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Application and Mechanism of SPS(Spark Plasma Sintering) Systems and Technology

Young-Soon Kwon
School of Materials and Metallurgical Engineering, University of Ulsan

<Abstract>

Spark Plasma Sintering (SPS) is a newly developed process sintering - a synthesis and processing technique - which makes possible sintering and sinter-bonding at low temperatures and short periods by charging the intervals between powder particles with electrical energy and effectively applying a high temperature spark plasma generated momentarily. It is regarded as a rapid sintering method, using self-heating action from inside the powder, similar to self-propagating high-temperature synthesis (SHS) and microwave sintering. SPS system offer many advantages over conventional system using hot press (HP) sintering, hot isostatic pressing (HIP) or atmospheric furnaces, including ease of operation and accurate control of sintering energy as well as high sintering speed, high reproducibility, safety and reliability. This paper reviews the application and mechanism of SPS process is expected to find increased use in the fabrication of porous media with high strength, which are difficult to sinter by conventional sintering methods.

1. Introduction

The SPS process is a unique synthesis and processing technique which makes possible sintering and sintering-bonding at low temperatures and short periods by charging the intervals between particles of the sample with electrical energy and effectively applying the high energy of spark plasma generated momentarily. It is regarded as an entirely new sintering process which makes use of the self-heating action from inside the powder sample, similar to the self-propagating high temperature synthesis (SHS) technique. The SPS process features a very high thermal efficiency because of the direct heating by the spark, and it can easily produce a homogeneous, high-quality sintered product because of the uniform heating made possible by

.spersing the spark points [1].

The SPS process is an electrical sintering technique which applies an ON-OFF DC pulse voltage generated by a special powder generator to the sample of compacted particles and, in addition to the factors promoting sintering described above, this also effectively utilizes the self-heating action caused by spark discharges between particles of powder occurring in the initial stage of the pulse energizing for sintering.

In the SPS process, the powder particle surfaces are more easily activated than in conventional electrical sintering processes and material transfers at both the micro and macro levels are promoted, so a high-quality sintered component is obtained using a lower temperature and in a shorter time than with conventional processes. The system us designed for the sintering of various materials from metals to ceramics at wide ranges of pressures and temperatures depending on the physical characteristics of the target materials and on the require material processing conditions, so that it is capable of short-period, low-temperature SPS at high pressures of 5 to 10 tonsf/cm2 as well as SPS in the high temperature range of 1000 to 2000℃ with a lower pressure in the hundred of Kgf /cm2 range. In addition to the easy control of the sintered microstructure with little grain growth, the system offers various advantages such as the possibility of processing of hard-to-sinter materials which can not be done with ordinary sintering processes and a reduction of undesirable reactions between additives and the base material. It can also be used to great advantage in the fabrication of porous sintered objects using materials which are normally difficult to sinter, for example, titanium and aluminum which form hard oxide layers [2, 3].

2. Pulse energizing effect

Conventional electrical hot press processes use DC or commercial AC powder, and the main factors promoting sintering in theses sintering processes are the Joule heat generated by the power supply (I2R) and the plastic flow of materials due to the application of pressure. There is also an induction heating type process using RF current, but the factors promoting sintering are the same.

