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Ultrahigh pressure SPS (HP-SPS) as new syntheses and exploration tool in materials science

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Abstract

Ultrahigh pressure SPS processing is demonstrated with high pressure (HP) Belt and high pressure Bridgman apparatus. These HP apparatuses are capable of working in temperature range 20°C-1800°C and the pressure ranges of 800 MPa - 6 GPa (HP-SPS-Belt) and 4 - 6 GPa (HP-SPS-Bridgman) coupled respectively with pulse electric direct current. The designed equipment can be employed for various applications in materials processing to fine tune materials properties through microstructure engineering by employing high pressure and pulse electric current. We have demonstrated the benefits of HP-SPS-Belt and HP-SPS-Bridgman in comparison to conventional SPS to obtain dense sintered compact with β - SiC as a demonstrative material at comparatively low temperatures (1300°C), duration (3 minutes) and pressure (4 GPa). The initial nanosized grains are retained in the sintered compact. The developed innovation has huge potentials for various applications for materials processing, consolidation and assembling in Materials Science.

Keywords

High pressure; Pulse electric current; HP-SPS; Belt; Toroid

1. Introduction

Spark plasma sintering (SPS) or field-assisted sintering technique (FAST) [1] is widely used for consolidation and/ or syntheses of materials with direct current (DC) pulse current activation in the presence of pressure. In the case of conductive samples, the current is transmitted to the sample by Joules heating [2], whereas in the non-conducting samples, the heat is transferred to the material by heating of the mold surrounding it. Pulse patterns

(ON:OFF) usually in milliseconds enables to reach high heating rates (1000°C/min) and rapid cooling/quenching rates of 200°C/min is possible. While using the classical graphite mold tools typical high temperatures up to 2400°C and 800°C with Tungsten Carbide (WC) molds can be reached. Simultaneously while heating, uniaxial mechanical pressure in the range of 50-250 kN can be achieved. Conventional SPS molds made of graphite limits the pressure to 120 MPa, whereas suitable alloys of composites of molybdenum, tungsten carbide (WC-Co) or Silicon carbide (SiC) allows to achieve high applied force by surpassing the limit [3]. Complex shaped molds such as double stage molds by combining graphite and WC/SiC was also employed to achieve high pressures up to 1 GPa [4]. SPS process is widely used for consolidating nanomaterials, ceramics, composites, functionally graded materials and diffusion couples for diverse applications such as optical, mechanical, thermal, electronic, electrical and biological applications.

It is well known that the sintering temperature can be decreased by applying external pressure. Mechanical properties of SPS sintered sample such as cemented carbide was considerably increased due to high pressure [5]. Shen et al [6] reported on the beneficial effect of high pressure to increase the sample density, hardness and simultaneously to limit grain growth. Pressure significantly reduces the sintering temperature which assists in limiting grain growth [7]. In the recent years, various research teams are focusing on employing high pressure and high temperature by combining pulse electric power source with high pressure Toroid (Bridgman) type and Belt type apparatus. They are successfully employed to sinter ceramics, composites and hard tools by HP-HT processes

The application of high pressure –high temperature (HP-HT) [8] sintering permit achieving simultaneously extreme high pressure (up to 10 GPa) and high temperatures during the process. Therefore, these sintering process proceeds much faster (usually in several minutes) than free sintering which usually takes few to several hours. The obtained ceramics are

characterized by degree of densification reaching nearly 100% by a degree of densification yielding isotropic properties. The use of such conditions can also reduce the diffusion between grains and prevent grains growth. Multidisciplinary expertise on the high pressure and sintering sciences lead to the development of high pressure clubbed with SPS. Currently the SPS technique employs pressures up to 500 MPa [9]. At high pressure conditions frequently phases are formed that do not appear during sintering in atmospheric pressure. The recently developed HP SPS/FAST (High Pressure Spark Plasma Sintering / Field Assisted Sintering) devices are changing the thermodynamic conditions of sintering and synthesis. In the present work, the sintering processes was carried out using High Pressure SPS/FAST methods with pressures up 6 GPa to 8 GPa using Belt and Bridgman type apparatus respectively. A conventional SPS (Dr. Sinter SPS 515 S Fuji) equipment sintering was also used for comparing with the High pressure SPS –Belt and Bridgman process. Both equipment, Bridgman type at Institute of Advanced Manufacturing Technology (IAMT) in Krakow and Belt type at Institute for Solid State Chemistry Bordeaux (ICMCB) at Pessac, are first in the world to design and work on such large volume under high pressure integrated SPS. Due to the different design, they are characterized by different sintering process conditions. Bridgman type apparatus allows for sintering at pressure up to 8 GPa at duration of 2 minutes, Belt type apparatus at pressure 6 GPa, at duration up to few hours.

