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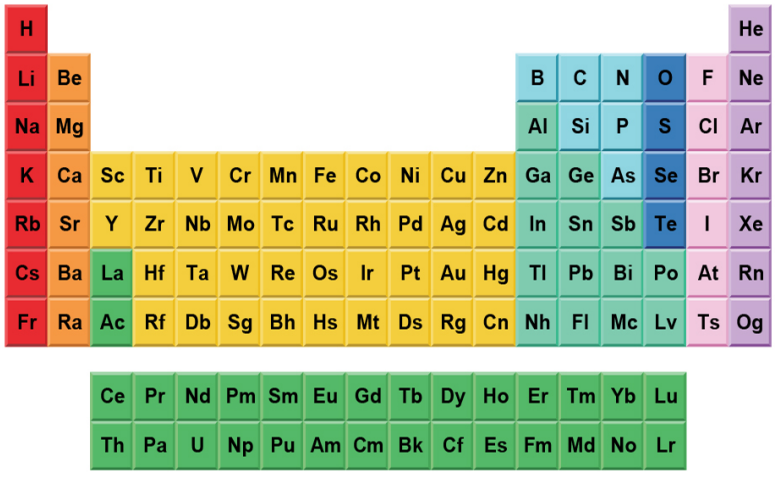
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Review of Spark Plasma Sintering Process

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Abstract. This paper reviews the spark plasma sintering process. The spark plasma sintering (SPS) technique is a sintering technique in which the plasma is produced when the sintering is conducted. This plasma is used for the sintering of the powders. This paper deals with the various variables with effect the plasma sintering process. First the effect of pressure on SPS Process and its advantages. The review also highlights the research conducted by researchers on various materials and various sintering pressures. The effect of heat, the electro plastic effect was considered during the SPS Process review. The conventional sintering process is also considered in this review paper.
Key words: Sintering, Plasma, Joule heating

1. Introduction

Sintering is a process in which materials such as metals and ceramics are compacted forming powder materials into solid state objects by the application of heat and pressure at temperatures bellow their melting point. The bonding of sintered particles together is by atomic transport mechanics which results in the growth of particles which decreases the porosity of the solid state object [1-3]. The sintering process is the reduction of system enthalpy due to the reduction of specific enthalpy. The factors which affects the sintering process can be categorized into two categorizes [4]. The first is the “Process Variables” which contains all parameters that will be affected by a change of sintering process (sintering temperature, furnace atmosphere etc.). The second category is known as “Material Variables” (geometrical structure of particles, their size and size distribution) [5-6]. The structure of particles and the size distribution of the particles affect the sintering process. Experimentally it is found that the particle size affects the sintering kinetics. i.e., the neck forming areas between the grains [7-8].Mathematical models of sintering process is based on the joining of two spherical particles [9-10].Metal particles can be used for production of round or spherical particles. Mono sized mass production of micro particles are produced by pulsated orifice ejection.

2. Objective of the review paper

The objective of this paper is to review the spark plasma sintering process and to understand the various process parameters which affect the spark plasma sintering process. This paper also helps us to find out the research gap in the spark plasma sintering of nanomaterials.

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3. Methods of powder compaction

The sintering methods followed gives the process variables. The sintering methods are divided into two groups. In the first group technique, the samples are produced without applied pressure. In the second group technique, the powder is compacted by applying pressure in the order of MPa. Each of these techniques are further divided according to sintering temperature [12-14].

