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DENSIFICATION OF CERAMICS BY GAS OVERPRESSURE SINTERING

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DENSIFICATION OF CERAMICS BY GAS OVERPRESSURE SINTERING

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ABSTRACT

The use of various gas pressure sintering (GPS) techniques
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INTRODUCTION

Gas pressure sintering (GPS) has been developed over the
past two decades as a pressure densification process for
ceramics which are difficult to conventionally sinter to full
density. It provides a useful, alternative processing strategy
for pressure assisted densification which permits the processing
of bodies more complex in shape than can be produced by uniaxial
hot pressing and by a lower cost process than hot isostatic
pressing (HIP). GPS is effective in suppressing material
dissociation at elevated temperatures, maintaining compositional
stoichiometry, and promoting densification with microstructural
control. A major advantage of the technique is that it does not
require the encapsulation or cladding and decladding of
specimens as processing steps. Use of the gas pressure
sintering method has been reported for producing ceramic
automotive components, ceramic tooling, rare earth cobalt
magnets, infrared windows and domes, lamp envelopes, and
electro-optic materials.

The gas pressure sintering or sinter/HIP process has been
used to densify various ceramics (oxides, nitrides, sulphides),
WC-Co cermets, rare earth cobalt magnets, and a variety of metal
alloys. A comparison of parameters and conditions encountered
in uniaxial hot pressing, gas pressure sintering (or
sinter/HIP), and hot isostatic pressing of various ceramics is
shown in Table I.

GAS PRESSURE SINTERING (GPS) METHOD

The process utilizes a pressurized gas, either reactive or
inert, to promote high temperature densification of specimens
from the "green"-body form. The gas pressurization/temperature
cycle has been conducted using several different modifications
but is usually described as either a one-step or two-step
process. In the one-step method, the gas pressure is raised to
a predetermined level and held at that level throughout the
sintering cycle. The sintering temperature may be held constant
or changed during the cycle depending on the densification
response and desired microstructural development. In the
one-step method, the primary role of the gas is to maintain

compositional stoichiometry and high temperature stability of the material being densified rather than to provide significant pressure to act as a driving force for densification. Since the gas used will be entrapped in the pores when they become isolated during densification, it is desirable that the gas have some solubility and diffusivity in the material to lower gas concentration within the pores and promote pore shrinkage. The densification of Si_3N_4 with MgO [1] or CeO_2 [2], in a N_2 gas atmosphere, are examples of this approach.

Table I. COMPARISON OF PARAMETERS AND CONDITIONS ENCOUNTERED IN VARIOUS PRESSURE DENSIFICATION PROCESSES

	Hot Pressing	Gas Pressure Sintering (Sinter/HIP)	Hot Isostatic Pressing
Container	Die/Plunger	Cladless	Cladded
Pressure (MPa)	10-70	0.1-200	70-200
Typical Temperature Range (°C)	-	-	-
Oxides	900-1700	1100-1800	900-1500
Nitrides	1600-1800	1700-2100	1600-1900
Sulfides	-	1000-1200	900-1100
Pressure Cycle	Single	Dual/Single	Single
Shape Capability	Simple	Complex	Complex

In the two-step method, the material is first densified to the closed pore stage (93-94% T.D.). Either a vacuum environment or a relatively low reactive gas pressure (0.1-2.0 MPa) is usually employed in this step of the process. For example, H_2S has been used for sintering sulfides [3,4], N_2 gas for nitride compositions [5-8], and vacuum or oxygen-rich environment for various oxides [9,10]. In the second step, the gas pressure is raised to provide an additional driving force to promote pore shrinkage and further densify the material. Since the pores have been isolated in the first step, encapsulation of the specimen is not required. The use of a gas with low solubility and diffusivity in the material is now desired unless high temperature compositional stability of the material remains an issue. Use of a combination of gases, each serving a different purpose, is also an option.

