

# Effect of Magnetic Pulsed Compaction (MPC) on Sintering Behavior of Materials

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## 1. Introduction

Sintering is one of the most frequently used processing techniques in the studies of material science that is used to produce density-controlled materials and components from metal or/and ceramic powders by applying thermal energy. Hence, sintering is categorized in the synthesis or processing element among the basic elements of materials science and engineering. As material synthesis and processing have become immensely vital in recent years for materials development, the importance of sintering is increasing as a material processing technology. Sintering is, in fact, one of the oldest technologies, originating in the prehistoric era with the firing of pottery. The production of tools from sponge iron was also made possible by sintering. Nevertheless, it was only after the 1940s that sintering was studied fundamentally and scientifically. Since then, remarkable developments in sintering science have been made. One of the most important and beneficial uses of sintering in the modern era is the fabrication of sintered parts of all kinds, including powder-metallurgical parts and bulk ceramic components (German, 1996).

Sintering mostly aims to produce sintered parts with reproducible and, if possible, designed microstructure through control of sintering variables (Chiang et al., 1996; Green et al., 1989). Microstructural control means the control of grain size, sintered density, and size and distribution of other phases including pores. In most cases, the final goal of microstructural control is to prepare a fully dense body with a fine grain structure.

Now in order to improvise the conditions of sintering, a number of researches have been carried out throughout the world. By sintering conditions, we mean the parameters involved in the process, namely, sintering time, temperature, rate of temperature increase, pressure involved in the process, or even the microstructural behavior in greater sense. This chapter focuses on one of these recently developed techniques called Magnetic Pulse Compaction, which may be used as an additional process on materials, prior to sintering. In special cases, based on commercial requirements, this process can stand alone, without even having the necessity of sintering.

In addition to the detailed description of Magnetic Pulse Compaction, a number of material fields have also been tried to cover where this process can either be used, or has successful

contribution compared to other conventional methods. Several experimental results and reports have been discussed, especially on ceramics and hard-to-compress materials. In each case, we have assayed to delineate the differences that this process is introducing, the contributions it has, or the necessity to incorporate this in the chain of processes. Finally, we made some concluding remarks based on real term experimental results about the advantages of this process, which may aid certain streams of material research. Applications of MPC, both as a stand-alone process and prior to sintering have been highlighted throughout the entire chapter.

## 2. Magnetic pulse compaction

In the commercial sector there is a constant demand for high density, net shape parts that come with an affordable price. At the moment automotive parts, such as powertrain gears for high- performance applications, are typically machined from forged and wrought blanks. Due to high machining costs, these components are much more expensive than conventional press and sintered powder metallurgy (PM) parts. Although PM hot pressing and extrusion techniques can be used effectively to produce bulk materials, these processes are costly due to numerous intermediate steps involved in the process (Kim, 1998; Seok et al., 2004). Beside, in static compaction methods like cold pressing, hot isostatic pressing etc., the particles often have slow movement with the range of  $10^{-3}$  to  $10^{-4}$  m/s of velocity, resulting in grain growth of nanopowders during the static process (Lee et al., 2004). One of the ways to produce high relative density powder product is magnetic pulse compaction (MPC), which is a kind of dynamic magnetic compaction (DMC) technique. Having superiority to other compaction methods in energy control and forming efficiency, DMC is a kind of high-energy rate method which can be used to produce composite powder, ceramic, metal etc. (Min et al., 2010). While using MPC, it is possible to apply a high pressure (up to 5 GPa) to powders within a short period of time ( $\sim 500\mu\text{s}$ ). This ultra high pressure in  $500\mu\text{s}$  brings about an enhanced deformation and microstructural rearrangement to these compacted powders, resulting in the improved green density (J.G. Lee et al., 2010).

In the static compaction methods, in general, the particles move slowly with the range of  $10^{-3}$  to  $10^{-4}$  m/s of velocity, whereas in MPC process it is possible to achieve the high speed movement of particle over 10-100m/s owing to very high pressure, more than 1GPa (Lee et al., 2004). During MPC, since effect time of the magnetic force is too short, it can be considered that there is an impulsive force acting in MPC and it is well known that impulsive forces are very high, which are capable of breaking the activation energy of the compacted particle to move them from their places in the bulk.

Magnetic Pulse Compaction (MPC) is a new PM process that can produce components like these at costs comparable to the single-sinter, single- sinter route. In addition, MPC technology is also expected to find use in applications where special shapes, characterized by radially symmetric geometries and/or long length/diameter (L/Ds), are required.

Magnetic forces have been used for more than two decades in high rate metal forming and powder compaction. The same electro-magnetic based pulse forces are used in MPC to realize net shape powder consolidation. The powder-pressing route makes use of transmitted impact energy in a process analogous to driving a nail into a board with a

hammer. While almost any material can be compacted to full density using a sufficiently large impact pressure, the important benefits of magnetic powder compaction when compared to die pressing are higher green densities and higher aspect ratios.

The basic working principle of the Magnetic Pulse Compaction process is shown in Figure 1. Powders are filled in a conductive container (armature) placed in the bore of a high field coil. The coil is pulsed with a high current to produce a magnetic field in the bore using the Biot-Savart law, which in turn, induces currents in the armature using Ohm's law.

Biot-Savart Law:

$$\vec{B}(t) = \frac{\mu_0 I(t)}{4\pi} \int \frac{d\vec{l} \times \vec{r}}{r^2} \quad (1)$$

where  $\mu_0$  is the permeability of free space,  $r$  is the distance from the element to the point  $P$ , and  $\hat{r}$  is a unit vector pointing from  $dl$  toward point  $P$ . We find the total field at  $P$  by integrating this expression over the entire current distribution. Now, Faraday's law of induction states that the emf ( $\varepsilon$ ) induced in a circuit is directly proportional to the time rate of change of magnetic flux through the circuit.

Faraday's Law:

$$\varepsilon = - \iint \frac{d\vec{B}}{dt} dy dx \quad (2)$$

Hence, current ( $I$ ) is induced using the Ohm's Law

Ohm's Law:

$$I(t) = \frac{1}{R} (V - \varepsilon(t)) \quad (3)$$

where  $V$  is terminal voltage,  $R$  is resistance of external resistor and  $\varepsilon$  is the induction magnetic field. The induced currents interact with the applied magnetic field to produce an inwardly acting magnetic force that collapses the tube, thereby compacting the powder, using Lorentz force.