In the present work, we demonstrate the beneficial aspects of HP-SPS-Belt and HP-SPS-Bridgman type apparatus employing extreme pressure conditions to obtain dense β -SiC, which is difficult to synthesize/densify by conventional SPS process (without addition of any binders). Silicon Carbide (SiC) materials are typically densified by liquid phase or solid phase mechanisms [10]. Polytype transformation of SiC occurred during SPS process, where faster heating favored 6H polytype while slower heating favored 4H for the hexagonal phase (α -SiC). To achieve full density via solid state sintering, consolidation of SiC with boron and carbon

were widely investigated. The beneficial aspects of our HP-SPS-Belt and HP-SPS-Bridgman apparatus can help in synthesizing materials with outstanding properties and favoring to obtain the densest phases [11] (example: graphite / Diamond) upon sintering. We will discuss about the densification of cubic phase SiC (β -SiC) with HP-SPS-Belt and HP-SPS-Bridgman process to demonstrate the beneficial influence of high pressure to yield high density in comparison to conventional SPS. A preliminary analyses on the pure β -SiC was studied by X-ray diffraction, density and microstructural studies. The results are presented in the following sections.

2. Experimental procedure

High pressure sintering experiments were performed using two different equipments: (i) the HP-SPS-Belt equipment at ICMCB (Bordeaux) [12] and (ii) the HP-SPS-Bridgman equipment at IAMT (Krakow) [13]. Conventional SPS experiments were done to test the influence of the pressure and pulse electric direct current on sintered compacts. For all experiments, the pressure was first increased up to the desired value; then the heating cycle was executed. The maximum temperature was maintained at dwell time for 2 or 5 min. The decompression was done after cooling. The heating and cooling rate was maintained constant in all the three different types of sintering apparatuses.

Conventional SPS

For each experiment 1 g of starting powder was filled into a graphite die having an inner diameter of 10 mm. The graphite die was covered with a thermal insulator carbon fiber to avoid contamination from the graphite die to the starting powder. An optical pyrometer was used to measure the temperature of the graphite die surface. The graphite mold was placed in SPS operating chamber (DR. SINTER LAB spark plasma sintering equipment, Model SPS-515S), capable to reach 1500 A for 10 V, heating provided by Thyristor, under vacuum of 10^{-3} Pa. The

pulse (3.3 ms) sequence for the SPS applied voltage for all the samples was 12:2 (i.e. 12 ON/2 OFF). A pressure of 100 MPa was employed for sintering.

HP-SPS-Belt

The belt apparatus is one of the most used high pressure generation apparatus. Since 2014, SPS at high pressure with the range 800 MPa- 6 GPa in a belt apparatus possessing a press of maximum force of 1000 tons with pulse electric current source was developed at ICMCB-CNRS. The originality of our work is to combine a high pressure Belt type apparatus (HP-SPS-Belt) with an electric pulse current source to carry out high pressure and high temperature experiments.

Our belt apparatus was supplied by Strecon (Denmark). The pressure vessel consists of a chamber inside a tungsten carbide die having a 32 mm inner diameter and 29 mm height cylindrical cavity. In order to perform high temperature and pressure experiment, the Belt apparatus (HP-SPS-Belt) was coupled to an electric pulse current source supplied by Fuji Electronic Industrial (SCM-3000 model) and is capable to reach 3000 A for 10 V. The operating temperature ranges between 20-1800°C is provided by direct pulse current provided by Thyristor. Unit pulse has a of 3.3 ms duration similar to a conventional SPS from Fuji to be capable to compare conventional and HP for the SPS process. A pulse sequence is defined by a given number of pulse trains followed by periods of zero current. We used a sequence of twelve pulses of total 40 ms followed by two periods of 6.6 ms of zero current. This sequence henceforth is termed 12:2. It should be noticed that such equipment is novel and is built for the first time through our work in the state of art. The electric generator can be controlled manually or automatically via a software control monitor. Experiments were generally run using automatic current control mode.

The high pressure cells contained graphite furnace with 17 mm internal diameter corresponding to the maximum sample diameter. The sintered sample had 17 mm diameter if it was in contact with the graphite furnace tube. However, in the HP-SPS-Belt apparatus, it is also possible to have 10 mm diameter, with pre-compacted pellet of hexagonal boron nitride (h-BN) ring to obtain comparable sample size with conventional SPS 10 mm diameter die. The external diameter of h-BN ring was 17 mm to match with the graphite furnace. The sample was compressed with two graphite studs similar to conventional SPS die assembly [12]. Temperature is adjusted by modification of the % of the filling of the set pulses with elementary 3.3 ms pulses.