VARIATIONS OF THE GPS TECHNIQUE

Various modifications of two-step gas pressure sintering can be devised to promote densification and control microstructure. In Figures 1a-c, some examples of such modifications are illustrated. In Figure 1a, a typical sintering cycle is shown where the temperature is held essentially constant during the run while the pressure is increased from P_o, the initial gas pressure, to P_a, the final, external gas pressure level used in the second step of the process. When gas pressures up to 20 MPa are used in this step of the process, it is referred to as gas pressure sintering (GPS). If pressures are extended into the 70-200 MPa range, the process is usually identified as sinter/HIP (S/H).
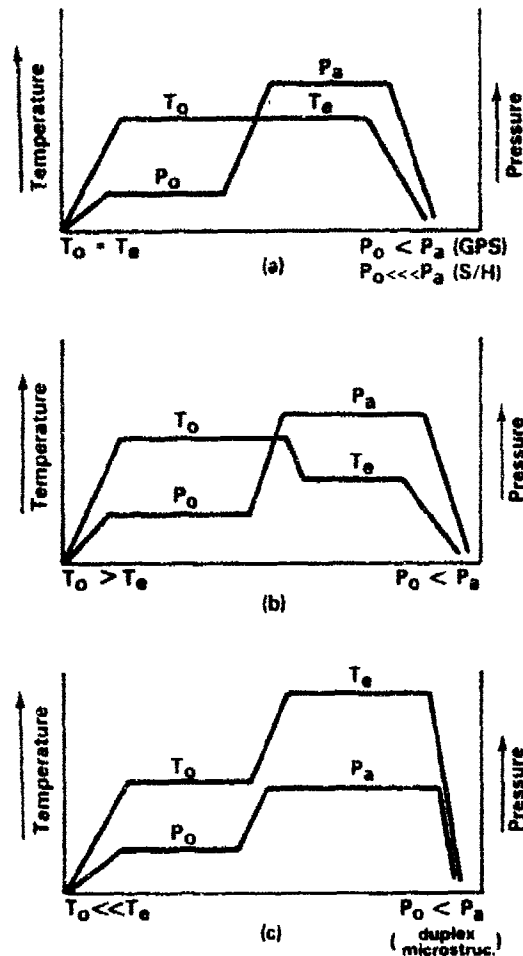
Figure 1. Variations of the two-step gas pressure sintering process.

Some GPS or S/H processing is carried out with a reduction in the sintering temperature in the second step as shown in Figure 1b. This is designed to restrict grain growth and pore coalescence. Additionally, Greskovich [7] has used this approach with BeSiN$_2$ doped Si$_3$N$_4$ to reduce equilibrium pore sizes and control the distribution of Be from the grain boundaries into the Si$_3$N$_4$ grains.

Another variation of the process, as shown in Figure 1c, employs a significant increase in temperature in the second step to promote an increase in grain size and formation of a duplex microstructure to increase material toughness. This approach is referred to as in-situ microstructural toughening or development of self-reinforced microstructures.

ANALYSIS OF PRESSURE/PORE SIZE RELATIONSHIP

A theoretical analysis of high gas pressure processing to reduce equilibrium pore sizes in ceramics has been recently discussed by other investigators [7,11,12]. When open porosity exists in a material, shown schematically in Figure 2a, the driving force for pore size reduction and pore closure is essentially the capillary stress or surface tension denoted as

$2(Y)/r_{op}$, where Y is the solid-vapor surface energy and r_{op} is the open pore radius. As pore isolation occurs, illustrated in Figure 2b, the driving force for pore size reduction includes both the magnitude of the external pressure, P_a, and the surface tension term. If the pore contains an insoluble gas and remains isolated without coalescence, the equilibrium pore size and gas pressure within the pore is given by,

$$P_e = 2(Y)/r_e + P_a \qquad (1)$$

Figure 2. Driving force for pore shrinkage
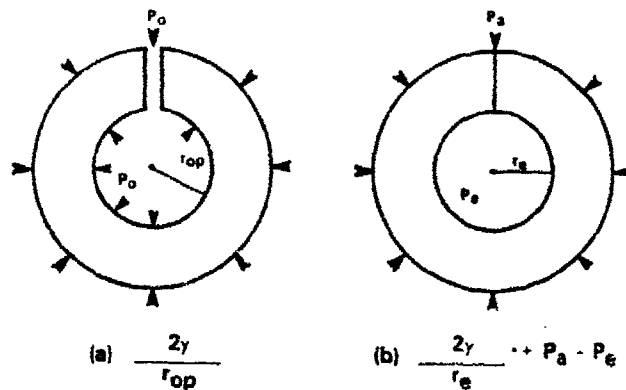
One Step: $P_o = P_a$
Two-Step: $P_o < P_a$

where r_e is the pore radius at equilibrium conditions, P_e is the equilibrium gas pressure within the pore, P_a is the external gas pressure, and Y is as previously defined. The presence of an insoluble gas within the pore limits pore size reduction and results in the forming of end point densities. If pore coalescence occurs due to grain growth, the driving force for pore closure will be reduced and larger pore sizes will result in lower densities. If the pores contain a soluble gas with high diffusivity through the ceramic, reduction in pore size occurs as a function of gas solubility and diffusion kinetics, and high densities are more easily achieved.