Lorentz Force:

$$\vec{F} = I(t) \int dx \times \vec{B} \quad (4)$$

where  $I(t)$  and  $\vec{B}$  are induction current and magnetic field, respectively. The armature is launched into the powders with a large kinetic energy within a few microseconds of the compaction cycle. The powders are pressed to full density via the transmitted impact energy, with the entire compaction occurring in less than one millisecond.

The process steps of MPC are similar to conventional PM compaction technique, and include tooling for compaction, powder filling, part extraction and sintering, as well as the optional steps of sizing and finishing. In most commercial applications, the powder filling and compaction are done in air medium at room temperature. The filling can also be carried

out in special environments, such as in an inert gas or other cover gases, for special applications. The powders can also be compacted at elevated temperatures with suitable system modifications. The *MAGNEPRESSTM* system used in the DMC process consists of four subsystems - pulsed power system, electromagnetic field coil, material handling capabilities, and programmable logic controller (PLC) systems. The power supply has a modular design and has been designed to accommodate expanding energy needs. High production rates up to ten parts per minute can be achieved with this system. Material handling capabilities, such as powder filling and transportation of filled cassettes, can be easily integrated into the system for a given application.

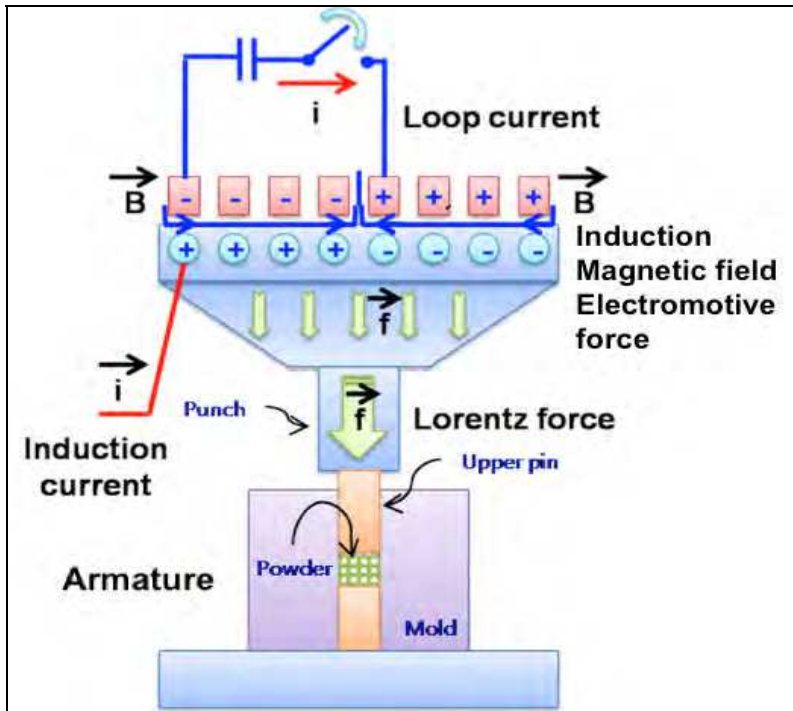


Fig. 1. Working principle of Magnetic Pulse Compaction

### 3. Effects of MPC on different types of materials

#### 3.1 Ceramics: Effects of MPC on alumina ( $\text{Al}_2\text{O}_3$ )

It is of great importance to know how ceramic nanopowders behave under Magnetic Pulse Compaction, and their consequent effects after sintering. So, a number of experiments were carried out on  $\text{Al}_2\text{O}_3$  nanopowders, and their microstructural behavior, characteristics and mechanical properties were studied.

In the present investigation, dynamic compaction by magnetic pulsed compaction (MPC) and precompaction were conducted in order to prepare the  $\text{Al}_2\text{O}_3$  bulks. The starting powder was  $\alpha\text{-Al}_2\text{O}_3$  (purity of 99.8%) with an average powder particle size of 50–200 nm.

The  $\text{Al}_2\text{O}_3$  powder was formed into the shape of a disk by magnetic pulsed compaction (MPC). Table 1 demonstrates the compaction arrangements, the apparent features and final density of the bulks fabricated by the combination of precompaction, MPC, and sintering processes. In order to improve the density and properties, the starting powder was precompacted in a die under 110 MPa, 220 MPa, and 330 MPa, and then each precompacted sample without separation from the die was MPC-ed at room temperature. The obtained density of the MPC-ed specimen increased with increasing MPC pressure. The pressure required to consolidate the nanopowders is related to the force required to push the particles together. In order to push two particles together, the applied force must be equal to or greater than the resisting force. The ceramic powders are not expected to plastically deform during compaction like metals. In hard ceramics, a plastic deformation of the particles or the formation of a particle-to-particle contact is so difficult that the stored strain energy by the compaction pressure could not be readily relaxed, which results in the formation of cracks in the materials compacted at high pressure.

Experimental conditions	Density (%)
Uniaxial static compaction (110 MPa) + Sintering (1450 °C for 3 h)	90.0
MPC (0.5 GPa) + Sintering (1450 °C for 3 h)	90.0
MPC (1.25 GPa) + Sintering (1,450 °C for 3 h)	92.0
MPC (1.8 GPa) + Sintering (1,450 °C for 3 h)	90.0
Pre-compaction (110 MPa) + MPC (1.25 GPa) + Sintering (1450 °C for 3 h)	93.0
Pre-compaction (220 MPa) + MPC (1.25 GPa) + Sintering (1450 °C for 3 h)	94.5
Pre-compaction (330 MPa) + MPC (1.25 GPa) + Sintering (1450 °C for 3 h)	94.5

Table 1. Consolidation conditions and relative densities of sintered bulks by a combination of MPC and precompaction processing.

In addition, it is clear that density also increases with increasing precompaction pressures from 110 MPa to 220 MPa and then becomes stagnant at 94.5 % for precompaction pressure beyond 330 MPa. The highest density of 94.5 % was achieved in the sample precompacted at 220 MPa, while the density of the sample without precompaction was 92%. The means that precompaction of the powder before MPC improves the final density of the sintered bulk. This may be due to the higher initial packing density introduced by precompaction pressure, as well as the higher MPC pressure. The increased green density may be due to better packing associated with small particles filling the voids between the larger ones. Therefore, the pressure may play a role in the initial stage through particle rearrangement and distribution of agglomerates as well as in the later stages of densification. Additionally, it was reported that a high initial density is not only effective to enhance the subsequent densification during sintering, but also capable of limiting the

rapid grain growth (Bernache-Assolant et al., 1993). Therefore, it is necessary to very carefully control the strain energy during the fabrication of ceramic compacts while using high pressures, like in MPC.