HP-SPS- Bridgman

At IAMT, the high pressure cells are similar as that ICMCB's HP-SPS-Belt contained graphite furnace with 17 mm internal diameter corresponding to the maximum sample diameter. A 17 mm diameter precompacted (at 10 MPa by uniaxial pressing) pellet was pushed directly into the graphite furnace. The cell assembly of the toroidal apparatus corresponding was described by Knaislová et al [14]. High tonnage hydraulic press is equipped with Bridgman anvils, a direct pulse current generator and a computer control system. The maximum force that can be exerted by the press is 2500 tons, with the pressure range of 4-8 GPa. The operating temperature ranges between 20-2400°C is provided by direct pulse current provided by high frequency inverter 1 kHz produced by Harm & Wende paired with transformer capable of DC pulse output of 8.3 V and with DC pulse characteristics of 40 ms impulse length and 20 ms impulse intervals. Temperature is adjusted by modification of the % of the filling of the set pulses with elementary 1 ms pulses.

Characterization analyses

Densities of sintered samples were measured by Archimedes' method with water as reference liquid. The microstructure of sintered samples were observed on the fractured surfaces using a JEOL JSM 6700F operating at 15 kV. SEM images allowed to estimate the grain size as well as the grain morphology. Sintered samples were milled and characterized by powder X-ray diffraction (XRD). XRD was performed on milled samples using a PANalytical X'pert Pro MPD diffractometer with Cu K α radiation and Bragg–Brentano geometry.

3. Results and discussion

3.1 High pressure equipment- A retrospective view

Kinetics governing solid transformation under high pressure can lead to structural phase transitions, diffusion or chemical synthesis. The high pressure phase transitions can be directly characterized with the help of *in situ* Diamond anvil cells for example. When large volume cells are involved, then the phase transformation is usually studied *ex situ*. An important application of high pressure is sintering of composites such as CERMET [15] and achieving very high density. One of the best example for the same is polycrystalline diamond compacts. The piston-cylinder [16] technology is an obvious method to generate pressure. Most often, the sample is compressed in a cylinder by a movable piston. The piston is pushed by adjustable hydraulic pump in most situations. Different other morphologies such as rectangular, oblong can be adapted [17] to piston and die depending on the experimental needs.

A piston-cylinder cell can be made from different materials [18]. The maximum pressure indeed depends on the compressive yield strength of the used material. Tungsten carbide, specific steels or alloys are often used to reach high pressure up to 5 GPa [19]. The temperature can be produced through different ways. Resistive heating - with heating sleeve or directly in the cylinder - is mostly used. New temperature generation technology such as inductive or microwave are being developed. One advantage of piston-cylinder technology is

its versatility. It can operate in liquid or solid media and can be coupled to other sealing or temperature technologies.

Piston must fit with cylinder dimension to prevent sample seepage which could likely cause damageable frictions. The pressure measurement in such device is quite easy as it depends on the load and the piston diameter. Most of designs include supporting materials to strength cylinder. Different version of high pressure piston-cylinder device consists of supported pistons [20]. In such case, the pistons load a deformable material which contains the sample. The deformable material serves as pressure transmitting medium (PTM). In such configuration a container is often used to retain the expansion of the PTM and to get high pressure.

The design of piston-cylinder apparatus were performed to lead to opposed anvil system in which the sample is compressed by two supported tapered pistons. The Bridgman cell (1952) was the first opposed anvils type. Flat tungsten carbide opposed anvils are pressed together to apply pressure to the sample. The development of diamond anvil cell (DAC) following this initial idea give rise to pressure values ranging up to 400 GPa [20]. DACs are mainly used in fundamental research. The temperature generation can consist of the resistive heating for all devices. Electric current assisted Joule heating is widely used through heating rings. Joule effects can also be generated in the sample container or in the sample itself. The use of pulse current has been developed since 1930's [21]. Number of advantages of pulsed current has been underlined [22].

3.2 Origin of the present work at ICMCB on high pressure- SPS-Belt

This work was done by two teams, who are experts in high pressure, one in France at ICMCB and the other in Poland at IAMT, presents the benefit of high pressure in the SPS process to achieve new materials with high performance. High Pressure research activities

initiated in the 60's by Prof G Demazeau at University of Bordeaux focuses mainly on material chemistry firstly and later in biosciences allowed technological development in solid and fluid state in ICMCB, research institute of CNRS [23].

High pressure equipment devoted to chemical synthesis were mastered under solid phase and fluid phase with gas or liquid as well as ambient at high temperature. Such equipment employed in the case of solid phase such as Belt type apparatus by the design of CEA "Atomic Energy Commission [24];

In 2010, High Pressure's department at ICMCB initiated the Spark Plasma Sintering (SPS) activities by the acquisition of the 515-S equipment of FUJI in Japan, to develop in particular the fabrication of transparent ceramics in comparison with the single crystal growth under hydrothermal conditions [25, 26].

