# HIP OF CERAMICS - NEW ENGINEERING APPLICATIONS

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## INTRODUCTION

The advantage gained by applying HIP techniques may be even greater for ceramic materials than they are for metals. The reduction in temperature, often several hundred degrees makes, as for metals, a better control of temperature influenced phenomena, e. g. grain growth, possible. In composites, both in particle and in fibber or whisker reinforced composites, is the ability to prevent reactions by lowering the temperature particularly valuable. HIP makes it still possible to reach full densification, also with very high fiber contents.

Elimination of defects in super alloy casting or in large sintered cemented carbide parts is probably the today most widely applied use of HIP for metals. The ability to mend faults introduced in previous processing steps – and (if HIP is used for that operation) to prevent faults from developing during the sintering step – is even more important for ceramics. The strength in tension of a ceramic material is determined by the largest defect in the stressed volume – the strength is extreme-value-oriented rather than mean-value-oriented as in metals – which makes the control of defects particularly important. Further-more the fracture toughness of ceramics is generally small – i.e. the $K_{IC}$ - value is low – which means that the strength determining defect must be kept very small if high strength is to be reached.

Some technologically very important ceramic materials – particularly nitrides – decompose at ambient pressure at far lower temperatures than they melt. In the HIP process the high pressure available can be used to counteract this. One way is to use one (but preferably all) of the species formed upon decomposing as high pressure gas. Nitrogen gas is for this reason used as pressure medium in container less HIPing of silicon nitride. An even more effective and general method is to enclose the powder body in gas impermeable container. The powder body material may also in this case start to decompose. However, the gaseous species formed cannot escape as long as the total pressure inside the container is lower than the HIP pressure, which is generally the case. Partial pressures needed to prevent further decomposition will build up and no further material loss take place. When the pressure in pores of the powder body increases as densification progresses, the equilibrium pressures of the gaseous species formed upon decomposing is exceeded and the solid material will reprecipitate.

Advanced ceramic materials in general are very hard and difficult to machine. It is therefore strongly desired to make the parts to final shape on as large areas of the surface as possible and, if machining cannot be avoided altogether, to limit machining to cylindrical or flat surface. For metals near final shape is often fully acceptable as the necessary removal of the metal container in a machining center can usually be combined with a finishing cut of the powder metallurgy product it self. Furthermore machining of ceramics, which is usually made by some type of diamond tools generally introduces defects in the machined surface, which reduce strength. Fortunately there are some techniques utilizing HIP which allow ceramic parts to be made without finish machining to a product precision similar to investment cast products.

## 1. DIFFERENT HIP TECHNOLOGIES FOR CERAMICS

HIP of ceramics can be carried out in many different ways depending on the requirements of the product. If good densification only is the goal and a minimum of mechanical handling of the powder is required, then the use of a bellows container may be a good alternative. Such a solution was applied for a mixture of oxides, called Synroc D, which was obtained by calcimine radioactive waste. Synroc D is a titan ate-based ceramic waist form obtained by mixing selected additives to an aqueous radioactive slurry which was dried and calcite. The ceramic powder mixture obtained, however, had a vibrated density of only 26% of TD (theoretical density). Any HIP container of conventional design would collapse in an uncontrolled manner with great risk for leaks if direct HIP was attempted of such a low density powder. A type of bellows container, originally developed for radioactive “cladding hulls” (short, thin-walled virally tubes with a fill density of only 13% of TD) was used. 50 kg of simulated Syncron D powder was filled into a large stainless steel container and HIPed to full density (Fig. 1). An essentially cylindrical ceramic block was formed inside the flat-to-flat compressed bellows convolutions. In this case the shrinkage mode can be described as an axial recompression of almost 50% (which gives compacted body such a strength that it takes control of the subsequent shrinkage mode) followed by a three-dimensional (is static) compaction of another 19%. In general, however, a product with both good material properties and a close shape control is required. An overview of alternative routes for the production of shaped, dense ceramic parts is found in fig. 2. HIP is an important parts of all the show alternatives except that indicated by the broken line. The method of HIP chosen has however a great influence on the results that can be achieved, not least for many high temperature ceramic materials.