Fig. 2 shows the variation in Vickers hardness as a function of MPC pressure and precompaction condition during consolidation. With increasing MPC pressure, the hardness of the bulk increases and the value is much higher than that of the uniaxial compaction. In addition, the difference in porosity in terms of size in different regions can be another important factor leading to the variation. The homogeneously distributed ultra-fine grains and higher density of the MPC-ed bulk might be the reasons for the increased hardness. The hardness of the bulk also increases with increasing precompaction pressure. This suggests that particle rearrangement during precompaction occurs at lower pressures.

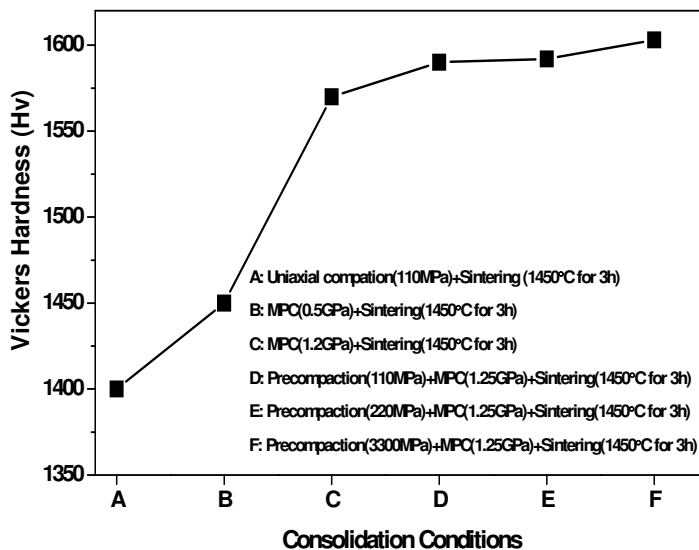


Fig. 2. Changes in Vickers hardness of sintered bulks as a function of different consolidation conditions.

This improved hardness with increasing precompaction pressure can be associated with the enhanced density of the bulk. These results clearly indicate that the precompaction prior to MPC and sintering is an efficient approach to promote higher density and Vickers hardness. Besides, the structural characteristics, which can be controlled by the consolidation and processing, such as porosity, internal stress, etc., may play an important role in determining the properties. However, it is ambiguous to distinguish the contribution of the property enhancement between the grain size effect and other effects such as pores or strains accompanying the consolidated samples.

Fig.3 represents the SEM micrographs (a and b) of the sintered bulks as a function of MPC pressure and precompaction, and TEM micrographs of the uniaxially compacted specimen and MPC-ed specimen (c and d). The microstructure of a commercial  $\text{Al}_2\text{O}_3$  plate (Fig. 3(a))

shows coarse grains and porous structures along the boundaries. The microstructure of the MPC-ed and sintered bulk exhibits small grains with fine pores. The average grain size measured for 0.5 GPa MPC-ed pressure without precompaction was  $0.74\mu\text{m}$  as shown in Fig. 3(b). On the contrary, the size range of the commercial  $\text{Al}_2\text{O}_3$  plate was nearly  $5.5\mu\text{m}$ , which is much higher than the MPC-ed one. Furthermore, the relative density of the sample precompacted at 220 MPa was the highest at 94.5%, and most of the resolved porosity appeared to be isolated at the grain interstices. Moreover, to distinguish the effect of the compaction on the microstructural characteristics between the uniaxial and MPC pressures, specimens were closely examined by TEM.

Fig. 3 (c and d) shows TEM micrographs of the uniaxially compacted specimen and MPC-ed specimen. A larger grain size with an average of 820 nm together with a considerable porous structure within the particles was observed in the case of uniaxial compaction (c) compared to the specimen compacted by MPC. The higher MPC pressure significantly influenced the decreased grain size as evident in figure 3 (d). Besides, the microstructure indicates a homogeneous dispersion of finer grains varying in size from 380 to 840 nm with an average of 600 nm.

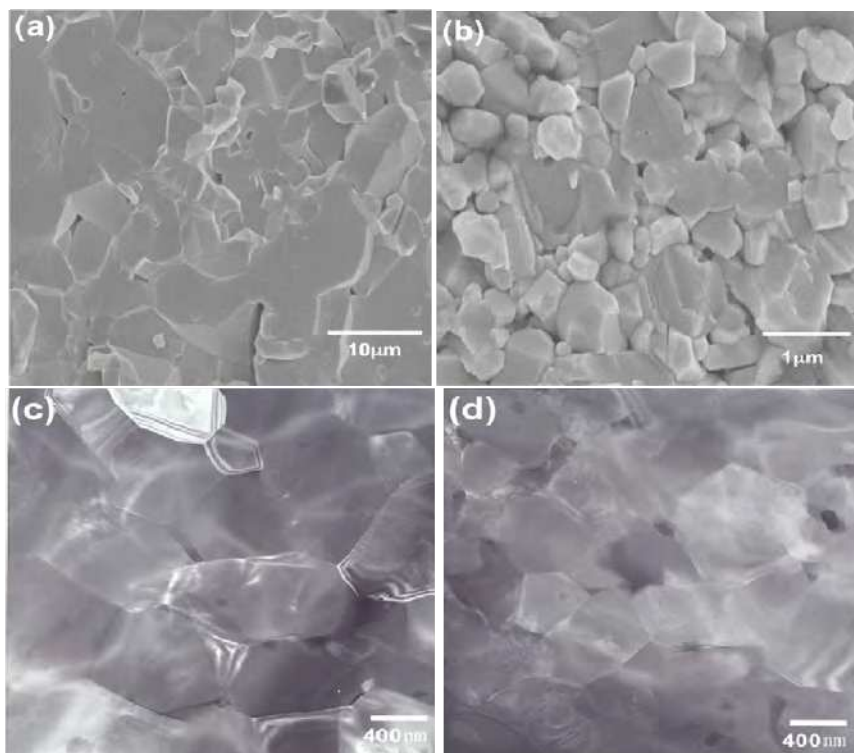


Fig. 3. SEM fracture surface of the sintered bulks as a function of MPC pressure and precompaction process; (a) Commercial  $\text{Al}_2\text{O}_3$ , (b) 0.5 GPa MPC, and bright field transmission electron images taken from (c) Uniaxial compaction (330 MPa) + Sintering ( $450\text{ }^\circ\text{C}$  for 3h) and (d) MPC (1.25 GPa) + Sintering ( $450\text{ }^\circ\text{C}$  for 3h).