When pores contain a gas with low solubility in the matrix, the smallest equilibrium pore size can be shown to be a function of both the initial pore size and the magnitude of the pressure used for densification. Combining equation (1) with ideal gas law equations for both initial and equilibrium conditions gives,

$$P_a(r_e)^3 + 2(Y)(r_e)^2 = (P_o)(T_e)(r_o)^3/T_o \qquad (2)$$

where P_o is the initial gas pressure, T_e is the equilibrium temperature, T_o is the initial temperature used in the process, r_o is the initial pore radius after pore isolation, and P_a, r_e, and Y are as previously identified. For Si_3N_4, Y is estimated to be 1 J/m^2. In considering the gas pressure densification of Si_3N_4, the effect of using different gas pressures, in a one-step sintering process, on the equilibrium pore size (r_e), is shown in Figure 3a as a function of the initial pore size (r_o). Both the gas pressure and sintering temperature are held constant during the process producing the conditions $P_a = P_o$ and $T_e = T_o$. It is observed that the values of r_e increase with

both increasing gas pressure and initial pore size. Therefore, to produce smaller pores and higher densities, the use of finer starting powders (producing finer pore sizes) has been suggested [11] along with using the lowest possible acceptable gas pressures before the closed pore stage is reached. Particle size distributions that produce high packing densities should also be effective.

A reduction in the equilibrium pore size can be further accomplished by the use of higher gas pressures after pore isolation has occurred, i.e., in the second step of a two-step process. In Figure 3b, the influence of gas pressures (P_a) of 3.6, 10, 100, and 200 MPa on the reduction of the equilibrium pore size is shown when initial pore sizes up to 2.5 um are considered. The sintering temperature is assumed to be constant with $T_e = T_o$. The initial pressure, P_o, is fixed at 2 MPa for purposes of the calculations. The plots illustrate the effectiveness of raising the external gas pressure to the sinter/HIP pressure levels which produce equilibrium pore sizes approximately 1/2 to 1/3 of the size achieved by gas pressure sintering at 10 MPa. The combination of higher gas pressure applied after pore closure and smaller initial pore size would result in the highest material density. Another advantage of using higher pressures may involve increasing the solubility of the gas entrapped in the pores into the matrix of the material being densified (13).

Figure 3. Equilibrium pore size (r_e) as a function of (a) initial pore size (r_o) and gas pressure and (b) initial pore size (r_o) and final external pressure (P_a).
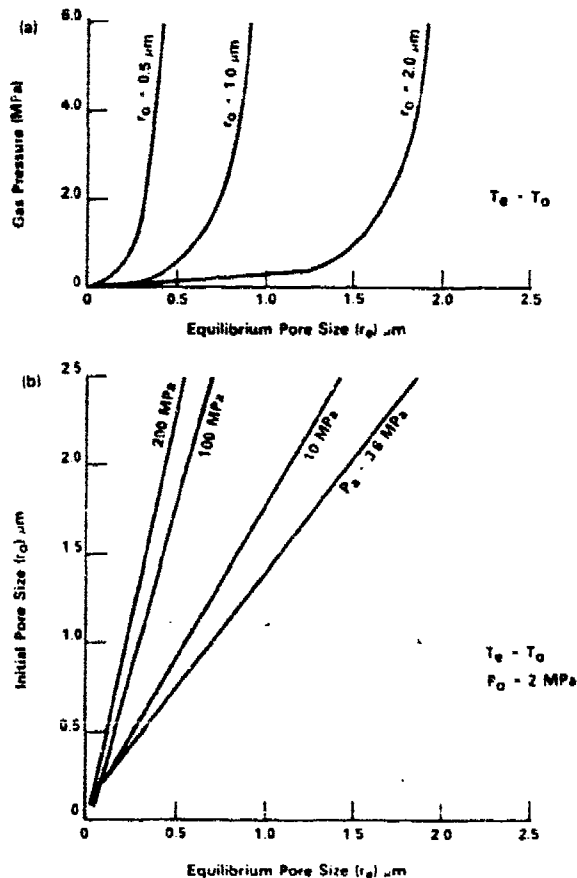
MICROSTRUCTURAL CONTROL