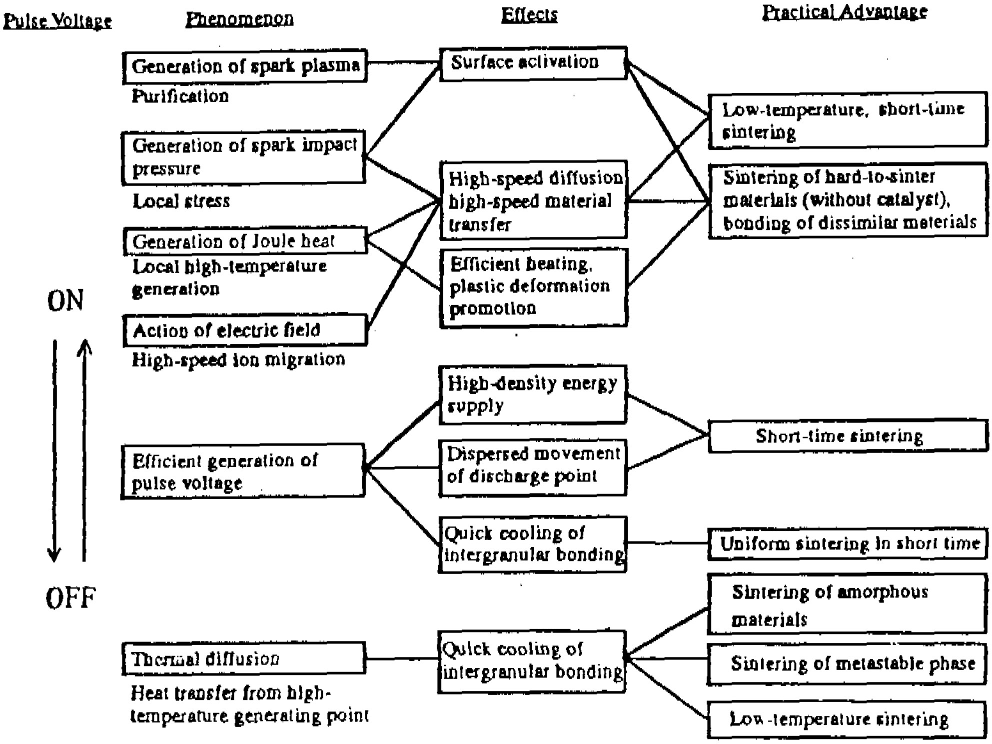


Fig.1 Effect of ON-OFF DC Pulse energizing

Here, a very important effect occurs in the OFF state with certain materials and the properties of the sintering target. After the high-speed temperature rise between the powder particles, intergranular bonding is rapidly cooled by thermal diffusion. Pulse energizing allows the supplied energy to be controlled with high precision while observing the progress of sintering. The action of the electrical field also causes high-speed diffusion due to the high-speed migration of ions. By applying repeated ON-OFF voltages, the discharge point (local high-temperature generation field) moves within the compacted particles and is dispersed throughout the sample. This is a repeated phenomenon and it effects the sample uniformly with the ON condition, so that efficient sintering with a relatively low power consumption is possible. The ability of the design to produce a positive concentration of high-energy pulses in places where intergranular bonds are to be formed is one of the main features of the SPS process, distinguishing it from other electrical sintering processes [4 - 6].

3. Intergranular Bonding and Surfaces

When a spark plasma moves in a void between the grains of a material, a local high-temperature state (discharge column) of several to ten thousands of degrees centigrade is generated momentarily. This causes evaporation and fusing on the surface of contact between grains which result in a welded state.

Compare the mechanism of sintering evaporative solidification, volume diffusion shown in the material transfer diagram in Fig. 2 with actual SPS intrergranular bonding. Fig. 3 is SEM photomicrographs showing the result of SPS experiment per formed at normal atmospheric pressure using a sintering die and punches made of graphite and a spherical bronze powder (Cu/Sn : 90/10% by weight) with a grain size of no more than 325 mesh. Fig. 3 (a) shows the phenomenon displayed between single grains which corresponds to Fig. 2, clearly capturing the behavior in the initial stage of neck formation due to sparks in the plasma. The heat is transferred immediately from the center of the heat generating part to the sphere surface and diffused so that intergranular bonding is quickly cooled. Because the vapor pressure at the necks is lower than in other parts, evaporated material condenses on the necks.

As seen in Fig. 3 (b) which show several necks, the pulse power supply method causes spark discharges one after another between grains. Even with single grains, the number of positions where necks are formed between adjacent grains increases as the discharges are repeated. At the same time, the intergranular contacts are subject to Joule heat and the places where gaps remain continue to present the possibility of the occurrence of a plasma discharge.

Fig. 3 (C) shows the condition of an SP-sintered grain boundary which is plastic-deformed after the sintering has progressed further. This state is the result of the processing conditions in which the applied pressure was 300kgf/cm2 , the SPS holding temperature was 500℃, the holding time was 120 sec, the SPS current 850A, and the voltage was 3.9V. Because the power grains are heated by a pulse power supply under vertical single-axis pressure, material transfer with the help of slipping between plastic flow grains is facilitated by the pressure and thermal softening as well as the migration of atoms and ions, so the gaps are filled and the number of holes is decreased. The sintering die and punches, which are resistance elements, are subjected to Joule heating with a small delay according to the progress of the sintering of the internal powder, and function as heating elements to assume the role of maintaining the temperature of the sintered object. Finally, the grain boundaries disappear and the material becomes an integral sintered object.

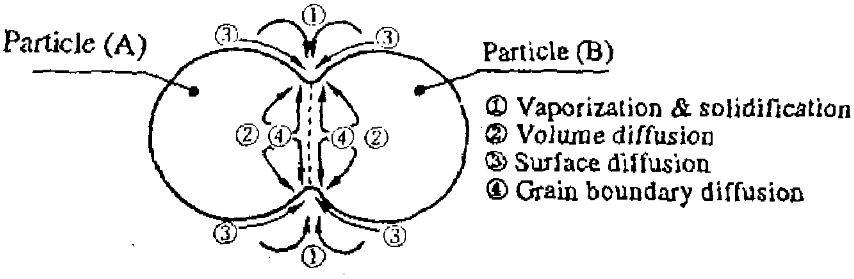


Fig. 2 Material transfer path during sintering

(a) Initial stage of neck growth (b) Expansion of neck (c) Start of plastic flow

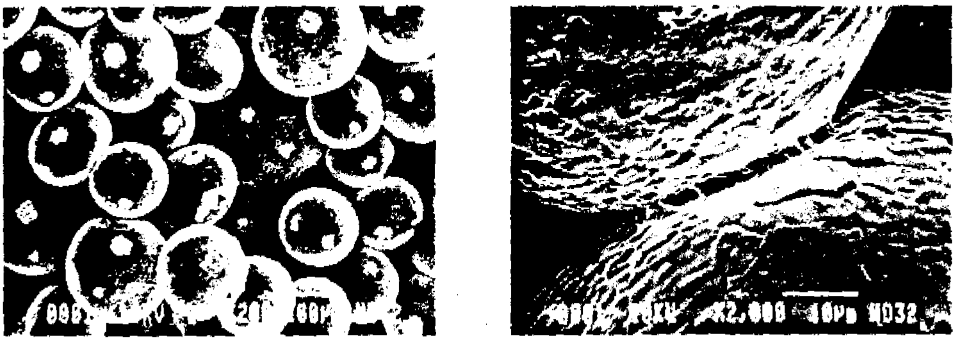
Fig. 3 SEM micrographs of Initial stage, Intermediate stage and Final stage of SPS processing