The reliability of the consolidated bulk is significant to the generation and propagation behavior of the microcracks, and the indentation test is considered as one of the most effective and convenient methods to determine the fracture of materials. Fig.4 shows representative SEM micrographs of the cracks formed from the corner of the indenter. Despite the secondary cracking, a long crack of 50  $\mu\text{m}$  formed from each corner of the indenter and easily propagated without tortuosity in a spire  $\text{Al}_2\text{O}_3$  plate as shown in Fig. 4(a). However, the sintered specimens after the MPC show a higher decrease in the crack length (about 40  $\mu\text{m}$ ) compared to that of the spire  $\text{Al}_2\text{O}_3$  plate (an example is shown in figure 4 (b)). In order to identify the fracture mechanism of failure during the breakdown voltage testing, the fracture surface of the bulks was also studied using high magnification SEM micrographs as shown in Fig.4(c and d). Fig. 4(c) displays the fully formed hole and melted grain during testing. However, the observed fracture surface was basically different for the precompacted specimen (Fig.4 (d)). The micrograph shows a shallow hole. The hole formed in the bulk was smaller than that of the bulks without precompaction due to the higher density. The microstructure is coarser as a result of the high temperature during the voltage loading. However, it was difficult to observe any severe cracks, which were easily observed in the samples without precompaction. A fracture analysis demonstrated that bulks with a lower density are more prone to failure than the higher density ones; the lower the density, the greater is the probability to develop cracks and holes in it, and consequently, the lower the load it can withstand before fracture.

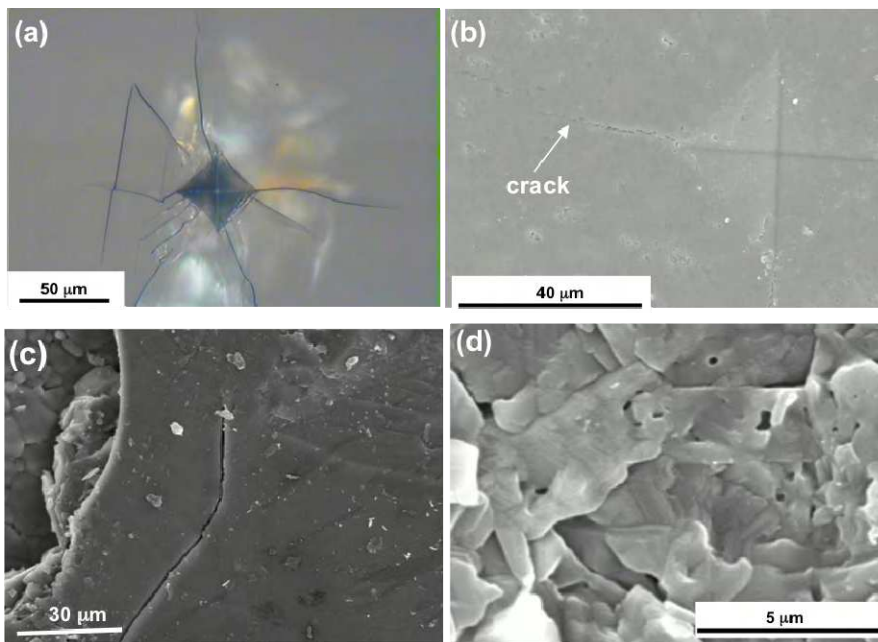


Fig. 4. SEM micrographs of cracks in sintered (a) commercial Sapphire, (b) 0.5 GPa MPC-ed  $\text{Al}_2\text{O}_3$  bulk, and SEM fracture surface of the sintered bulks after breakdown voltage testing as a function of compaction conditions; (c) commercial, (d) 220 MPa uniaxial precompaction+1.25 GPa.



Another reason is that the increased breakdown voltage in the MPC-ed and precompacted specimens is related to the grain size of sintered bulks. Though the commercial  $\text{Al}_2\text{O}_3$  showed a high density, a breakdown voltage decreased due to the coarse grain size. The intrinsic defects like the F, F<sup>+</sup> center in a single crystalline  $\text{Al}_2\text{O}_3$  act as heat generation sites during passing the high power wave by a second electron formation. As a result, cracks can be formed at those sites. Alternatively, the grain boundary can absorb the second electron. Therefore, it can prevent the heat generation. In this study, it is expected that the specimen consolidated by the combination of MPC and sintering process showed higher breakdown voltage values than that of the commercial  $\text{Al}_2\text{O}_3$  due to fine microstructure. Finally, these research results suggest the possible fabrication of the  $\text{Al}_2\text{O}_3$  bulk showing improved mechanical properties and breakdown voltage due to the ultra-fine microstructure and higher density by the combination of precompaction, MPC, and the sintering process.

It should be noted that the high strength and low dielectric constants of ceramics are attractive for application as a microwave window, but the statistical failure of ceramics severely limits their use. Especially, sapphire has been the best material for a microwave window because of its high strength, absorbed powder and good tolerance to radiation damage. A microwave window includes a transparent disk made of glass or ceramic that lets microwaves through and an attachment device made of metal that is used to attach the transparent disk. Specially processed and mounted sapphire windows have been shown to provide a significant improvement in microwave/radio frequency powder transmission capabilities compared to the current technology.

### 3.2 Effects of MPC on zirconia ( $\text{ZrO}_2$ )

Now similar experiments were carried out on  $\text{ZrO}_2$ .  $\text{ZrO}_2$  nanopowders of nanocrystalline polygonal particles with an average size of 50~100 nm were used. In case of specimens where the changes in densification behaviors were studied due to addition of  $\text{Y}_2\text{O}_3$  as sintering additives,  $\text{ZrO}_2$  powders were mixed with  $\text{Y}_2\text{O}_3$  with a binder (water in this study) to form a solution through mechanical mixing while the rest of the specimens were prepared without  $\text{Y}_2\text{O}_3$ .

Densification experiments were performed on  $\text{ZrO}_2$  powders using a die of superalloy 718 with 20 mm internal diameter and 100 mm external diameter, on a punch driven Magnetic Pulsed Compaction machine. Most of the experiments began by cold pressing a powder sample weighing ~10 g by loading up to 0.3~3.2 GPa range and holding the pressure for less than 6 microseconds. The cold compaction pressure was chosen so that a crack-free compact with a known low relative density could be produced. Cold compaction pressures up to 3.2 GPa were used in the set of experiments to study the effect of cold compaction on the hot pressing behavior.