The lack of toughness in monolithic ceramics has spurred the development of whisker and fiber reinforced materials. However, depending on size and aspect ratio, processing of the whisker materials is costly due to the need for a more complex materials synthesis route and associated health issues. This led to the development of monolithic Si_3N_4 with a duplex or composite type microstructure. Early studies [6] demonstrated that by increasing gas pressure sintering temperatures to higher levels than normally used, i.e., 2000C, the fracture toughness of the material could be significantly increased due to the development of a fiber-like structure, that is, large, high aspect ratio grains forming in-situ in a finer grain Si_3N_4 matrix. The additions of rare earth oxides, such as Y_2O_3, La_2O_3, or CeO_2, with Al_2O_3 produced high densities and a grain boundary phase that promoted toughness by tailoring interfacial properties between the grain boundary phase and the Si_3N_4 grains [6,14]. The two-step gas pressure sintering process was found to be the most effective for producing toughness.

A comparison of microstructures for a gas pressure sintered and hot isostatically pressed Si_3N_4 is observed in Figures 4a and 4b, respectively. The GPS material exhibits a coarse, duplex structure with high aspect ratio grains, whereas the hot isostatically pressed material has a fine, more equiaxed structure. The use of higher GPS temperatures, e.g., 1975C-2100C, or longer sintering time results in the formation of large, elongated grains in the matrix and produces higher toughness values. The densification temperatures for HIPPED Si_3N_4 are usually 200-400C lower than for the gas pressure sintered material.

(a)   (b)
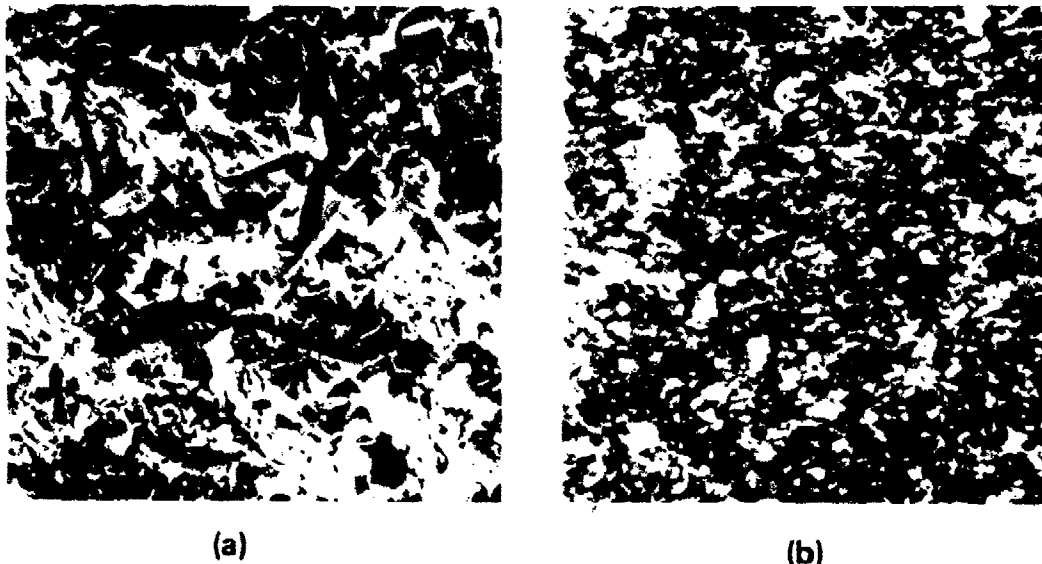
Figure 4. Microstructures of Si_3N_4 containing Y_2O_3 sintering additive. (a) Gas pressure sintered (b) Hot isostataically pressed.

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EXAMPLES OF THE APPLICATION OF GPS TO ADVANCED CERAMICS

Specific examples of different classes of ceramic materials densified by gas pressure sintering (GPS) or sinter/HIP (S/H) processes are shown in Table II. In the Table, the ceramic materials are identified along with the pressures and temperatures used to densify them. It can be determined under the Densification Parameter columns whether a one-step or a two-step gas pressure process was used. Examples of cermets and metals densified by GPS or S/H are available in References 15 and 16.