The competences acquired under high pressure technology and SPS knowledge led to the new concept of HP-SPS consisting of conventional HP-HT equipment (heating by direct current) with a pulse electrical generator and not with a direct current generator. The HP cell was slightly modified to drive the current inside the sample similarly to the SPS's mold in the conventional SPS [12]. This pulse electrical current manufactured also by FUJI allows a perfect comparison with the conventional SPS 515-S from FUJI based on the same electronic system to generate the pulsed electric current and to pilot the ON/OFF regulation.

Following this technical evolution, at Krakow, the institute IAMT developed an HP-SPS equipment based on the toroidal apparatus with a homemade pulsed electrical generator [14].

The interest of HP-SPS equipment clubbed with Belt and Bridgman is demonstrated in this work based on the materials such as SiC in comparison with conventional SPS by the effect

of high pressure. The application of pressure (as "driving force") manifest itself in one of the following manners as,

1. to reduce the sintering temperature in order to :

1.a- avoid grain growth (which occurs at high temperature),

1.b- preserve a structural phase existing only at low temperature (ex: α -quartz SiO_2 which presents structural transformation at 573°C)

1.c- allow the sintering of thermally unstable compositions containing OH and H_2O or other volatile elements in it, to be preserved in the final material (ex: Hydroxyapatite),

1.d- allow the sintering of thermally unstable composition, which undergoes thermal decomposition (ex: BP which decomposes at 1130°C and impossible to sinter under this temperature even with conventional SPS but we successes at temperature above 1300°C by HP-SPS-Belt at 3GPa without any decomposition of boron phosphide)

2- to promote a stable structural phase at high pressure (ex: c-C (diamond), c-BN, c- Si_3N_4 , ...),

3. to induce chemical reactivity for obtaining phases with the highest density (ex: c-C (diamond) instead of h-C (graphite))

4- to allow the sintering of refractory materials reaching a density close to 100% (ex: boride, carbide, nitride),

5- to initiate a new microstructure finer than the grain size of the initial precursor powder with the help of polymorphs of the material (ex: α - Al_2O_3 from γ - Al_2O_3 [27])

3.3 Influence of direct electric current pulse on sintering in conjunction with high pressure

The interest of using electric current [28] in SPS lies in that an intense electric current is allowed to flow directly through the sample. The passage of current through the sample are

associated to cause one or many of the following processes such as Joule's heating, electromigration, electro-plasticity, Peltier effect, electromagnetic effect, interfacial reactions, electro-transport and polarization. Pulse DC current with a frequency between 10^2 to 10^3 Hz is conventionally used in standard SPS equipment. There are instances of using AC currents in SPS, but their influence on sintering and microstructures were quite similar to that of DC current. To date, there are few impediments to SPS technology in terms of scaling up, such as inhomogeneity in maintaining homogeneous temperature in the samples and power limitation of 25 – 30 kA, which in turn limits the upper sample dimension to ~ 40 cm and complex shapes. Further threshold for pressure is also another aspect that is interdependent on the type of mold which is used.

SPS technique involves heating by impulse current source. The minimization of the grain growth deriving from the high heating rates and fast sintering cycle enhances the effect of pressure. At elevated pressures, the densification of nanopowders can be accomplished mostly by instantaneous plastic deformation or particle deformation or particle rearrangement, reducing to a minimum the role of the classical sintering mechanisms such as bulk diffusion or grain boundary diffusion. This probably explains why in most cases, SPS densification is achieved in just few minutes even at relatively low temperatures in all kinds of materials.

As described above, the fabricating condition at very high pressure method favors densifying with less sintering time and at low sintering temperature. Grasso et al [9] have shown using graphite sample that with increase in pressure, the maximum peak temperature and radial temperature gradient across a sample during SPS progressively decreases with increasing pressure due to tighter (die/punch) contact at higher pressure leading to higher pressure leading to higher conductivity. External pressure has a strong influence on the overall temperature distribution on the punch/die/sample assembly. The ceramic's density and micro-hardness can

be controlled by the sintering condition such as sintering time, sintering pressure, sintering temperature.

3.4 Densification of β -SiC

Silicon Carbide (SiC) materials are typically densified by liquid phase or solid phase mechanisms [10]. Nano-sized SiC powders could be densified at 1700-2200°C without additives under an applied pressure of 40-70 MPa. In contrast, limited densification of nano-sized SiC powders was reported [29] when they were spark plasma sintered at 1850- 1950°C under a higher applied pressure (130 MPa). Dense β -SiC ceramics (relative density = 96%) could be manufactured at an extremely high heating rate and with a short processing time (17 seconds) by flash SPS. Application of external load causes intensification of the consolidation processes and among others, simplifies the elimination process of pores at the final stage of sintering. Among techniques limiting grain growth and accelerating consolidation are sintering under pressure (Hot isostatic pressure or High pressure High temperature) or microwave sintering or by using current impulses.