The change in the behavior of the density and shrinkage as a function of varying MPC-ed pressure and 1450°C sintering temperature is delineated in Fig. 5(a & b) for the bulks having standard composition. Fig. 5(a) indicates that both densities of the MPC-ed specimen after first and second sintering increased with increasing MPC pressure from 0.5 GPa to 2.0 GPa, although depending on PVA condition, cracks occurred at times. It was possible to attain a density of 99% on sintered samples after the second sintering. It is due to the fact that high

MPC-ed pressure can break-up the weak agglomerates in the powder during compaction, consecutively making the pores smaller and size distribution narrower (Skandan et al., 1994). The increased green density is due to better packing associated with the smaller particles filling the voids between the bigger ones. The MPC pressure plays a role during the initial stage through particle re-arrangement and distribution of agglomerates as well as during the later stages of densification through plastic or super-plastic deformation. The removal of smaller pores requires much lower sintering temperature and/or shorter sintering period compared to the larger ones. Therefore, a high initial green density is not only effective to enhance subsequent densification during sintering, but also can potentially reduce the duration and sintering temperature significantly (Bruch, 1962; Occhionero & Halloran, 1984). Increasing green density is found to delay the onset of enhanced grain growth during the later stages of sintering (Bruch, 1962).

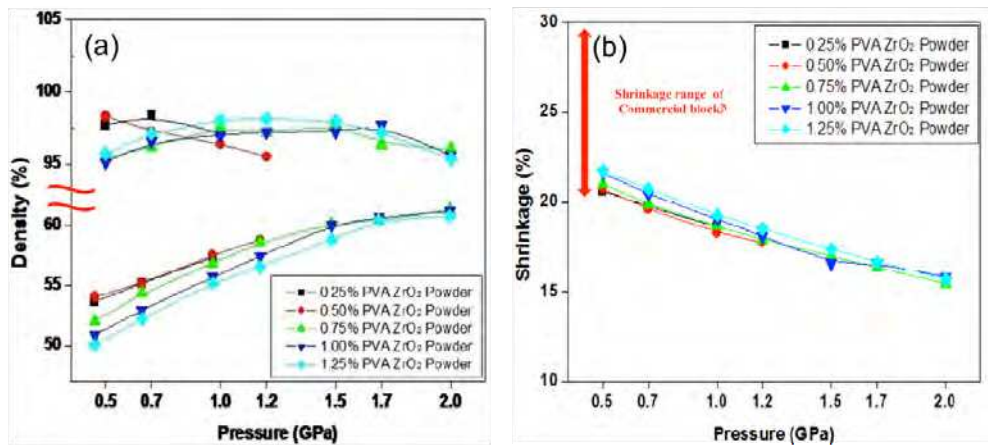


Fig. 5. Variation in (a) relative density and (b) shrinkage, as a function of MPC pressure at 1450°C.

During sintering process, on a microstructural scale, the bonding between particles occurs as cohesive necks grow at the particle contacts. The bonds between contacting particles enlarge and merge as sintering progresses. At each contact, a grain boundary grows to replace the solid-vapor interface, resulting in two particles to coalesce into single particle. Now, while MPC process is being used prior to sintering, particles on a first level, form bonds between contacting particles and initiates particle coalescing to a certain extent, which is then further progressed during sintering, requiring less sintering temperature and time. This interparticle neck growth, with a slight loss of surface area, can occur in powder compacts while improving properties. The densification parameter of the sintering process depends also on the pore elimination process in powder compacts. Now, during this short period of time while MPC process is being executed, pores tend to diminish with increasing pressure until certain limit, which in turn also helps the consequent sintering process to act faster. Apart from the contribution in particle coalescing and pore reduction, the pressure implied during MPC process also helps in shrinking the particles to a much greater extent compared to sintering process alone, given the same time. However, beyond certain extent, this gradually increasing

pressure can produce residual stress so high, that it can cause formation of cracks within and along the particles and thus prevents the required neck growth.

Fig. 6 shows an SEM image sequence obtained from consolidated  $ZrO_2$  bulks at a pressure range of 1.7 and 1.9 GPa, and sintered at  $1450^\circ C$ . It can also be delineated that the grain size is decreasing with increasing pressure and has reached a minimum, ranging  $2\sim 3\ \mu m$  at 1.9 GPa pressure, with temperature kept constant at  $1450^\circ C$ ; which is also in good agreement with results reported elsewhere (Li et al., 2006; Hong et al., 2007, Kim et al., 2011). Furthermore, in the sintered bulks, as shown in fig 6(a), pores can be observed although they are not widely dispersed and homogeneously distributed. As pressure tends to increase, pores are filled up and remarkably decreased as shown in fig 6 (c).

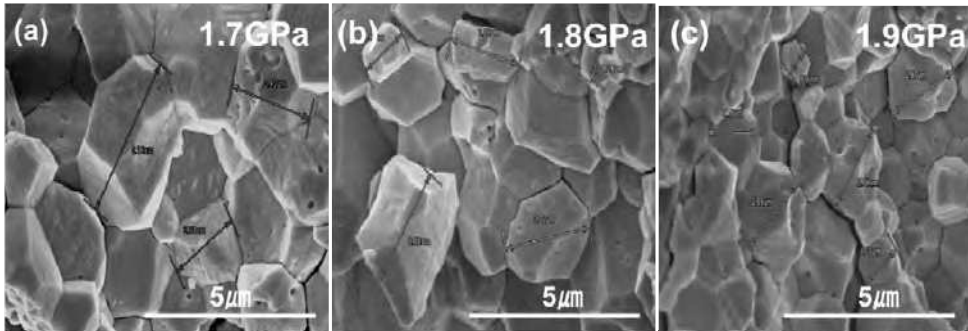


Fig. 6. Microstructures of consolidated bulks with varying pressure a) 1.7 GPa, b) 1.8 GPa, c) 1.9 GPa at  $1450^\circ C$ .