Table II. CERAMIC MATERIALS DENSIFIED BY GAS PRESSURE SINTERING OR SINTER/HIP

| | | Densification Parameters | | |
| | | 1st Step | 2nd Step | |
Material	Process	Temp (°C)/Press (MPa)	Temp (°C)/Press (MPa)	Ref
$Si_3N_4 \cdot MgO$	GPS	1800-1900°C/1 MPa N_2		1
$Si_3N_4 \cdot BeSiN_2$	GPS	2000°C/2.1 MPa N_2	1900°C/7.1 MPa N_2	7
$Si_3N_4 \cdot Y_2O_3/Al_2O_3$	GPS	1850-2000°C/1 MPa N_2		5
$Si_3N_4 \cdot R.E.$ Oxides	GPS	1700-1800°C/0.2-4 MPa N_2	2000°C/4 MPa N_2	14
$\beta' \cdot$ Sialon	GPS	1600-1800°C/0.5 MPa N_2	1650°C-1700°C/10 MPa N_2	8
Al_2O_3	S/H	1650°C/vac.	1650/100 MPa Ar	9
PLZT; $BaTiO_3$	GPS	1170°C/0.1 MPa O_2	1170°C/20 MPa Ar	10
$CaLa_2S_4$	S/H	1050-1150°C/0.1 MPa H_2S	990°C/200 MPa Ar	4
$CaLa_2S_4 \cdot La_2S_3$	S/H	1200°C/0.1 MPa H_2S	1090°C/200 MPa Ar	3

Oxides

Early gas pressure sintering or sinter/HIP studies focused principally on oxide materials, such as, Al_2O_3 [9], $Pb(Zr,Ti)O_3$, $BaTiO_3$, and $SrTiO_3$ [10]. The technique involved sintering in a vacuum environment or using a gas with high diffusivity through the material being densified in the first step of the densification process. Oxygen was used for this purpose and its presence was important to maintain the stoichiometry of the oxide being densified. When the closed pore stage was reached, a gas with a low diffusivity in the oxides (either He or Ar) was used at higher pressures to increase the density. A helium gas pressure of 100 MPa was used to further densify Al_2O_3 [9] while approximately 35 MPa of argon was used for densification of the titanates [10].

Sulfides

The sinter/HIP process has been applied in the densification of sulfide compounds [3,4] to produce a material with improved optical and infrared transmittance and increased erosion resistance. Hot pressing of $CaLa_2S_4$ or a $CaLa_2S_4$-La_2S_3 solid solution compound in a graphite die results in poor optical transmission due to material decomposition and loss of sulphur. Sintering of the compound in H_2S to the closed pore stage followed by cladless hot isostatic pressing, in an Ar environment, produced a more stoichiometric material with higher transmittance. Sintering temperatures used in the first step of the process were 1000-1200C. The cladless HIP step was performed in an Ar environment at temperatures between 990-1090C. Ar gas pressure used was 170-200 MPa.

Nitrides

Early attempts to densify Si_3N_4 using a conventional sintering approach [17], i.e., under 0.1 MPa N_2, were only partially successful because of difficulty in suppressing dissociation reactions. Maximum density attained was limited to approximately 80-85% of full density. The need to use high sintering temperatures to promote diffusional processes for densification and, at the same time, restrict decomposition of the material led to the use of high N_2 gas pressure during sintering. To determine the amount of nitrogen gas pressure required to stabilize the Si_3N_4 at a given temperature, a diagram defining the region of sinterability was thermodynamically determined from the reaction in which the nitrogen pressure and silicon vapor pressure are in equilibrium with Si_3N_4. This diagram is presented and discussed in Ref.[18]. In addition to suppressing the decomposition of Si_3N_4 into Si and N_2, high N_2 gas pressure will suppress the rate of dissociation reactions producing SiO and N_2, limiting oxygen removal from the system. The use of a cover powder in which the specimen is embedded has also been shown to be effective for this purpose [19].

Si_3N_4, doped with 5 w/o MgO [1] or CeO_2 [2] as densification aids, was sintered by single step GPS to >95% dense with weight losses up to 6-8%. Temperatures between 1800C and 1950C were used for sintering. Nitrogen gas pressures of 1-2 MPa were used principally to suppress decomposition of the Si_3N_4.

