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Hot Isostatic Pressing technology for dental ceramics

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Abstract: The microstructure of ceramics can be defined as the arrangement of grains (individual crystals) and pores of different size and shape and occasionally of a crystalline or vitreous intergranular phase. This microstructure is generated at the end of the densification process from a powder compact following heat treatment. No microstructure is perfect and homogeneous and it is strongly dependent on powder characteristics and sintering mechanism.

Keywords: *hot isostatic pressing,dental ceramics,fabrication techniques of ceramics.*

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The matter transport to reduce the porosity of a powder compact occurs at high temperature by: (1) diffusion of atoms through the crystal lattice (Dl), along the surface (Ds) or the grain boundaries (Db) in the absence of liquid phase, (2) dissolution-diffusion-precipitation mechanisms in the presence of a liquid phase, or (3) by viscous flow in the case of a large amount of liquid phase. In all cases, there is a competition between the densification phenomenon and grain growth which strongly impacts the final grain size.

As most of the ceramic properties, such as mechanical properties, optical properties or electric conductivity depend on microstructure, it is therefore crucial to control all the steps leading to the microstructural development. Microstructure can be controlled by optimization of each of the fabrication process steps:

(1) Careful choice of raw materials particularly taking into account the chemical purity and particle size and shape

(2) Selection of the appropriate shaping technology with optimized parameters, and (3) Optimization of the temperature–time schedule and furnace atmosphere during sintering (air, O2–Ar, Ar, N2, water).

In addition, the thermal treatment can be modified, for example using:

Pressure assisted methods such as Hot Pressing, HIP, Post-sintering HIP treatments, where the driving force includes an extra parameter related to external pressure, (2) Methods such as Spark Plasma Sintering and Flash Sintering where the application of an electrical field induces increases in densification rates, and (3) Microwaves, a process which involves more homogeneous heat source distribution. [1]

I.Definition:

<u>Hot Isostatic Pressing</u> (HIP) is one of material processing methods, which compresses materials by applying high temperature of several hundreds to 2000 °C and isostatic pressure of several tens to 200MPa at the same time. It was invented in 1955 for diffusion-bonding applications in the nuclear industry and has since found numerous applications in other fields. Argon is the most commonly used pressure medium. Since the applied load (gas pressure) is hydrostatic, deformation of the porous body is supposed to be isotropic. The workpiece is usually encapsulated in an evacuated capsule of **sheet metal,**

ceramic or glass. [2] [3]

II. Difference between HIP and Hot Pressing

HIP applies isostatic pressure to materials using gas pressure, while hot pressing applies only uniaxial pressure. To explain the difference of HIP and hot pressing clearly, suppose that HIP or hot pressing is applied to Material A (metal with pores inside) and Material B (metal with uneven ends).

In case of HIP, Material A, as shown in Figure 1, will contract keeping its initial shape until pores inside disappear, and bond together due to diffusion effects. On the other hand, Material B undergoes no shape change at all because uniform pressure is applied to the uneven edges.

In case of hot pressing, Material B, however, can't keep its initial uneven shape because pressure is applied only to the convex portions. Both Material A and Material B will have different final shapes after hot pressing depending on shapes of a mold and a punch used. Fabrication of large products and moldings under high temperature might be difficult because of ununiformity due to friction force with a mold and constraints due to temperature and dimensions during the deformation. [3]

Compared to hot pressing, HIP can provide material shapes not much different from the initial one after pressure. A material even after changing its shape can keep its initial shape, and will be relatively less restricted by processing of products. By making full use of these features, HIP has been applied in various fields. [3]