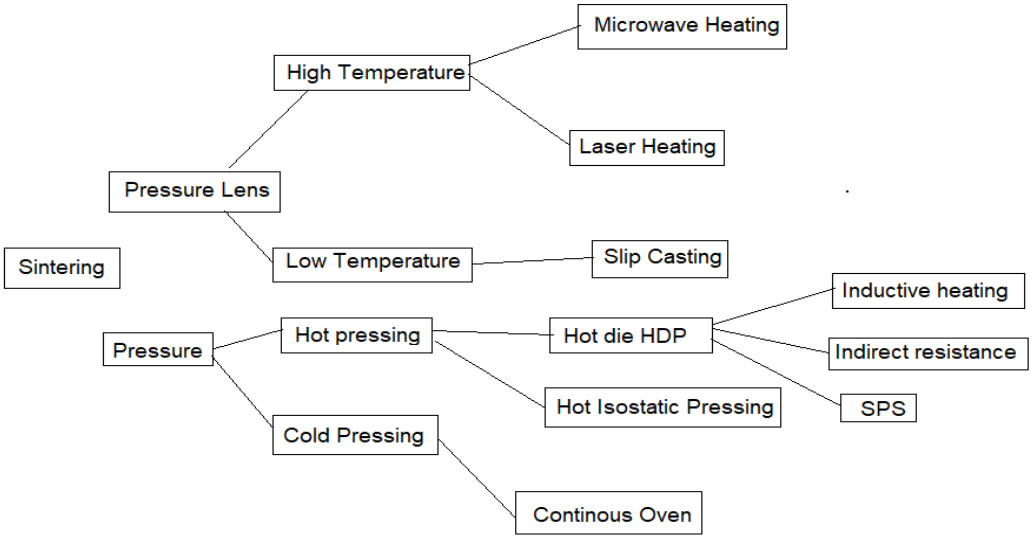


Figure: 1 Standard classification of sintered process

3.1. Pressure less Sintering

The figure 1 shows the standard classification of sintered process. The pressure less sintering is a compacting process in which pressure is not applied (except gravity). In this PLS fine grained starting powder (<3 μm) and additives are used for high compacting density [15].

3.2 Slip-casting

Slip casting is commonly used for mass producing complex shapes and identical ceramic parts [16].

3.3 Pressure less/high temperature sintering

High temperature furnace is used for high temperature sintering. The metal or ceramic is mechanically vibrated in a mould. The mould is heated using microwave heating source [17] [18]. The mathematical model predicts faster rate of grain-growth during pressure less sintering than during HIP [19].

3.4 Pressure-Assisted Sintering

Nano-micro powder of dense ceramic or metal parts are pressed and heated by pressure assisted methods. The powder is placed between punches at high sintering temperature and long sintering time and it results in grain growth [20]. HP process of WC grains grew from 50 nm to 500 nm [21].

3.5 Cold Pressing

The mechanical and hydrostatic pressure applied to the mould filled with powder is formed into the required shape. Application of pressure on the mould bonds the powder particles together (green body) so that it can be handled during successive high temperature sintering process. The density of green body material is 95%.High temperatures are obtained for nonconductive materials. Heating is slow in indirect resistance heating. Higher heating rate is achieved because only the powder is heated (not punches and dies). Simple shapes can be produced by this process.

3.6 Hot Pressing (HP)

In hot pressing, the pressure is used to densify the material, temperature is required in this type of process [22]. The grain growth is slow in HP. It is used for producing simple shapes and the tolerance is achieved by diamond grinding [23]. Hot pressing technologies are divided into hot isostatic pressing, induction heating, indirect resistance heating and FAST ISPS direct hot pressing [24].

3.7 Hot Die Pressing

Pressure and heat are applied together at the same time on the sample in hot die pressing [25-27]. Simple shapes can be formed from very hard and brittle materials such as diamond metal composites, cutting tools and technical ceramics [28]. The mould material used in this process allows heating up to 2500° C.

3.8 Indirect Resistance Heating

The mould is kept inside in a heating chamber and it is heated by graphite heating elements and it generates heat with the help of joule heating [29]. This is transferred to the die by heat connection. High temperatures are obtained for nonconductive materials. Heating is slow in indirect resistance heating [30-33].

3.9 Inductive heating

High frequency electric field by an induction coil and HF power source is used to produce heat in inductive heating. The pressure and induced current are totally independent of each other and the heating is very fast. The electric field can generate between 0.5 mm to 3 mm. The transfer takes place by thermal conductivity [34-35].

4. Spark Plasma Sintering Technique (SPS)

Classical sintering technique results in slow heating rate 5-10° C and substantial particle growth [36]. Due to the above disadvantage in classical sintering technique a new spark plasma sintering technique was developed [37]. It is also known as Field Assisted Sintering Technology (FAST), Plasma Activated Sintering (PAS) and Electric Current Activated/ Assisted Sintering (ECAS) [38]. Sumitomo Heavy Industries Ltd in 1990 produced the first commercial SPS machine [39].Heating rate of 1000° C/min is achieved depending on the geometry of die, punches and power supply. The advantages of SPS are low sintering temperature short holding time, no pre-compaction and shorter sintering time. Nanometric powder can be sintered to lean theoretical density. SPS process can produce only simple shapes [40].