The use of a two-step approach to sinter Si_3N_4 close to full density was initially applied using Be_3SiN_2 [7], and Y_2O_3/Al_2O_3 (5) as additives. The two-step, pressure-temperature approach used to densify the Be_3SiN_2 doped Si_3N_4 was designed to utilize differences in the temperature dependent solubility of Be in the Si_3N_4 grains. Since the solubility of Be in Si_3N_4 decreases at temperatures greater than 2000C, a temperature of 2050C was used in the first step to keep a Be containing liquid at the grain boundies to promote diffusion and densification. N_2 gas pressure was kept at 2 MPa during this sintering step. When the closed pore stage was reached, the sintering temperature was lowered to 1950C to increase diffusion of Be into the grains. The gas pressure was increased to 10 MPa to

reduce the equilibrium pore size and promote further densification.

COMPARISON OF GPS WITH HOT ISOSTATIC PRESSING

Gas pressure sintering and hot isostatic pressing are both useful processes for densifying complex shapes. However, each process has certain advantages and disadvantages associated with the technique. Some of the strengths and limitations for each process are listed in Table III. From the standpoint of materials properties, such as high strength and reliability, the HIP process is the preferred method of densification. It can produce material with uniform microstructures and compositional stability. The major concerns are the need to use or develop cladding materials that have properties appropriate for the stress-temperature conditions required for densification and the possible reaction of the cladding materials with the specimen. Higher equipment and processing costs are further concerns. With the GPS process, cladding/decladding steps are not required and the versatile temperature-pressure cycles can be used for tailoring microstructures, such as in-situ toughened materials. Some problems with GPS may include loss of volatile compositional components, reaction with the gas environment, and the inability to close surface porosity. The latter problem may be particularly undesirable for the densification of ceramic components, such as bearings, where high surface or near-surface stresses are encountered and an exceptional quality of surface finish is required. Since higher temperatures are usually required for GPS than for HIP, coarser microstructures are generally produced by GPS. However, overall processing costs are lower for the GPS technique.

Table III. ADVANTAGES AND DISADVANTAGES ASSOCIATED WITH GAS PRESSURE SINTERING AND HOT ISOSTATIC PRESSING PROCESSES

	ADVANTAGES	DISADVANTAGES
Gas Pressure Sintering	• Versatile for tailoring microstructure	• Larger grain size than HIP
	• No cladding required	• Deleterious reaction with sintering environment
	• Higher matl. toughness	• Loss of compositional components
	• Lower Cost	• Surface porosity remains open
Hot Isostatic Pressing	• Fine grain size	• Need to develop appropriate container material
	• Higher strength and Weibull modulus	• Reaction with container material
	• No reaction with gas environment	• Higher processing cost
	• Reduced additive levels where required for densification	
	• Compositional stability	

SUMMARY

(1) Gas pressure sintering can be used to densify both oxide and non-oxide ceramics for a variety of applications.

(2) Process parameters are tailored to material densification and microstructural goals, usually in a one or two-step process.

(3) The smallest equilibrium pore sizes (highest densities) are produced by:

    (a) starting with a small initial pore size (r_o), i.e., a fine powder
    (b) using a vacuum or the lowest gas pressure possible prior to pore closure (P_o)-step 1 of the process
    (c) raising the external gas pressure (P_a) to high levels after pore closure-step 2 of the process
    (d) using gas with a high solubility/diffusivity in step 1 and a low solubility/diffusivity in step 2.

(4) GPS is competitive with HIP where, (a) only moderate conditions of pressure are necessary for densification, (b) changes in specimen composition do not occur by volatilization or undesired reactions with the gas environment, and (c) reductions in processing cost must be considered.

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DENSIFICATION OF CERAMICS BY GAS
OVERPRESSURE SINTERING -
George E. Gazza and R. Nathan Katz

Key Words

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The use of various gas pressure sintering (GPS) techniques for densifying ceramics
are reviewed for both oxides and non-oxides. Variations of the process are dis-
cussed with respect to process parameters selected, process sequence, and micro-
structural development. Theoretical considerations underlying the technique are
presented. GPS and hot isostatic pressing are compared and the advantages and dis-
advantages of each are briefly discussed.