Traditional methods of producing SiC based ceramics include loose sintering, reaction sintering and hot pressing or pressure sintering. These methods so far fail to sinter ceramics of pure SiC to a density close to the theoretical value. To achieve higher density, the original SiC powder is supplemented with additives sintering a liquid phase during sintering. Additives generally include MgO, Al₂O₃, Y₂O₃, AlN or their mixtures. However, a low melting component significantly limits an operating temperature range for such ceramics. Maximum operating temperatures of known SiC based composites do not exceed 1500°C. There are earlier reports on SPS for densification of SiC, but full density was never achieved.

Plastic deformation can be neglected during SiC sintering, as this material is characterized by strong ionic-covalent bond of carbon and silicon [30] atoms that explains high

values of the dislocation glide activation energy. Sintering acceleration usually occurs under pressure starting with 1 GPa. There are many reports [31] on the decomposition of SiC to Si+C at low pressure. SiC decomposed up to 8 GPa based on quench texture and composition. Incongruent melting was also observed up to 10 GPa in SiC.

In the present work, by using very high pressure sintering (HP-SPS-Belt and HP-SPS-Bridgman) method, β -SiC ceramics were sintered at much lower sintering temperature (1300°C), with much less sintering duration (< 30 minutes). While traditional sintering methods sintering temperature is much higher (> 1850°C) and sintering time is much longer (> 1 h).

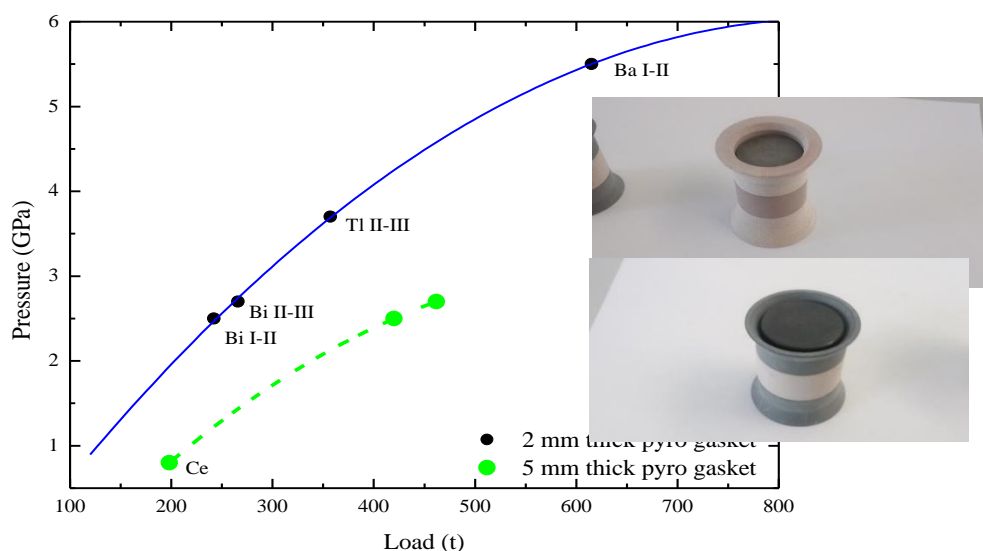


Figure 1. Pressure calibration for low threshold limit of 800 MPa with HP-SPS-Belt with top inset photo: low pressure design; bottom inset photo: high pressure design

For Belt type and Bridgman type apparatus pressure and temperature should be calibrated first. The temperature was calibrated under different pressure values and the pressure was calibrated at ambient temperature. The pressure was calibrated using the pressure induced transition of electrical resistivity of pressure calibrants such as Bi, Ba and Tl. The temperature was calibrated using graphite heater tube. The studied sample is usually surrounded by a pressure transmitting medium (PTM), which can be insulator or conductor in the HP cell. The

versatility of HP-SPS-Belt allows to work at Pressure starting in the range 800 MPa – 6 GPa (Calibration curve represented in Figure 1 not), which can allow to compare with the conventional SPS equipment's experiment. The detailed procedure on the pressure and temperature calibration can be found in Balima et al [12]. A specific HP cell has been designed in the range 800 MPa – 2 GPa which was impossible to reach by the HP cell designed in the article of Balima et al [12]. The pressure calibration by using metal such as pressure calibrant at 800 MPa with Cerium and higher with Bismuth, like explained in this article.

