engines, and powdered beryllium and tungsten for space applications.

Between 1965 and 1970 gas-pressure bonding took on a new name—Hot Isostatic Processing, and Battelle researchers Gripshover and Hanes coined the term HIP. HIP began to be recognized as a viable method to process materials in commercial applications.

Also about this time Battelle acquired two additional vessels to increase its HIP capabilities. One had a 20-inch by 108-inch working area for operations up to 30,000 psi. The other was 3 by 24 inches for use up to 150,000 psi. It contained a 1-inch-diameter furnace with a 3-inch-long hot zone at 2,300 F. This unit was an advanced system obtained for high-pressure research, primarily in ceramics, to determine the practical limits of using high pressures in the HIP process.

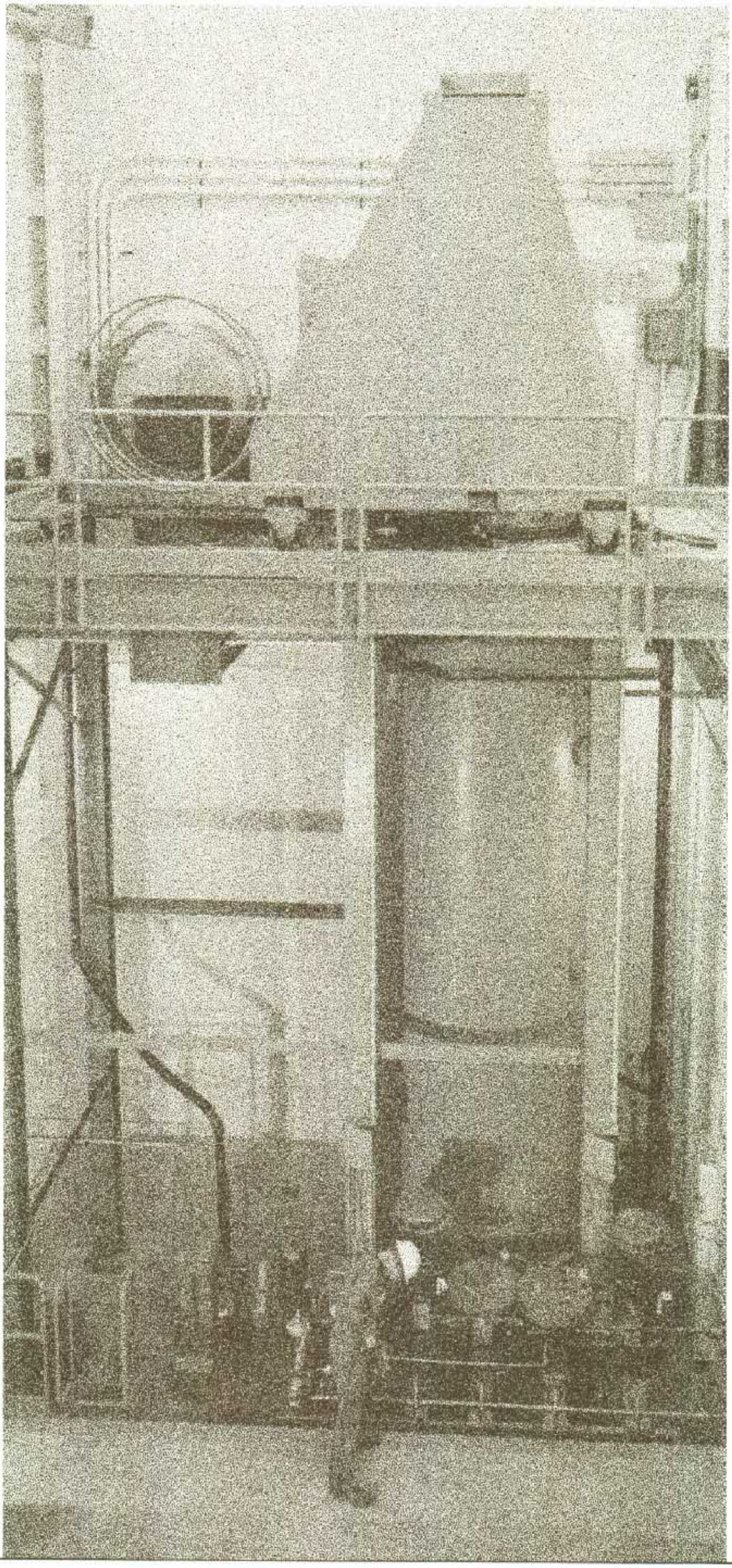

FIGURE 8. Battelle's large HIP system in the early 1970s.