4.1 Basic SPS Configuration

The SPS system consists of vertical single pressurization axis. The pressure is transferred to the ram of the cylinder, which is also used as electrodes. The sintered powder is stacked in a graphite die, which placed between graphite spacer. The upper space is attached to the water cooled upper electrode which is mobile. The lower water cooled lower electrode is fixed. The

whole setup is placed in a vacuum chamber. DC power source is given to the unit. The electric current is non pulsed DC or pulse DC (PDC). Plasma generation and pulse current effect [41]
The ON-OFF DC pulsing creates joule heating point and formation of spark discharges is formed in the gaps between particle surfaces. The temperature of 1000º C is created by spark discharges. The vaporization and melting surface of both particles of spark area. The necks are formed in the area of contact between particles. The frequency is responsible for homogeneous temperature distribution through sintering powder the heat is generated during a pulse on the surface of the particle is conducted to the volume of the sintered powder during the "Pulse off time" [42].

4.2 Electro Plastic Effect

The electro plastic effect can happen only on metals, because the valence electrons of metal atoms have ionization energy. Valence electrons in metals are in solid state and are free to leave the atom. Electric field moves these valence electrons. Metal powders exhibit lower yield strength under an electric field [43]. The research gap is microscopic mechanism of electro plastic have to be analyzed. The electro plastic effect in metals is the defining of dislocations from the paramagnetic obstacles by the magnetic field induced by the electric current [44]. The model shows that an electrostatic field applied during plastic deformation of metals reduced.
Flow stress retarded cavitation [45] and grain growth. The third model explains that the drift electrons can generate a force on dislocations and affect the plastic flow of metals.

4.3 Joule Heating

Joule heating is a form of resistive heating; it is the process by which the passage of an electric current through a conductor releases heat. The amount of heat Q is given by equation1:
Joule-lens law,

$$Q=I^2_{rms}.R.t$$

The resistance between two particles depends on their geometry. From the literature it is found that resistance is indirectly proportional to the size of contact area of two particles and to-the cross section of the particles [46]. When the contact size of two particles is smaller than the cross-section, more heat is generated on the border of the particles. This is one of the reason for mechanism of neck growing in an early sintering step.

4.4 The main benefits of SPS are:

- Effect of the current flow
- Effect of DC pulsing
- Effect of rapid heating
- The effect of pressure

4.4.1 Effect of the current

SPS has high heating rate, due to joule heating from the current floating through punches, sintered powder and through the die [47]. In addition to joule heating which produces joule heat [48]. Heat generation is also obtained by generation of plasma between sintered particles [49]. The plasma generation is due to a source localized heating between two particles. The researcher published the influence of plasma effect [50]. The researcher measured the current passing through sintered Al_2O_3 powder and found that at a temperature 1000º C [51].
The current was around 100 mA, and they concluded that no current flowed through alumina powder, and no discharge generation took place between two non-contacting samples [52]. The researcher in his experimental work found that sintering of pure Al with pulsed frequency 40 KHz was used and found that no discharge presence during a sintering cycle [50]. The researcher conducted experiment on copper spherical powder (550 μm) placed in a non-conducting die and pressed by copper punches [53-56]. They found observable discharges alone

from the current density exceeded 100 A.mm-² at the pressure of 6.9 MPa [57]. The researcher discussed the absence of plasma in spark plasma sintering and also found that there was no plasma or spark present during the sintering process [58]. The various powders in the experiment conducted by the researcher is given in the below table 1[59].

Table: 1 Powders used in sintering Process

Powder	**Temperature**	**Atmosphere**
None	650	Vacuum
Al	500	Vacuum
Al_2O_3	650	Vacuum
NaCl	650	Vacuum
Mg	450	Vacuum
Zn	225	Vacuum
Zn	225	Ar

4.4.2 Role of DC pushing

Researchers have published the effect of pulse length and frequency on material properties of sintered samples. The effect of pulse current during spark plasma sintering is clearly seen on the reactive alumina-hernatite powders. An reactive spark plasma sintering process produce material with surface layer composition and microstructure different to that one of the cone. An pulse current influence on a change to surface microstructure, surface position to a cone sample. Average composite layer thickness was about 38 μm for pulse ratio 12:2 and 48 μm for pulses 2.2 and 2.6. The bottom thickness was 32 μm for all samples. It is observed from the samples where upper and bottom sites have different thickness of the same composition, and it is described as cationic migration on densification, mechanical and tribological properties of ZrB_2 by SPS[58]. Effect of pulse current investigated ZrB_2 ceramic powder was sintered and the influence of current pulse on its densification, mechanical and tribological properties [59]. It is concluded that the maximum relative density (98.65% measured by the Archimedes method) is achieved by 50 ms pulse on and 50ms pulse off. The minimal density was 97.41% and the Vickers hardness was 15.32±1.29 Gpa. The relative density of a carbon fiber-reinforced aluminum matrix (Al-CF) composite fabricated by SPS in various pulse conditions have been analyzed it is found 500 μm-long CF is the highest relative density obtained.