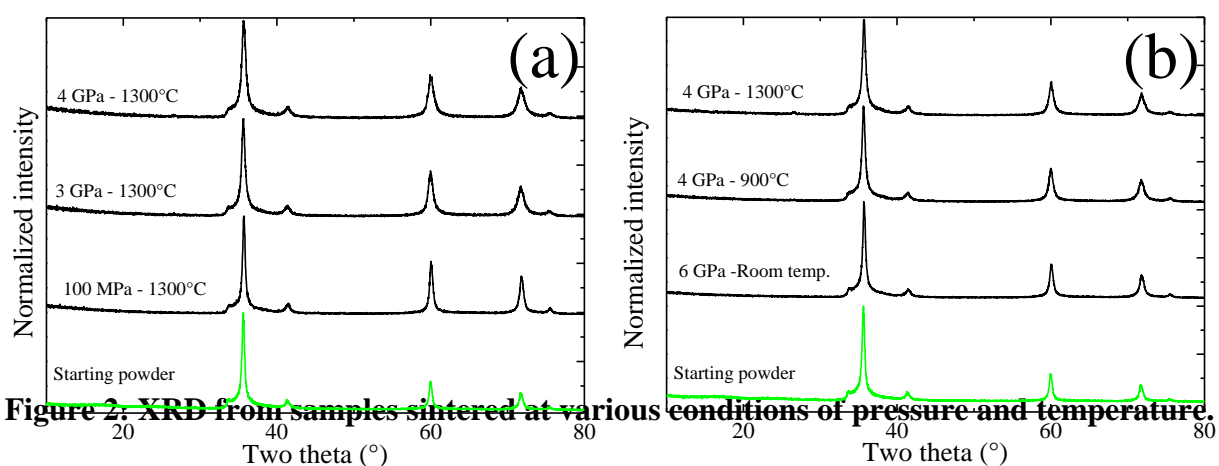


Figure 2. XRD from samples sintered at various conditions of pressure and temperature. Experiments were performed (a) at ICMCB and (b) at IAMT.

It can be observed from Figure 2 that there is no phase transitions or decompositions induced due to the pressure and temperature in neither of the HP-SPS-Belt and HP-SPS-Bridgman equipment. The initial chemical composition is retained in the sintered compact. From the X-ray diffraction of the samples after sintering, it was observed that there is no transformation from the β -SiC, cubic phase, irrespective of the temperature used in the given pressure range. The highest density (~ 85-90%) among the samples were obtained for a pressure of 3 GPa and 4 GPa in both the Belt and the Bridgman SPS equipment. Whereas the conventional SPS lead to feeble densities (< 55%) obtained at 100 MPa. We found that as the pressure increased, the density increases (Figure 3), comparison done for the same temperature

1300°C during 10 min (blue triangle). There was no particular differences in the trend of density variation based on the HP-SPS-Belt and HP-SPS-Bridgman equipment. The driving force provided by the high pressure may have increased the density. The density of the samples were observed to be correlated between the temperature and pressure. With the increase of pressure and temperature, the density of SiC increases

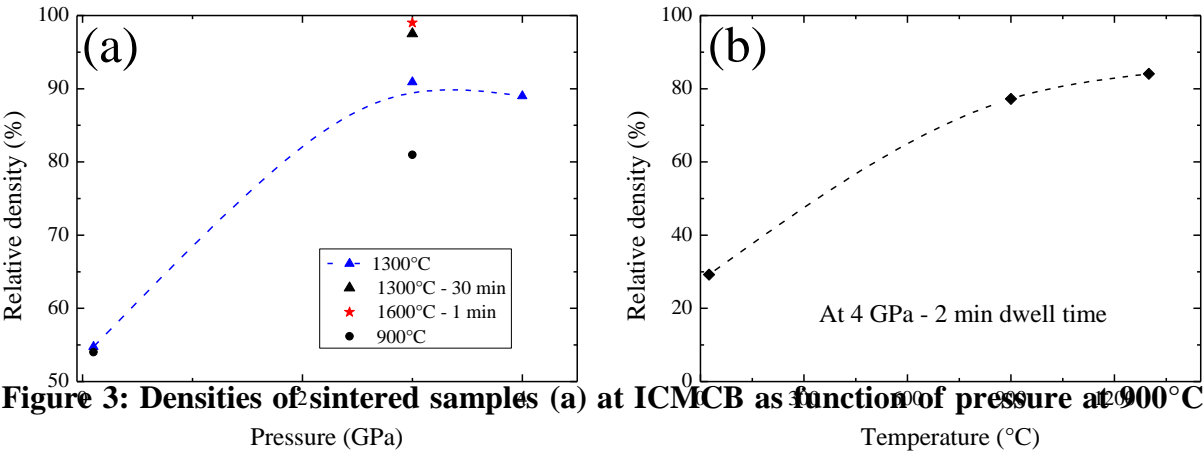


Figure 3: Densities of sintered samples (a) at ICMCB as function of pressure at 900°C, 1300°C and (b) at IAMT as function of the temperature at 4 GPa.