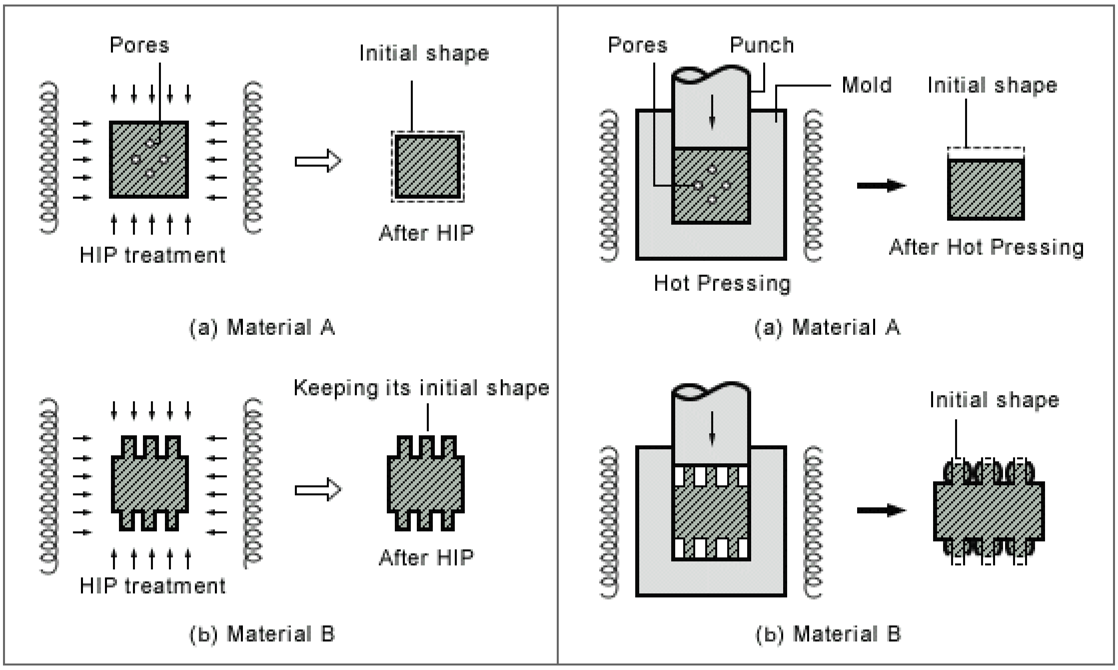

Figure 1: difference between hot pressing and isostatic hot pressing

III. Difference between HIP and Sintering:

Sintering occurs when heat is applied to a powder or to a body containing pores. Grain boundaries and dislocations are also regions of disorder and, therefore, are zones of high energy relative to the perfect crystal in the bulk. All systems try to achieve their minimum energy configuration and, in the limit, for a crystalline material, this is a single crystal containing no defects (pores, grain boundaries, dislocations). [4]

From a thermodynamic standpoint, the first step toward the goal of reduced energy is for pores to be eliminated from the system (the specific surface energy of pores is greater than the grain-boundary energy). However, during any sintering activity, the process of grain-boundary elimination (grain growth) often begins before the process of pore elimination has been completed. This state can then inhibit further pore removal for mechanistic reasons.[4]

While isostatic hot pressing, the combination of pressure and temperature can be used to achieve a particular density at a lower temperature than would be required for sintering alone. The effect of the lower temperature is that unacceptable grain growth can be avoided. In addition, the methods identified previously for enhancing densification of powders by introducing additives such as low-melting-point constituents (which may have deleterious effects on mechanical properties) are not needed.[5]

IV.Advantages of HIP:

HIP removes the impurities (pores) out of materials creating a product with an homogenous microstructure (compact solid) with minimal or no impurities in the material.HIP gives the manufacturer and ultimate user a number of unique benefits:

- Reduced porosity
- Higher Density
- Higher uniform strength in all directions (isotropic properties)
- No segregation or grain growth during manufacture
- Higher yield and tensile strength
- Homogeneous microstructure
- Maximum abrasion resistance
- Near-Net shaped parts
- Shape flexibility: Isostatic pressing makes it practical to produce shapes and dimensions that are difficult or impossible to produce by other methods. [5]

V.Limitations of HIP:

The tooling cost and complexity of the process are higher than for uniaxial hot pressing.