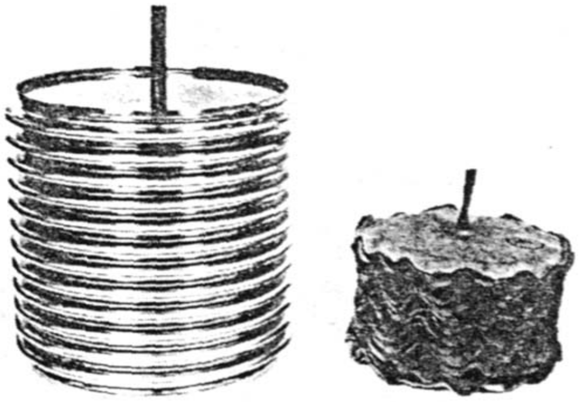

**Figure 1**. Large-bellows container loaded with 50 kg of Synroc D before and after hot isostatic pressing at 1100*c and 150 MPa for 2 h to density > 99% of theoretical. Initial bellow dimensions were 44.5 cm outer diam. by 50 cm high.

The method to the left in fig. 2 is the most common for near net shape manufacture of powder metallurgy parts. Sheet metal or glass containers are used, which have a similar shape as the product to be made but are enlarged to compensate for the fill density to final density relation of the powder.

This HIP method was used for large – alumina canisters in a technical feasibility study of making long term resistant containment for spend nuclear fuel. A low carbon steel container of 3000 mm length and largest diameter of 600 mm used. A fully dense alumina canister with a diameter of 500 mm with a tolerance of 1 to 2 mm was produced. This tolerance is acceptable in this case but could only be reached by the use of an efficient system to uniformly pack the alumina powder into the steel container and because of the relatively simple geometry. It is difficult to meet close tolerance, to find suitable container materials and to control and limit reactions with produced part. These findings have all contributed to make interest for this HIP method very limited for high performance ceramic products.

Another alternative HIP-method starts with sintering of a shaped ceramic powder body to such a density, typically 94 % to 96 % of TD, that the pores cease to be interconnected to the surface of the part. HIP is then carried out with the HIP gas pressure acting directly on the part surface (second right in Fig. 2). The method is called sinter-HIP or "sinter plus HIP".

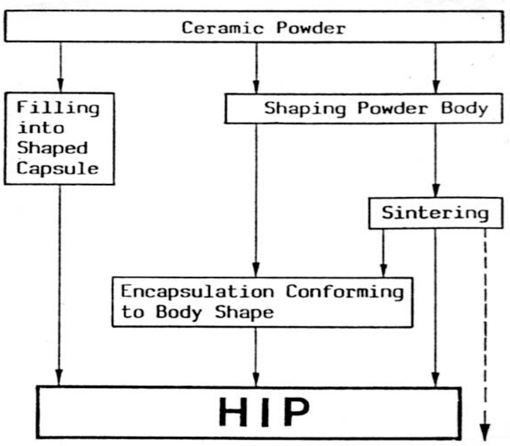

**Figure 2**. Alternative ways to manufacture shaped and dense ceramic parts.

The gas composition is important for many materials. For silicon nitride, for example, nitrogen gas is common which reduces the dissociation of material. The necessary presintering to about 95 per cent density limits the selection of materials that can be processed. For many ceramic materials with high strength at high temperature, however, other limitations occur. In presintering of silicon nitride, for example, elongated - silicon nitride grains are formed. These new grains often form a very rigid, interlocking structure. Usually only 1 % to 1,5 % density increase is obtained in the HIP treatment, regardless of the initial density. Considering the high strength and low creep rates of pure – silicon nitride grains, this may not be surprising. If a sufficiently high volume fraction of boundary phase is present, however, good densification is also positively effected by long processing times, 6 h or more.

In silicon carbide materials the grains formed during the profiteering often resist further densification. If grain growth in the profiteering stage can be restricted and a fine grained material used, good densification during HIP can be obtained.