Minor problems with each of these systems were encountered—as expected—and were overcome. With the larger unit, for example, a problem was to develop a way to place several thermocouples within the vessel. Multi-wire lead-throughs were unavailable at the time for high-pressure work. So a Battelle team solved the problem by placing a 24-point rotary switch inside the vessel, synchronized with an exterior 24-point recorder. To obtain up to 24 thermocouple readouts required only five electrical lead-throughs.

Also during this period high priority was placed on developing more reliable furnaces with greater size capability. Furnaces were constructed at Battelle that used ceramic insulator units and molybdenum elements to obtain temperatures of 2,800 to 3,000 F, with hot zones of 10 inches in diameter by 36 inches long. Battelle also designed and patented a spool-type furnace using the inverted-container concept and metallic construction, eliminating dependence on a ceramic muffle support. This 18- by 64-inch furnace created a controllable and reliable 48-inch hot zone that occupied a larger portion of the vessel capacity and operated at 1,900 F and 15,000 psi.

Battelle also developed and put into practice several other innovations. These include:

- A method to outgas specimen containers during pressure compaction
- A method to hot-isostatically form sheet materials

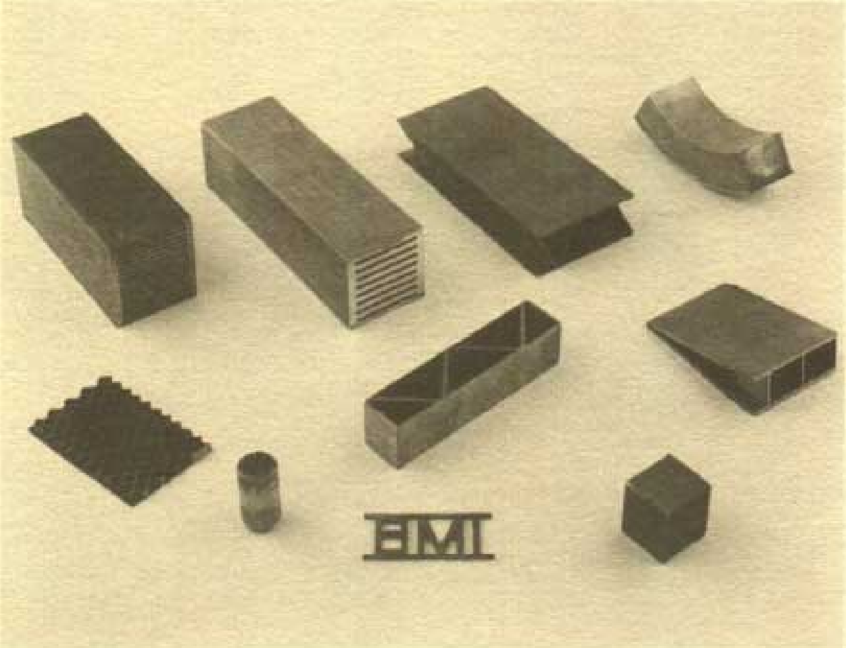

FIGURE 9. Examples of work done with HIP bonding.

- A system for rapid through-put
- A method for high-pressure melting and casting with the HIP unit
- A device to hot-load and unload an idling HIP furnace through the upper vessel opening
- A plug-in furnace concept, allowing furnaces to be inserted into or removed from vessels with no manual connection required
- A production application concept of multiple furnaces for a single HIP vessel, allowing one furnace to be unloaded and reloaded while the second furnace was being HIP cycled
- A concept for bottom loading modular inverted-container design furnaces
- An isostatic pressure-transmitting apparatus for a 30,000 psi HIP vessel that allowed hydrostatic compaction without contamination and quick turnaround between hot isostatic and hydrostatic processes
- Feasibility concepts for much larger hot isostatic pressing than had been considered previously.

It has been said that the pioneering days of HIP equipment, system, and processes at Battelle were through the years from 1955 to 1970. Many Battelle researchers, as listed on Page 9, had roles in this development.

During 1965 to 1970 manufacturers began to offer complete package systems and several laboratory and commercial units were placed into operation. The HIP process began to receive recognition and its possible utilization for many applications was realized.

The development of both the hot isostatic processing system and process from 1970 to the present time has been well documented in the open literature.

With the systems proven in operation, the way was opened for broadened applications of hot isostatic pressing. These applications involved processing new materials as well as using HIP to perform new tasks. Some of the new tasks included healing defects in castings, compaction of prealloyed metal powders for tool steels, powder consolidation, rejuvenation of fatigue-damaged parts, near-net-shape processing, ceramics fabrication, and diffusion bonding of various metals.