VI.HIP equipment and HIP process:

The HIP process uses the combination of elevated temperatures and high pressure to form, densify, or bond raw materials or preformed components. The application of the pressure is carried out inside a pressure vessel, typically utilizing an inert gas as the pressure-transmitting media. A furnace located inside the vessel is the temperature source. Parts are loaded into the vessel, and pressurization occurs usually simultaneously with the heating. Parts are then cooled inside the vessel and removed.[5]

A hot isostatic pressing system usually consists of five major components: pressure vessel, internal furnace, gas handling, electrical, and auxiliary systems. Hot isostatic pressing systems currently range in size from 1 to 80 in. (25 to 2000 mm) diameter. The **smaller** units usually are used for research. It is common practice to design one unit universally for research processes such as: densification of ceramics at (2,000°C). This can be accomplished with one basic system but with various plug-in furnaces and a versatile control system. Larger size production units are usually designed for handling a specific process but can also accept various plug in furnace types. [5]

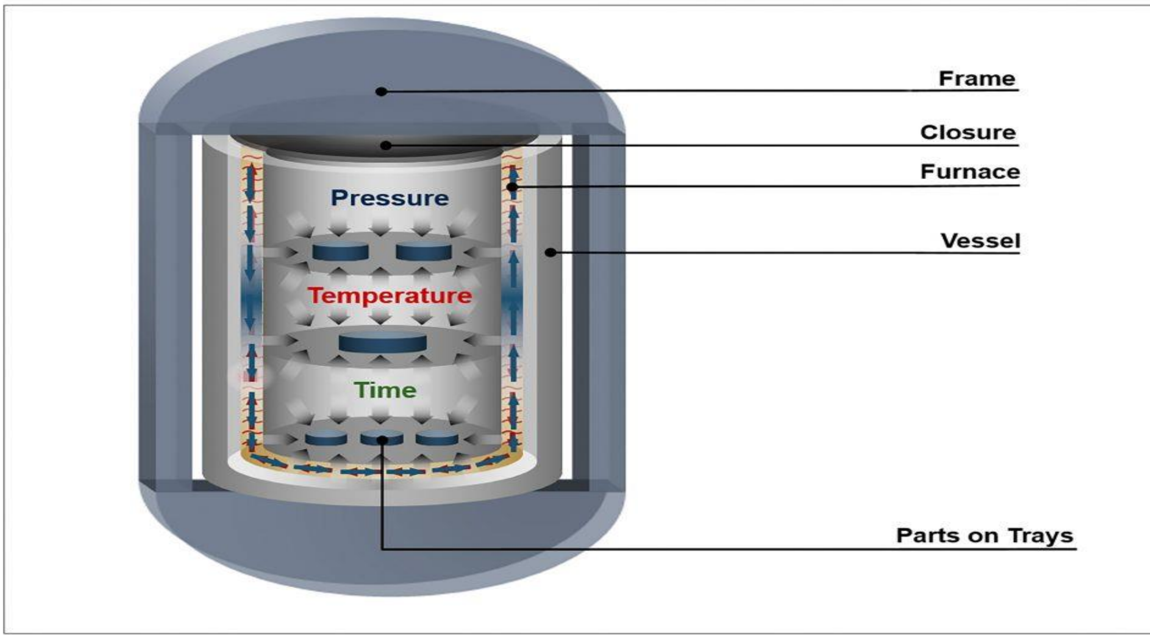

Figure 2: HIP equipment

A.Pressure Vessel

The pressure vessel in a HIP system contains the high-temperature furnace and retains the high-pressure gas. Utility connections to the furnace typically extend through the bottom cover and require a reliable pressure seal as well as electrical isolation from the vessel. Elastomer O-rings seal the gas in the vessel since the vessel temperature is kept below (250°C). The design of any pressure vessel must meet two essential requirements. They are the minimum dimensions based on allowable stress and fatigue life.[5]