In the HIP method in the center of Fig. 2 the green powder body is encapsulated before any significant shrinkage of the body occurs. The positive effect of pressure can be fully utilized throughout the sintering and shrinkage stage. In first published realization of this principle for silicon nitride a conformable high silica glass container was used. Improved techniques which are now commencing to find industrial applications use glass powder to form the encapsulation.

This method offers the greatest freedom of all discussed methods to select material composition. Many high temperature ceramic materials can be fully dandified at comparatively low temperatures without any sintering additives. Fiber or whisker reinforced ceramics with high fiber content can also be efficiently produced. If a silicon powder body containing some like yttrium is reaction sintered in nitrogen, encapsulated and HIPed, very good high temperature properties can be obtained. Another advantage of this process (third from right in fig. 2) is that the shrinkage during HIP is lower in encapsulated HIP from powder (but larger than in sinter-HIP).

## 2. APPLICATIONS OF HIPED CERAMIC MATERIALS

HIP has been used as the consolidation method for a very wide range of ceramic materials. The compositions listed in Table 1 represent some of the materials studied but the complete list would of course be much longer.

Most of the HIP experience worldwide up to now has probably been gained using the sinter-HIP-process (second right in fig. 2). It works well for example with many oxide ceramic materials for witch a density of about 95% of TD can be reached in pressure less sintering, without sacrificing the desired composition or microstructure of final product. An almost complete elimination of porosity can then be reached during the HIP-cycle. This method has for example been successfully used for the production of tool bits of oxide ceramics. In Mn-Zn and Ni-Zn-ferrites freedom from pores results in high permeability, high saturation induction and improved wear resistance. The fine and uniform grain size that can be obtained gives better high frequency characteristics and low ferrite noise.

In pies-electric ceramics, which for example are used in surface acoustic wave filters and oscillators, the fine and uniform grain size gives ease of processing to thin discs, e.g. of 0.05 mm thickness. The absence of pores is important in order to improve strength and obtain a surface without defects.

The most effective utilization of the inherent powder and versatility of HIP is however attainable encapsulation is used. Only in this realization of HIP can pressure be utilized throughout the shrinkage and consolidation phase – from green powder body to virtually pore free product. This is particularly valuable for non-oxide ceramics and for whisker or particle reinforced composites, regardless if the matrix material is an oxide or non-oxide ceramic. Usually metals like tantalum and niobium or glass systems are used for the encapsulation. The very high driving force for densification in encapsulated HIP due to the high and is statically applied pressure which opens unique possibilities to consolidate materials that cannot be made by pressure less sintering, often not even with uniaxial hot pressing.

Silicon carbide with a heat conductivity of ever 200 W/mk could be made by HIP, about 1.5 times and 2.5 times higher than for hot pressed or sintered silicon carbide, respectively. By using encapsulated HIP a fine grained, fully dense material was obtained, even with high purity powders. This in combination with complete abandonment of sintering aids is given as explanation to the high thermal conductivity. The author and coworkers have for similar reason found high heat conductivity in HIPed fully dense titanium diorite without sintering additives.

High purity silicon nitride powder without any additions of sintering aids have for a long time resisted full densification. By using high pressure and temperature it has recently been possible to fully density silicon nitride powder of very high purity, e.g. UBE SNE-10, and even make turbo charge rotor shapes of such powder without any additives. The UBE SNE-10 powder contained only about 20 ppm Al , 10 ppm Ca and 50 ppm Fe. A pressure of 250 MPa at 1950ºC for 2 hours using glass powder encapsulation resulted in a fully dense material with fine grains of low aspect ratio.

The 4 -point bend strength at 1350ºC was found to be 590 MPa, while the 3-point bend strength at 25ºC was only 500 MPa. This unusual behavior may however mainly be caused by the relative difficulty to machine test bars of this material without introducing grinding damage, which reduces the facture strength the most at low temperature. The density of the untapped material is in good correspondence with the theoretically calculated value and co-incites with a line previously drawn using data from HIPed silicon nitride compounds with 0.3 to 2.9 mol-% oxide additives.