Logically, the density increases if time increases from 10 min (blue triangle) to 30 min (black triangle). The density increases if the temperature at 1600°C even for 1 min (red star) and decreases for temperature at 900°C (black star).

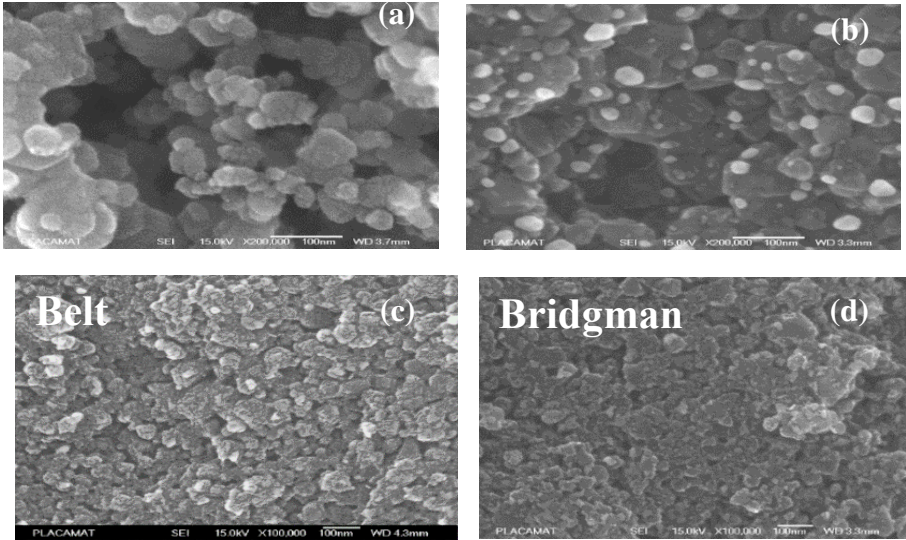


Figure 4: Microstructures of (a) Starting powder (b) Sintered compact by HP-SPS-beltat 900°C (c) 1300°C – 4GPa – 5 min by -HP-SPS-Belt (d) 1300°C – 4GPa – 5 min by HP-SPS-Bridgman

By using the HP-SPS-Belt/ Bridgman processes, pure β -SiC was sintered to high density (90%). From the microstructure (Figures 4-6) it can be observed that the grain size was retained to a nano-scale and the microstructure shows that no porosity is present and many nano-grains are sintering through neck formation between the particles. The microstructure shows solid state sintering structure.

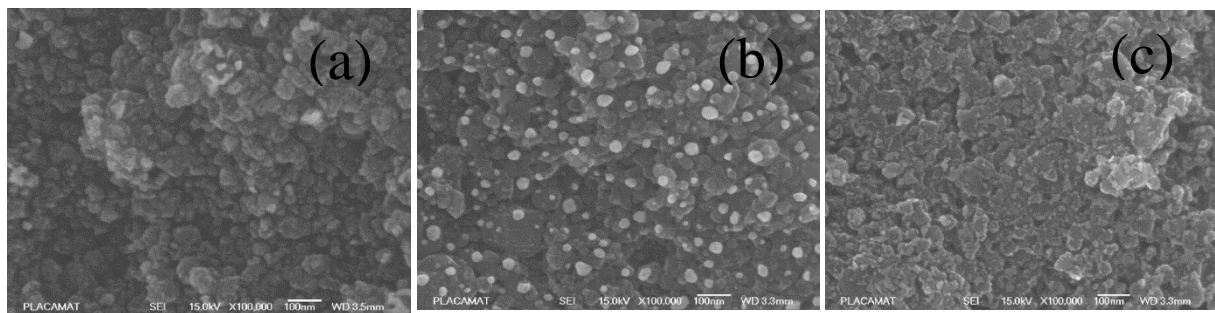


Figure 5: Microstructures for 4 GPa HP-SPS-Belt (a) ambient temperature (b) 900°C (c) 1300°C