B.Furnaces

The hot isostatic pressing furnace, contained within the pressure vessel, provides the heat required either from direct radiation, gas convection, or forced-gas convection. Within the furnace are electrical resistance heating elements and a space for placing the work piece (tooling and components to be HIPed). The pressure vessel is designed as a "cold wall vessel" and is protected by a thermal barrier that prevents hot gas penetration to the inside of the vessel wall. Furnaces can be constructed to "plug-in" with the work piece in place. Direct thermocouple attachment takes place outside the vessel. [5]

A.Radiation furnaces are typically multi-level, multi-zone styles with the heating elements surrounding the work piece. There are two types of radiation furnaces used; the cold load system where the element and work piece start at room temperature and are heated together, or the hot load system where the work piece is preheated outside the vessel then loaded into the hot furnace cavity.

B.Natural convection furnaces are common for many sizes of HIP units. They work by heating the dense gas in the furnace element area and conveying it to the work piece above by the buoyancy of the hot gas molecules. In this type of furnace, a convection liner creates a path for the gas flow as its energy dissipates to the work piece and as more hot gas molecules move upward from the element. This gas circulation continues until the temperature is equalized throughout the work area. [5]

C. Forced convection furnaces are also of single or multi-level construction but have a fan to circulate the gas. Heat transfer to the work piece is a function of the full coefficient of heat transfer of the gas. By increasing the gas velocity the final coefficient is increased to provide high heating/cooling rates.

There are several advantages to using convection furnaces. The work piece is not exposed to direct radiation from heater elements. There is a larger work cavity available for a given vessel diameter. Heating elements are not susceptible to damage by the load/unload process, and construction is much simpler than that for multi-level radiation furnaces.

Many different furnace element materials are available for hot isostatic pressing furnaces. Each element material has characteristics that affect its capability in HIP applications. The three most common element materials are graphite, molybdenum, and nickel/chrome. [5]

C.Gas Handling

Hot isostatic pressing processing requires an inert gas to apply an equal (iso) force to the part for densification. Most systems use argon as the pressurizing medium. HIP systems often require pressures to 15,000 psi and sometimes even up to 45,000 psi (N/mm^2) depending on the material being processed. Gas pressures can be achieved with a compressor and/or by thermal expansion.

Gas purity is very important when processing parts which are susceptible to oxygen, hydrogen, carbon monoxide, carbon dioxide, water vapor, and hydrocarbons contamination. [5]

D.Controls

The control system for a hot isostatic pressing unit is the subsystem that links the vessel/furnace, gas handling, and auxiliaries into a functioning production tool. Computer control ensures repeatability of these parameters to maintain consistency of operation.

E.Auxiliary Systems

A HIP system is supported by a number of important subsystems classified as auxiliaries. These include cooling and vacuum systems, material handling with work piece fixtures and facilities subsystems including exhaust fans, oxygen monitoring equipment, and cranes. [5]

1. The cooling system keeps the pressure vessel temperature below its design limits.

2. The vacuum system provides a means of removing the atmospheric contaminants from the furnace/vessel. Commonly a mechanical pump with a blower is used for handling high flow rates at low pressure. Isolation is provided by a high-capacity high-pressure vacuum valve. System interlocks ensure safety and prevent exposure of the vacuum components to high pressure.

3. Work piece fixturing is determined by the material being processed as well as by the operating temperature. Carbon steel has a relatively high melting temperature and is used.

Materials such as nickel/chrome alloys or a combination ceramic and graphite fixture can be used also for work piece fixtures. [5]

4. Oxygen monitoring equipment are essential for the safety of personnel. The monitoring equipment can be set up to automatically start exhaust fans and give warnings of an oxygen deficiency.

VII. Applications of hot isostatic pressing:

HIP is widely used during the manufacturing of high integrity and precise components for a diverse range of applications and industries from Aerospace and Medicine to Automotive; Composites, dental Implants, Sintering (Powder metallurgy), Coatings, Ceramic parts, Titanium Castings and diffusion bonding.