In early 1970 Battelle acquired the world's largest pressure vessel, as shown in Figure 8. It had inside dimensions of 5 feet diameter and 14-½ feet long and was designed for 15,000 psi operation. The system was designed and installed by Battelle researchers with a furnace having a hot-zone cavity measuring 4 feet diameter by 10 feet long. After more than 200 successful cycles this system was transferred to a manufacturing firm and is now in commercial operation.

Today, scaled-up HIP vessels such as the 5-foot-diameter model originally developed at Battelle are being used commercially worldwide to perform six distinct processes:

- Powder consolidation (called hot isostatic pressing, hot isostatic compaction, or isostatic hot pressing), which is particularly helpful in fabricating parts with complex shapes and improving their properties at a lower cost

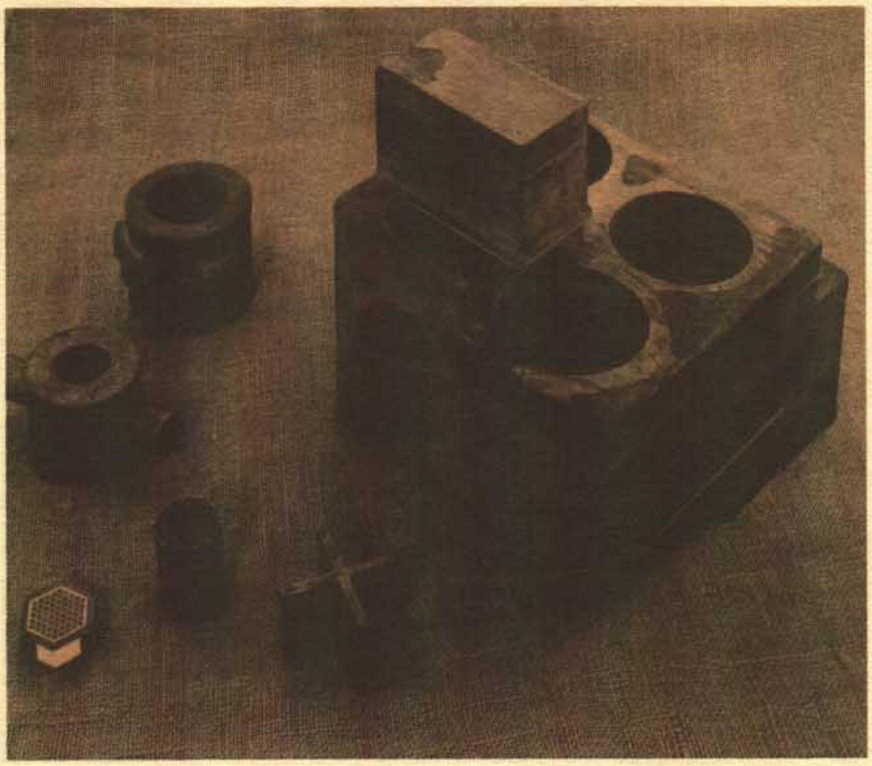

FIGURE 10. Examples of parts formed through HIP powder compaction.

- Diffusion bonding (called pressure bonding, isostatic diffusion bonding, or HIP welding), used for fabricating complex nuclear elements and complex shapes from wrought materials that cannot be fabricated by conventional means
- Densification of cemented carbides, to improve the properties of tool bits and remove flaws from steel-making rollers
- Healing defects in castings to improve their properties and enhance their resistance to fatigue
- Healing creep damage in used parts—called HIP rejuvenation, used in extending the life of turbine blades in jet engines
- Pressure infiltration of molten materials into porous solids, to obtain the combined properties of the two materials.

There are now more than 300 research and production HIP systems in the United States being used by some 60 companies, and likely as many throughout the rest of the world. Meanwhile, process development work continues at Battelle and other facilities.

This unique process today is recognized as an important manufacturing technique, and its applications continue to expand rapidly.

Battelle Researchers Who Played a Role in the Development of HIP

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The hot isostatic processing (HIP) vessels are the 18th International Historic Mechanical Engineering Landmark to be designated since the program began in 1973. Since then, 75 National and 8 Regional Landmarks as well have been recognized by the American Society of Mechanical Engineers. Each represents a progressive step in the evolution of mechanical engineering and each reflects an influence on society.

The Landmarks program illuminates our technological heritage and serves to encourage the preservation of the physical remains of historically important works. It provides an annotated roster for engineers, students, educators, historians, and travelers, and helps establish persistent reminders of where we have been, where we are, and where we are going along the divergent paths of discovery.