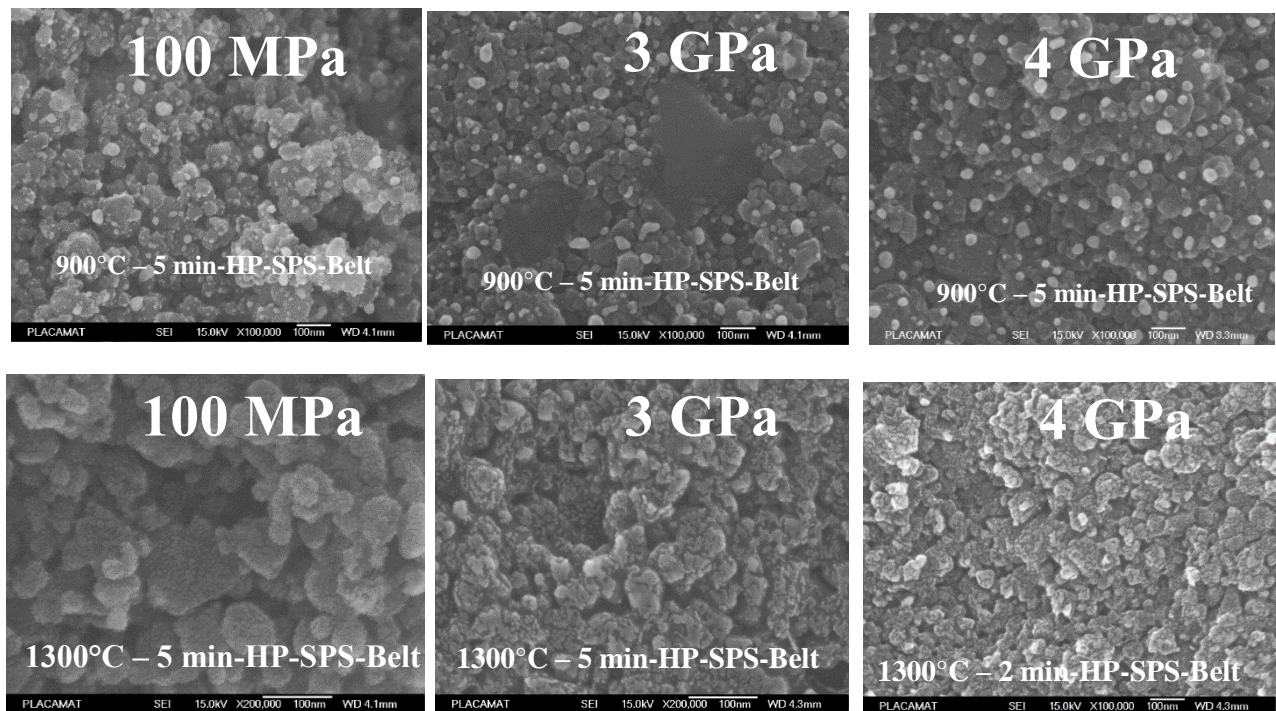


Figure 6. Microstructure of HP-SPS-Belt at different pressure and dwell times

There is no increase in grain size during the sintering process and all the crystal structure of SiC remained in the cubic structural form. With the increase of dwell time, the density of the sample increases and this can be attributed to the reduction of oxide layer, which could have thus promoted diffusion. There was a slight increase in the grain size and density with the increasing dwell time.

4. Conclusion

The goal is to optimize (control) high pressure sintering process to obtain highly densified materials with phase composition as close as possible to starting composition. The employment of low temperature, high energy current pulses and short sintering duration should allow to reach set goal. Pressure aided sintering methods allow to obtain dense materials with lower restrictions in regard to powders. Spark Plasma Sintering (SPS) one of those methods. The simultaneous application of pressure and pulsed current additionally to high temperature zones also results in higher heating rates, shortening of the sintering process duration and temperature resulting in consolidation of powders without excessive grain growth. SPS method also does not require preliminary powders densification. The powder is introduced directly into the graphite die, the pressure and electrical current flow is applied, which results in obtaining of nearly fully densified material with exceptional mechanical properties. At high pressure conditions frequently phases are formed that do not appear during sintering in atmospheric pressure. The recently developed HP SPS/FAST (High Pressure Spark Plasma Sintering / Field Assisted Sintering) devices are changing the thermodynamic conditions of sintering and synthesis with pressure up to 8 GPa (using Belt and Bridgman type anvil apparatus). Both devices, Bridgman type at IAMT in Krakow and Belt type at ICMCB in Pessac, are first in the

world high pressure SPS devices. Due the different design they are characterized by different sintering process conditions. Bridgman type apparatus allows for sintering at pressure up to 8 GPa at duration 2 minutes, BELT type apparatus at pressure 6 GPa, at duration up to 30 minutes. In the present work, though the heating modes of electric pulse generators (Thyristor and inverter) were different, no significant changes were obtained in the densification kinetics of SiC. High pressure sintering both by HP-SPS-Belt and HP-SPS-Bridgman yielded similar results on density and microstructures for obtaining dense phase β -SiC. The current technology can be successfully employed to obtain the densest phase and extended to many thermosensitive/phase transition prone materials. It can be observed that the combined effect of high pressure technology and SPS process restricting diffusion and prevents growth of grains in the sintered materials. In the present work, even at short sintering time of 2-5 minutes, 90% density was obtained for β -SiC, further investigations and study of sintering parameters such as dwell time and sintering temperature could lead to 100% density without addition of any binders/ additives. The aforesaid studies are in progress and will be reported as article elsewhere.

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