VIII. Effect of hot isostatic pressing on ceramics properties:

A. The influence of HIP conditions on densification, mechanical properties and biocompatibility of zirconia ceramics was investigated. Compressive strength increased considerably after HIP treatment, from approx.101 MPa (for pre-sintered samples) to at least approx. 602 MPa. All samples presented high Young's modulus values between 1739 MPa and 4372 MPa. Dental zirconia ceramics have proved a good biocompatibility. The results reported in this work showed that ZrO_2–CaO dental ceramics manufactured by hot isostatic pressing presented high density and compressive strength, elastic behavior and a good biocompatibility with human cells. [6]

B. High transparency: vacuum sintering to closed porosity with a subsequent HIP'ing step provides an alternate processing route to fully dense Lu2O3:Eu. It has been shown that evolution of porosity and microstructure during sintering and densification must be optimized to achieve fully dense transparent ceramics. Over-sintering led to rapid grain growth at high temperatures and the grain boundary motion was faster than pore mobility, resulting in entrapped pores in the interior of grains. During the subsequent HIP stage, closed porosity was achieved while maintaining the minimal grain size and the maximum grain boundary area during the vacuum sintering step. This enabled the pore mobility to be fast enough to keep up with the grain boundary motion during HIPping, and the pores were completely annihilated forming fully dense Lu2O3:Eu. By this method, highly transparent Lu2O3:Eu was formed.[7] [8]

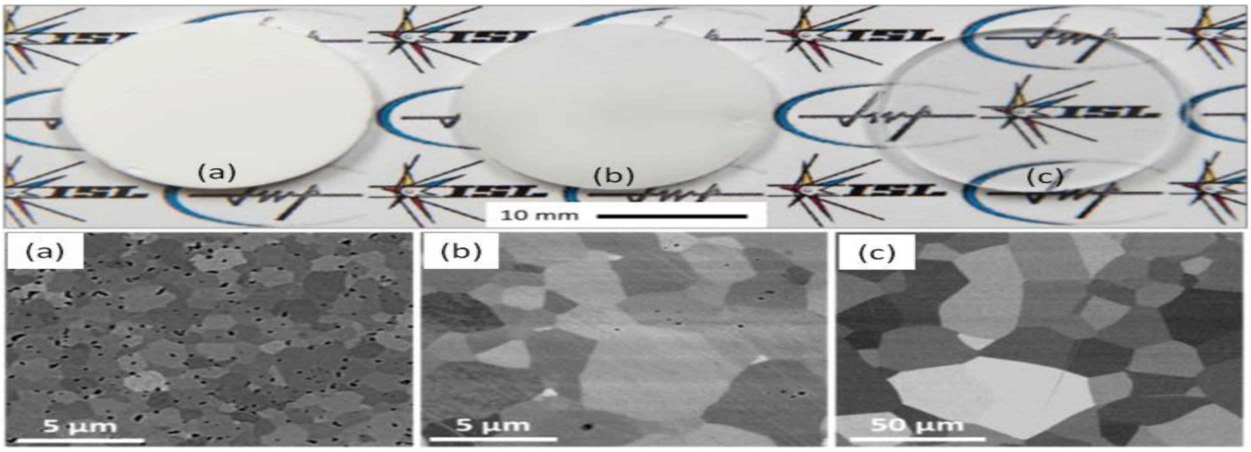

Figure 3: Transparency after hot isostatic pressing

C. High cyclic fatigue: the cyclic fatigue strength (888 MPa) of HIP Y-TZP with sandblasting and acid-etching was more than twice that of Y-TZP as specified in ISO 13356 for surgical implants (320 MPa), indicating the clinical potential of this material.[9]

D. However, HIP nano zirconia exhibited inferior strength, surface polishability and behaviour to loading.[10]

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