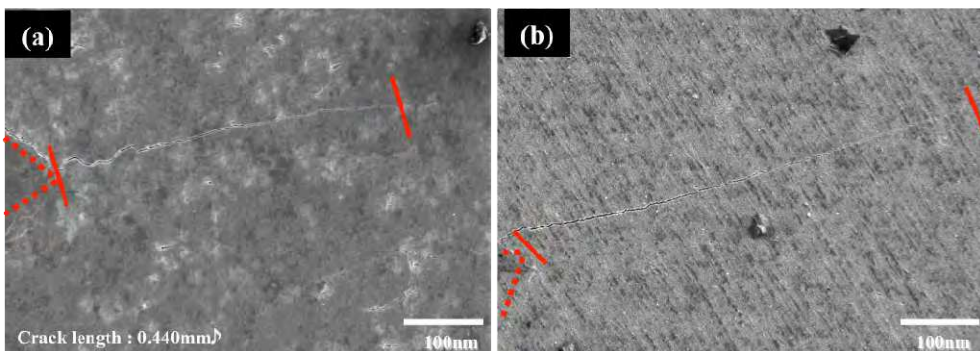


Fig. 7. Comparison of crack length between MPC-ed and commercial bulks (a) MPC-ed block, (b) Commercial block.

The effect of MPC on crack length behavior of sintered  $ZrO_2$  bulk has been compared with commercially available bulks, as shown in Figure 7. It is clearly seen that using MPC on ceramics can result in having smaller crack lengths compared to commercial bulks that haven't gone through this process. This behavior in crack length evidently suggests a resistance against fracture, external force or wear. It also reduces chances of having secondary cracking, or even propagation of crack that may result in failure.

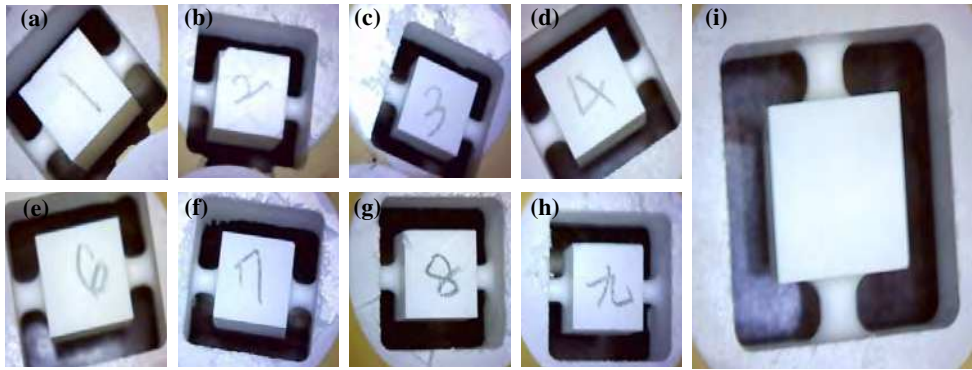


Fig. 8. Formability evaluation of sintered bulks (a) 0.75%PVA, 0.5GPa, (b) 0.75%PVA, 1.2GPa (Edge Crack) (c) 0.75%PVA, 1.5GPa (High Hardness), (d) 1.00%PVA, 0.7GPa (Good Quality) (e) 1.00%PVA, 1.2GPa (Good Quality), (f) 1.25%PVA, 0.7GPa (High Hardness) (g) 1.25%PVA, 1.5GPa (Crack), (h) 1.25%PVA, 1.7GPa (i) 1.00%PVA, 1.0GPa (optimum Quality).

In ceramics, it is also important to know how well or when materials can be machined. The term machinability refers to the ease with which a metal can be machined to an acceptable surface finish. Materials with good machinability require little power to cut, can be cut quickly, easily obtain a good finish, and do not wear the tooling much; such materials are termed as machining free materials. The factors that typically improve a material's performance often degrade its machinability. Therefore, to manufacture components economically, engineers are challenged to find ways to improve machinability without having to compromise with performance.

Machinability can be difficult to predict because machining has so many variables. Two sets of factors are the condition of work materials and the physical properties of work materials. The condition of the work material includes eight factors: microstructure, grain size, heat treatment, chemical composition, fabrication, hardness, yield strength, and tensile strength. Physical properties are those of the individual material groups, such as the modulus of elasticity, thermal conductivity, thermal expansion, and work hardening. Other important factors are operating conditions, cutting tool material and geometry, and the machining process parameters.

Now figure 8 shows the conditions of the sintered bulks, where the machinability as well as the formability of the sintered bulks was evaluated. The formability of most of the bulks was close to perfect. It could also be possible to machine the preforms right after using MPC, which can be highly efficient for industrial purpose.

The MPC process is ideally suited to produce net shape parts with cylindrical symmetry, thin walled tubes, high aspect ratio components and parts with internal features. Shapes and sizes of typical parts produced so far from powders range from 12.7 mm diameter x 76.2 mm long to 127.0 mm diameter x 25.4 mm long. Figure 9 shows some typical MPC-ed ZrO<sub>2</sub> targets (a) and their application as dental blocks (b).

One of the applications being developed is the production of zirconia dental blocks, which are being widely used in dental industries. Special applications of ceramics that go through

Magnetic Pulse Compaction are of great importance, due to the remarkable reduction of shrinkage in sintered bulks. While commercial bulks have considered double step sintering due to machining requirements in ceramics, it was found that simple MPC-ed bulks are capable of going through conventional machining. This could lead to an easier sintering approach altogether, if MPC process is applied in ceramics.

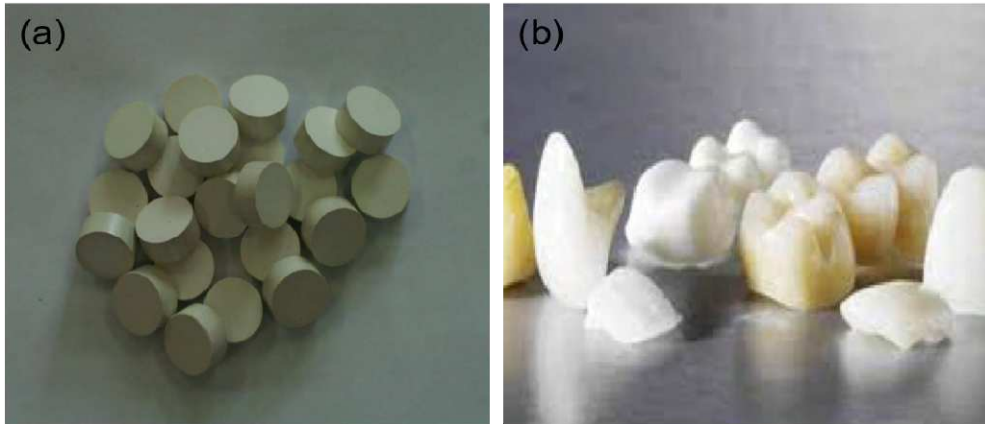


Fig. 9. (a)  $ZrO_2$  metalizing targets of the sintered bulks ( $1450^\circ C$  for 2hrs) and (b) their application as dental blocks compacted by MPC

