HIP Technology Enables Ceramic Manufacturers to Control Material Properties and Increase Productivity

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Abstract. Hot isostatic pressing (HIP) has been known for more than 50 years, and is considered today as being a standard production route for many applications. The HIP process applies high pressure (50-200 MPa) and high temperature (300-2,500°C) to the exterior surface of parts via an inert gas (e.g., argon or nitrogen). The elevated temperature and pressure cause sub-surface voids to be eliminated through a combination of mechanical deformation, plastic flow and diffusion. The challenge is to reach the highest possible theoretical density while maintaining productivity goals.

Uniform rapid cooling is a process by which thin-walled pre-stressed wire-wound HIP units increase productivity up to 70% compared with natural cooling, and increase the density to ~ 100% of theoretical density for many alloys. The added cost to reach this density is around $0.20/kg for a large production HIP system, depending on the material.

HIP introduction

HIP technology was introduced in the early 1950s and has since gained interest for many applications. HIP is a fabrication process for the densification of castings, consolidation of powder metals (as in in metal injection molding or tool steels and high-speed steels), compaction of ceramics for dental and medical parts, additive manufacturing (3-D printing), and many more applications.

Today, about 50% of all HIP units are used for the consolidation and heat treatment of castings. Typical alloys include Ti-6Al-4V, TiAl, aluminum, stainless steel, nickel-based super alloys, precious metals like gold and platinum, and heavy alloys and refractories like molybdenum and tungsten. More applications will likely come rapidly due to the increased interest in the additive manufacturing of ceramics for aerospace and automotive applications.

The pressure applied in a HIP is generally between 100 and 200 MPa using pure argon gas. However, both lower and higher pressure can be used for some special applications. Other gases like nitrogen and helium are also used, while gases like hydrogen and carbon dioxide are more seldom put into use in production units. Combinations of these gases can also be used. The application determines which gas is used for which purpose, especially since helium is quite expensive compared with argon/nitrogen and hydrogen in incorrect concentrations is very explosive.

The parts to be HIPed are initially heated either at elevated pressure or in vacuum. Introducing the gas early in the process, and while heating, causes it to expand and help to build up the pressure in the HIP furnace more effectively. The material composition and suggested HIP cycle govern the startup procedure. The three main advantages of HIPing include:

Increase in Density
- Elimination of internal porosity for defect healing.
- Predictive lifetime
- Lighter and/or low-weight designs

Improvement of Mechanical Properties [1, 2, 3, 4]
- Fatigue life increased up to 10 times, depending on the alloy system
- Longer lifetime of HIPed parts
- Decrease in variation of properties
- Increase in ductility and toughness
- Form metallurgical bond between dissimilar materials (diffusion bonding)

More Efficient Production
- Decreased scrap/loss
- Less or no non-destructive testing (NDT)
- Freedom to choose casting methods for optimal productivity

Uniform Rapid Cooling (URC®) and Quenching (URQ®)

The demand from industry has always been to have shorter HIP cycle times and thereby increase the productivity for the HIP unit to achieve better payback for the investment. The HIP pressure vessel itself plays an important role in increasing the cooling rates for the efficient removal of the heat generated in the furnace. The wire-wound technology shown in Fig. 2 provides several advantages to enhance this heat removal.

The heat removal is achieved by the thin-walled pre-stressed wire-wound cylinder; the wire-winding and an internally water-cooled liner produce an effective heat exchanger between gas-gas and gas-water. Without this thin-wall solution, the cooling rates would be significantly lower and true rapid cooling could not be achieved. An extra advantage with rapid and uniform cooling is that the wear life of the furnace is increased dramatically, thereby dramatically lowering the costs associated with maintenance and spare/wear parts.

The gas cooling rates can be over 3,000°C/min, which is called uniform rapid quenching. The pressure is controlled and maintained through the full cooling time. After cooling, the temperature can increase again to heat treat the material and enable optimal grain growth before the final part cooling is done. Pressure is maintained during the whole cycle, which aids in achieving optimal grain size [5].

With this procedure, heat treatment becomes very advantageous in terms of softening, annealing and even tempering. The outcome is a better-quality material quality that results in reduced costs by reducing the scrap rate and shorter lead times due to less re-work. In addition, parts can be heat treated in the same furnace, thereby eliminating separate handling and additional steps for heat treatment (e.g., heating and subsequent quenching in water, oil or salt baths). This lowers the total capital investment, as well as the running costs.

Material Properties

In many metal alloy systems, avoiding detrimental phases, such as the sigma phase in stainless steel or phase transformation between α- and β-phase in titanium, is crucial. By rapidly cooling the parts down into the safe region of the phase diagram, no detectable levels of these phases can be measured. Without rapid cooling, an increased level of mixed phases will negatively affect the

material properties, grain growth and formation of oxides, carbides, and nitrides at the grain boundary [6].