4.4.3. The heating rate effect

Investigated the influence of SPS heating rate on the synthesis reaction of Tantalum diboride investigated and found that the heating rate on grain growth and densification of tantalum diboride, the SPS technique was used to realize reactive sintering process powders, the final product is TaB_2. The table 2 shows the influence of different heating rate on the reaction of Ta+2B mixture obtained at 2200° C during 5 min.

Table: 2 Influence of different heating rate on the reaction of Ta+2B mixture

Heating Rate ° C/min	**Apparent Density g/cm³**	**Relative Density %**	**Young's Modulus GPa**	**Hardness Gpa**	**Grain Size (μm)**
50	11.74±0.03	93%	577±10	17.4±0.4	3.8±0.3
100	11.70 ±0.06	93%	599±10	20.5±0.9	4.2±0.5
200	11.96±0.03	95%	571±13	20.7±0.8	3.8±0.1
300	11.49±0.03	91%	532±9	20.6±0.7	4.0±0.2
400	11.77±0.03	93%	571±10	19.1±0.2	4.0±0.2

The XRD patterns for all the samples of MgB_2 were the same in his work on influence of heating rate on superconducting characteristics of MgB_2, the XRD of the MgB_2 as the main phase and two secondary phases MgO and MgB_4, it is found that higher heating rate at 355 and 475° C/min promoted formation of a lower amount of secondary phases. The SEM analysis samples at 2°C/min should straight grain boundaries and samples sintered at 355 and 475° C/min grain

boundaries should round shaped edges. The effect of heating rate on densification and grain growth during field assisted sintering as α-Al_2O_3 and $MoSl_2$ powders. The research was carried out on two types of sintered samples by different heating rates from 50° C/min and 700° C/min. $MoSl_2$ powder was α-Al_2O_3 was insulating he found that there was no effect on the final density of samples.

4.4.4. Effect of Pressure

Sintered powders are heated by floating current and higher densification by uniaxial pressure applied simultaneously to the heating. The advantage of pressure is heating direct mechanical effect on particle rearrangement and destruction of agglomerates. The pressure is also a driving force for sintering it decreased temperature and it also had a limitation of grain growth. Effect of pressure and nano-boron carbide addition on microstructure and mechanical properties on TaC and TaC- 1 wt% BoC were consolidated by SPS at 1850°C and varying pressure on the densification and grain size. The density increased with the pressure and 100% density was achieved for 255MPa and 363MPa for TaC [60]. The sintering pressure 100MPa had little impact on particle size growth which with the particle growth was significant under high pressure was concluded in his work on observation and control of done formation in SPS synthesized Alumina.

5. Conclusion

Research gap in this review is to identify the process parameters involved in the manufacturing of sintered samples. It is found from this research, process parameters affect the sintered process. The research gap is microscopic mechanism of electro plastic have to be analyzed. The electro plastic effect in metals is the defining of dislocations from the paramagnetic obstacles by the magnetic field induced by the electric current. When dislocations are more the electroplastic effect is less. Finding optimal parameters for the plasma arc sintering process is the key to the strong sintered products. Joule heating is a form of resistive heating. The resistance between two particles depends on their geometry. The necking between two particles have to be reduced minimize heat in the border of the particles. Effect of the current apart from joule heating the effect of plasma heating should also be considered. The research gap in the effect of current is the amount of current to be passed to the nano-powders has to be optimized.

The effect of pulse length and frequency on material properties of sintered samples; the surface roughness of the material has to be maintained. The heating rate effect has the heating rate on grain growth and densification of metals. The effect of pressure is the heating direct mechanical effect on particle rearrangement and destruction of agglomerates. The research gap is the particle rearrangement and how it affects the strength of the material; and how to increase the strength of the materials.

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