### 3.3 Hard materials: Effects of MPC on sintered tungsten carbide-cobalt (WC-Co)

A number of alloy powders, namely ceramics, have gone through the combined MPC and sintering process. At this point, it is necessary to know how hard-to-compress materials react to similar processing conditions. Hard metal is the term used to signify a group of sintered, hard, wear-resisting materials based on the carbides of one or more of the elements; tungsten, tantalum, titanium, molybdenum, niobium and vanadium, bonded with a metal of lower melting point, usually cobalt. Tungsten carbide is however the most widely used one. These materials are commonly referred to as cemented carbides or simply as carbides as, for example, carbide tools. By varying the carbide particle size, the amount of binder metal, and the sintering conditions, the properties such as wear resistance, impact strength, resistance to cratering, and hot hardness may be optimized for a given application.

For example, in the case of a wire drawing die, wear resistance is a major requirement, but for a cutting tool, especially if subject to intermittent loading, high impact strength is required. Figure 10 shows the as-pressed density achieved in WC-Co. In most cases, high initial densities were achieved after MPC, with results getting higher when consecutive pressures were applied. The process parameters, used for some alloys, indicated in Figure 10, are not optimized; and higher densities than shown are possible.

The WC-Co MPC preforms were sintered at conventional PM sintering temperatures and were easily heat treatable. Their densities and hardness after sintering are illustrated in Figure 11 (a) and (b), respectively. It can be seen that hardness values greater than 1450 Hv were achieved in MPC processed WC-Co alloys. Figure 11 (a) illustrates the changes in

relative density for both WC-7.5wt%Co and WC-12wt%Co samples after magnetic pulsed compaction and sintering. Before and after sintering, samples of both compositions showed similar pattern with increasing pressure, and successfully reached a maximum of 97% dense state for WC-12wt% sample after sintering, which is higher than most commercial bulks. Here, the conventional explanation of the sintering mechanism of WC-Co comprises of some of the WC dissolving into the cobalt binder phase, migrating and re-precipitating on the surface of the original WC (Schwartzkopf & Kiefer, 1960). The final product consists of a three-dimensional skeleton of WC grains with cobalt as a binder phase matrix (Silva et al., 2001; Cha et al., 2001). In hard alloys such as WC-Co or TiC, even a slight difference in hardness value with respect to their constituents' content, pressure or grain size can mean a change to their mechanical behavior.

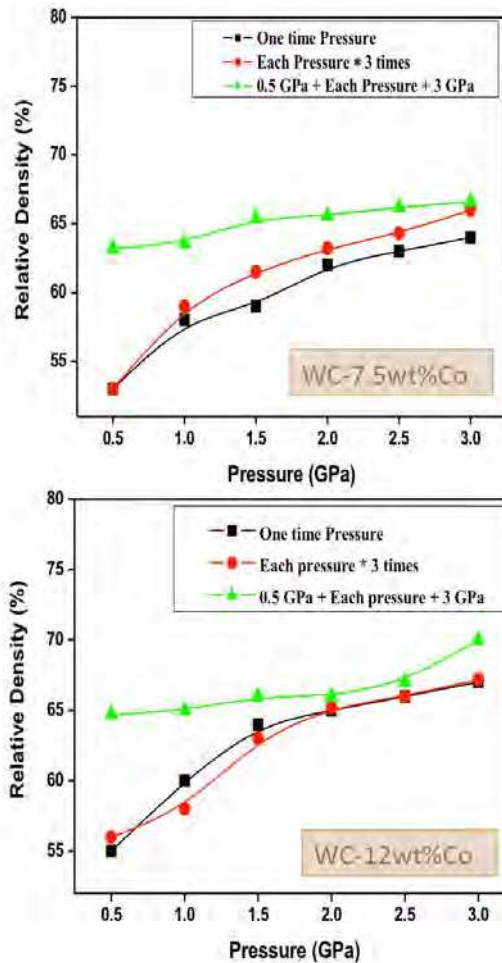


Fig. 10. Changes in initial density with varying MPC-ed pressure on a) WC-7.5wt%Co and b) WC-12wt%Co at room temperature.



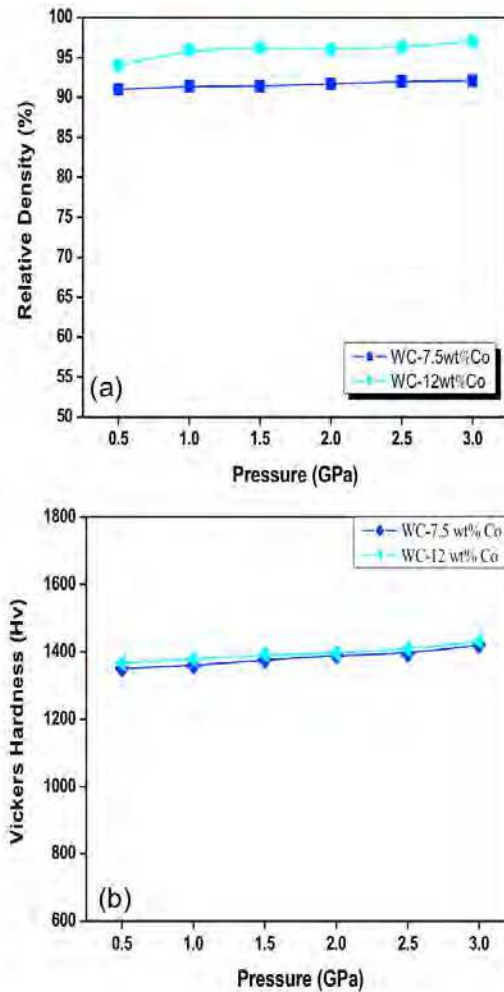


Fig. 11. Changes in (a) relative density, (b) hardness of WC-7.5wt%Co and WC-12wt%Co samples with MPC-ed pressure.