Since ceramics are more fragile and far less ductile than metals, see Fig. 1, the uniform rapid cooling function is all about temperature control. For a ceramic part (e.g., nitrides, carbides, borides, spinels, etc.), having control of the cooling rate and the cooling curve is equally or more important than speed. The brittleness of the ceramic part makes it essential to make time for the cracking and re-stacking of ceramic powders in a Cold Isostatic Press (CIP) to move below 40% porosity. Sufficient time in the HIP is also necessary to form necking and pore closure, so the density can increase to close of the theoretical density (above 99% of theoretical density).
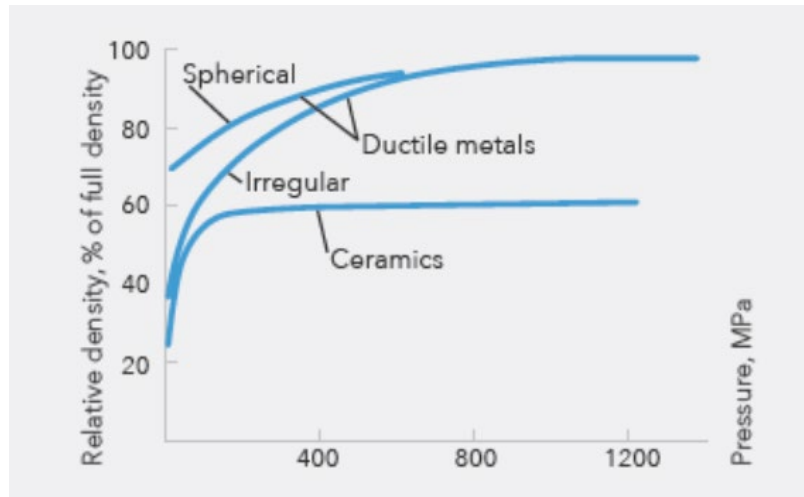

Fig. 1. Density changes in powders.

Zirconia for Dental Implants [7]

Zirconium ($ZrSiO_4$) belongs to the mineral group of silicates and was discovered in 1789 by the German chemist M. H. Klaproth. Zirconium dioxide (zirconia ZrO_2) is a natural compound of the element zirconium which occurs in nature. It has been used in dentistry for up to 25 years.

Zirconia has already been used over 40 years for industrial purposes. It is exceptionally durable and 100% biocompatible. For these reasons, it is used increasingly in surgery for ear, finger and hip prostheses. Applications for dentistry are found in zirconia pins, crowns, bridges and implants. The material's natural white base allows individual coloring in prescribed dentin shades. The biotechnical characteristics of zirconia result in high quality crowns, bridges and implants with excellent biocompatibility and aesthetic appearance.

The raw base material to produce zirconium dioxide is the mineral zirconium (ZrSiO4). Zirconium dioxide is produced in a chemical process using additives. Distinctions are made between sinter additives which influence the actual sinter process and characteristics of the final material, and auxiliary additives which facilitate workability. So-called "green blocks" are produced through a certified manufacturing process.

Sinter additives remain in the zirconium oxide, while the auxiliary additives (mainly volatile organic compounds apart from water) are removed from the material before sintering, leaving no residue. The green blocks are stabilized in a pre-sinter process to a degree that allows machining with tungsten milling tools.

Framework milled from the chalky green blocks is enlarged by about 25%. Shrinkage during the final sinter fire at 1500°C results in the frame's final 1:1 size. The sinter process effects a shrinkage of 20%. Through it the frame acquires the final flexural strength and hardness by compaction of the material particles.

This is achieved by temperature-dependent diffusion processes with varying degrees of surface-, grain boundary- and volume diffusion. If the solidifying diffusion process happens too slow, sintering can be carried out under pressure in addition by hot isostatic pressing (HIPing) of the zirconia, see Fig. 2. Chemical composition and processing methods very much determine the quality characteristics of the end product.
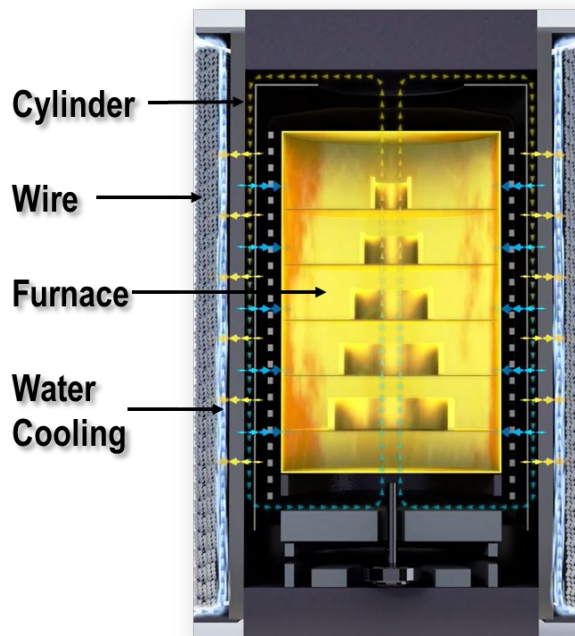

Fig. 2. Illustration of a wire-wound HIP system with parts under operation.