The possibility to produce fully dense, very fine-grained and strong ceramic materials by selecting a low processing temperature and a high pressure in encapsulated HIP is of interest also for such materials as alumina. Alumina powder of 99.99% purity and without additives (Sumitomo AKP-30) could be HIPed to a fully dense material (> 99.5 % of TD) with fine structure and of high strength (Fig. 3).

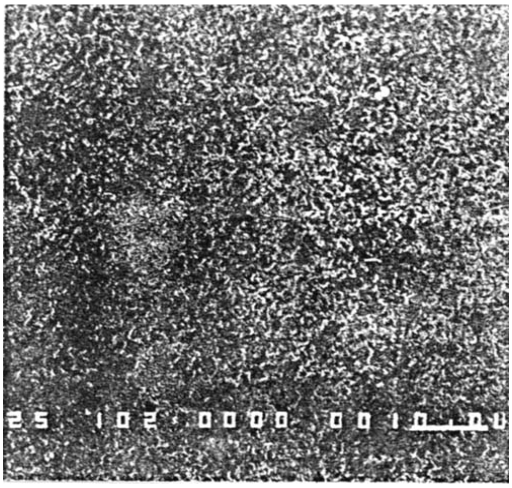

**Figure 3**. Fine grained, high purity alumina HIPed to full density at 1250°C, 200 MPa with modified glass powder encapsulation.

Silicon nitride material (Fig. 4) is a product made by glass encapsulated HIP that has now started its commercial introduction. Absence of porosity, homogeneity and consistency in all respects are crucial factors in many aircraft related applications.

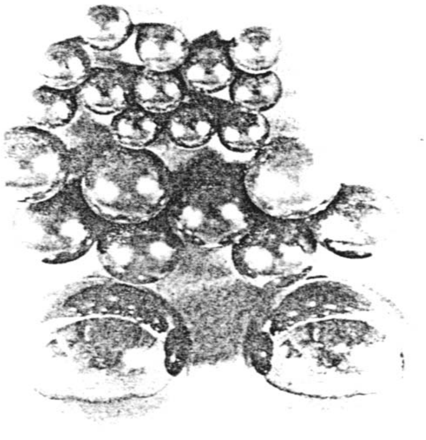

**Figure 4.** Silicon nitride balls for bearings.

Lucek and Walker found that finished silicon nitride bearings could be produced at a tenth of the cost of using hot pressed Norton NC-132 silicon nitride that was previously the preferred material for this application (and at only a slightly higher cost than for steel0. Still the improved micro structural uniformity increases fatigue life 4 to 10 times compared to NC-132. A 7.0 GPa contact stress, roughly twice the test load used to test steel materials, had to be used in order to obtain fatigue failures in reasonable times. Density is a physical property which is of great importance for example for balls for high speed bearings. Precise measurement of 25 HIPed silicon nitride blanks that had been ground to diam. 12.7 mm balls of high precision gave a density of 3.2483 g/cm with a standard deviation of only 0.0013 g/cm.

## 3. HIP AS MEANS TO IMPROVE SHAPE CONSOSTENCY AND TO JOIN DISSIMILAR MATERIALS

A theoretical study has clearly shown that encapsulated HIP has the inherent ability to help controlling the shape of a powder body, even if it has a complicated shape. The outside gas pressure, acting on the external surfaces of the powder body, gives rise to a very high pressure at the points of contact between powder particles.

The study show that the driving force for densification (effective pressure in particle contacts) can be several orders of magnitude higher than in pressure less sintering. The influence of gravity on unsupported parts of the body becomes virtually negligible and sagging or slumping of the part can be avoided. Consistent shape from part to part can consequently be obtained. A number of airfoil shapes with a 16 mm chord were NC machined from cold is statically pressed block. After HIP and encapsulation the airfoils were measured. The maximum deviation from the average profile was 0.03 mm of the parts with a chord of 13.5 mm. The deviations from ideal shape of airfoil made by injection molding and glass encapsulated HIP can be even lower. The best general comparison with existing production methods regarding product precision is probably with investment casting.

The high pressure available in HIP can also facilitate joining processes, both of similar and dissimilar materials, such a glass ampoule technique to join for example silicon nitride and alumina.

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