Fig 12 shows the changes in microstructure of sintered samples with varying MPC-ed pressure and Co content. It can easily be comprehended that these fine grained WC got more homogenously distributed in Co binder phase with increased pressure and Co content, at an elevated sintering temperature of 1450°C. It may have as well resulted in having these samples display a higher relative density and hardness for high magnetic pulsed compaction, especially after sintering, even at a lower operating temperature. Now, it is comparatively easier for finer grained WC material to sinter at lower temperature and become more homogeneous. The first stage in this mechanism is the spreading of binder phase on the WC grains. This happens within minutes after the temperature reaches 1000°C for fine-grained WC. For coarse-grained WC it happens at ~1100°C, even though the W-Co-

C system has a ternary eutectic at  $\sim 1275^{\circ}\text{C}$ . The second stage is that Co coated WC particles agglomerate into the Co liquid matrix; this happens faster for the fine-grained WC. The next stage is the forming of a network of agglomerates. The capillary forces close the pores and contract the sample towards an equidimensional shape in competition with the gravitational force, which forces the WC particles to sediment vertically. The last stage is the slight growth of the original WC grains from the tungsten and carbon dissolved in the cobalt.

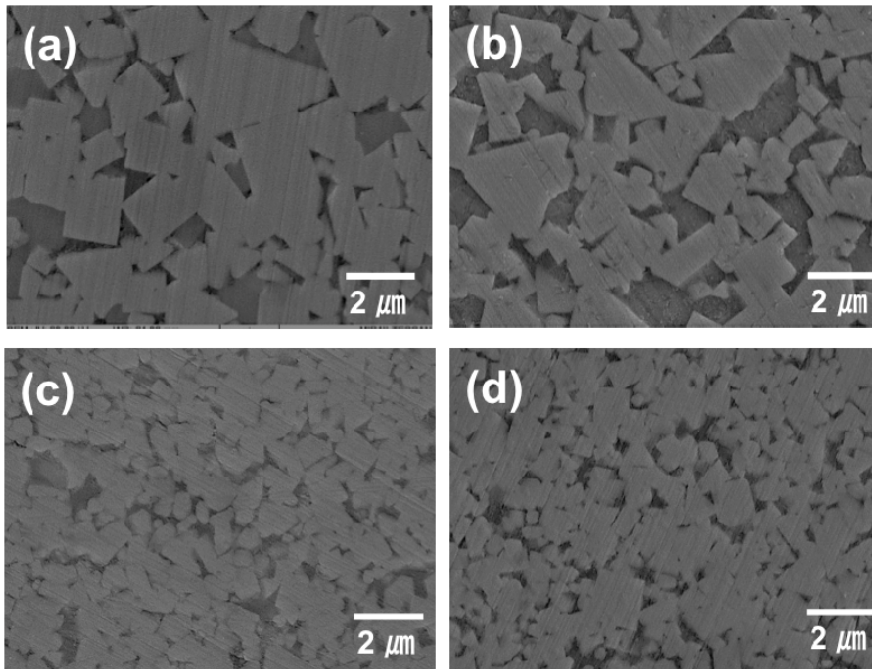


Fig. 12. Changes in microstructure of sintered samples (a) WC-7.5wt%Co at 1 GPa, (b) WC-7.5wt%Co at 3 GPa, (c) WC-12wt%Co at 1 GPa and (d) WC-12wt%Co at 3 GPa.

#### 4. Conclusion

This chapter has mainly focused on the unique advantages of using Magnetic Pulse Compaction as part of commercial material production line. Through most of the observed results, it can be said that this process adds special value to materials and alloys in properties like densification, shrinkage and other microstructural aspects. While MPC is being employed, high initial and final density, and reduced shrinkage have been observed in most of our studies mentioned throughout this chapter. This has resulted in illustrating better microstructural arrangement in many ways. The unique feature of this process also highlights the fact that compaction is performed within fraction of a second with options of



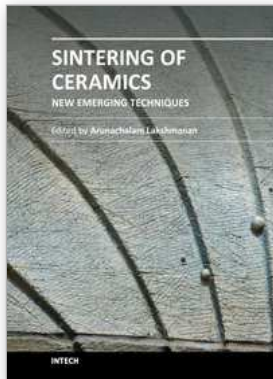
varying the pressures. The process can be carried out both at room and elevated temperatures, making it possible to employ this method on a wide range of materials.

A number of applications have already been discussed in this chapter. There are however, more fields of application that MPC can cover. For example, even transmission ring gear of AGMA 9 (American Gear Manufacturers Association) rating for automotive powertrain applications has recently found interest in using MPC. The objectives of these efforts are to replace the cast and machined gear with a lower cost net shape PM gear and to increase the power density through enhanced material properties. In addition, a large number of industrial drilling parts, made of hard materials like WC-Co or WC-Ni-Co, such as drill bits, go through an initial phase of MPC. Initial development efforts have focused on achieving the target density; developing tooling that can survive the dynamic compaction conditions, and producing the desired geometry. With a number of applications now almost ready for commercial implementation, new applications are continuously being sought and developed for the MPC process.

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## **Sintering of Ceramics - New Emerging Techniques**

Edited by Dr. Arunachalam Lakshmanan

ISBN 978-953-51-0017-1

Hard cover, 610 pages

**Publisher** InTech

**Published online** 02, March, 2012

**Published in print edition** March, 2012

The chapters covered in this book include emerging new techniques on sintering. Major experts in this field contributed to this book and presented their research. Topics covered in this publication include Spark plasma sintering, Magnetic Pulsed compaction, Low Temperature Co-fired Ceramic technology for the preparation of 3-dimesinal circuits, Microwave sintering of thermistor ceramics, Synthesis of Bio-compatible ceramics, Sintering of Rare Earth Doped Bismuth Titanate Ceramics prepared by Soft Combustion, nanostructured ceramics, alternative solid-state reaction routes yielding densified bulk ceramics and nanopowders, Sintering of intermetallic superconductors such as MgB<sub>2</sub>, impurity doping in luminescence phosphors synthesized using soft techniques, etc. Other advanced sintering techniques such as radiation thermal sintering for the manufacture of thin film solid oxide fuel cells are also described.

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Soon-Jik Hong, Md. Raihanuzzaman Rumman and Chang Kyu Rhee (2012). Effect of Magnetic Pulsed Compaction (MPC) on Sintering Behavior of Materials, Sintering of Ceramics - New Emerging Techniques, Dr. Arunachalam Lakshmanan (Ed.), ISBN: 978-953-51-0017-1, InTech, Available from:  
<http://www.intechopen.com/books/sintering-of-ceramics-new-emerging-techniques/effect-of-magnetic-pulsed-compaction-mpc-on-sintering-behavior-of-materials>

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