There is a difference between fully stabilized zirconia (FSZ) and partially stabilized zirconia (PSZ). Partial stabilization can be created by adding 3-6% CaO, MgO or Y_2O_3. Depending on production methods, the cubic, tetragonal or monocline modification can be stabilized. Partially stabilized zirconia displays high resistance to temperature changes which makes it suitable for use in an environment subject to high temperatures.

By adding 10-15% CaO and MgO, the cubic makeup of zirconia can be stabilized from absolute zero to fully stabilized zirconia. This makes the material thermally and mechanically resistant to temperatures up to 2600°C.

Due to low heat conductivity and a high thermal expansion coefficient fully stabilized zirconia has a lower resistance to temperature changes than partially stabilized zirconia. A composition of partially stabilized 95% ZrO_2 + 5% Y_2O_3 (5YSZ) presents the ideal material for dental restorations.

Table 1. Typical composition of partially stabilized 5YSZ.

Chemical composition (%)	ZrO_2 (+HfO_2)	Y_2O_3	Al_2O_3	SiO_2	Fe_2O_3	Na_2O	Density (g/cm3)	Hardness (HV)
5YSZ	Balance	4.9-5.2	0.15-0.35	< 0.02	< 0.01	< 0.04	6.02	> 1200

Productivity and Cost

Cosiderably shorter cycle times in a HIP can be achieved with uniform rapid cooling. The obvious target is to increase productivity, which lowers the parts' cost by decreasing processing costs and investment depreciation time. The cycle time can be reduced by as much as 70% (see Fig. 3). For example, for a small- to medium-sized HIP unit, it is possible to run two cycles per 8-hr shift, instead of one cycle per shift (with natural cooling), and have time to achieve heat treatment in the HIP.
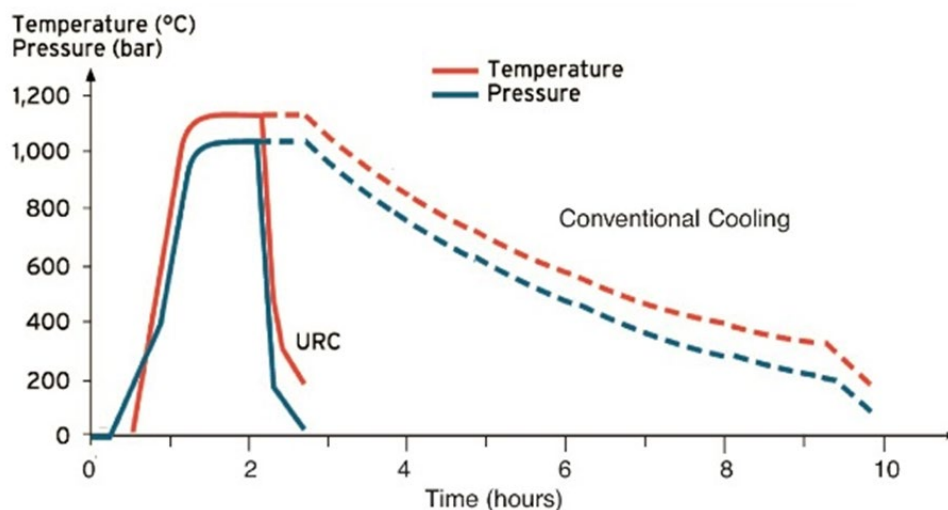

Fig. 3. Typical HIP cycle times without and with uniform rapid cooling.

The operating cost of HIPing has reduced drastically during recent years. This is due not only to uniform rapid cooling, but also to less maintenance-sensitive solutions, particularly of the furnace. For a large production HIP unit used for powder, or consolidation of large forgings and casting, the cost per kg HIPed material is around $0.20. This should be compared with the cost and time spent on NDT, X-rays, weld repairs and higher scrap rates, which to a large extent can be excluded when HIPing is used.

For partially stabilized 5YSZ, the main improvements after HIP is an increased density and drastically improved flexural strength. Typical values can be seen in table 2.

Table 2. Material properties of partially stabilized 5YSZ, after sintering and after HIP.

	Density (g/cm3)	Flexural Strength (MPa)
Sintered	6.02	1250
HIPed	6.07	1725

Summary and Conclusion

HIP technology provides manufacturers with opportunities to control their material properties and increase productivity. Productivity is increased two-fold through the use of uniform rapid cooling. In addition, combining HIP and heat treatment in the same equipment results in shorter lead times, better material properties, and processing and investment cost savings.

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