4. Sintering of porous media

As the SPS process is based on the principle of utilizing the self-heating phenomenon in intergranular space, it is capable of localized high-temperature diffused bonding and therefore suitable for fablicating porous objects both with and without gradients. The applications being studied include metallic and ceramic filters, catalyst carriers, energy-related functional materials and bio-materials.

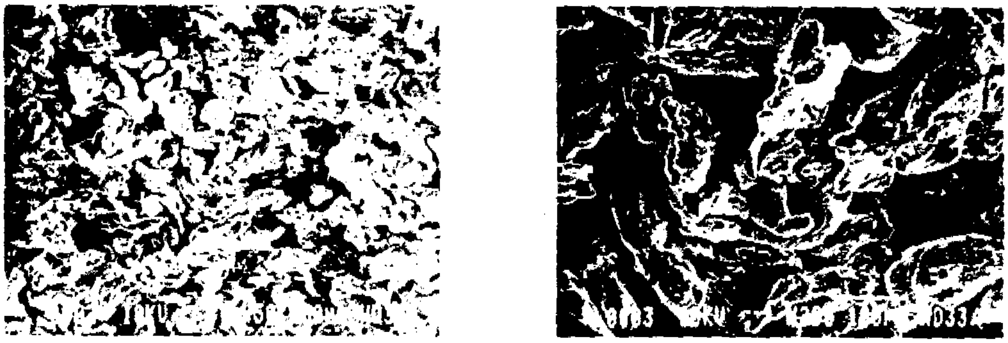
Fig. 4 shows metallurgical micrographs and SEM micrographs of the intergranular bonding state of a porous compact obtained from spherical pure Iron powder with a grain size of 100-125㎛.These were sintered with an applied pressure of 86kgf/cm2, a holding temperature of 800℃, temperature rise time of 7 minutes, holding time of 1 minute, in a vacuum. The micrographs show that spherical particles are crushed by the applied pressure and bonded. The broken surface state of the fractured section in the neck shows that this is a homogeneous porous object in which the particles are bonded to each other with high strength.

As is clear from the example of spherical bronze alloy powder in Fig. 3 described before, the SPS process can sinter porous objects at any desired porosity by setting the applied pressure and sintering temperature (current, voltage, power supply period) as necessary.



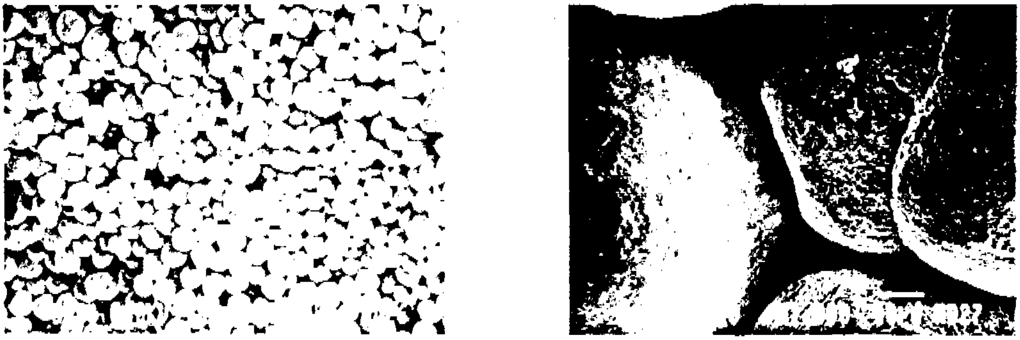
(a) (b)

Fig. 4 SEM micrographs of (a) sintered Iron powder by SPS treatment, and (b) enlarged (a)
[Sintering condition : 86kgf/cm2 pressure, 800℃, 1min]



(a) (b)

Fig. 5 SEM micrographs of (a) sintered SUS316L powder by SPS treatment, and (b) enlarged (a)
[Sintering condition : 200kgf/cm2 pressure, 650℃, 3min]



(a) (b)

Fig. 6 SEM micrographs of (a) sintered Ni powder by SPS treatment, and (b) enlarged (a)
[Sintering condition : 86kgf/cm2 pressure, 500℃, 5min]

Fig. 5 shows an example of SUS316L stainless steel powder with irregular shape. Pores generally below 100㎛ are formed continuously with 20% porosity using a starting material powder with a grain size of ~300㎛.

Porous object with high porosity (~30%) can also be fabricated using spherical metal powder materials such as Ni with grain size of less than 100㎛ (Fig. 6).

5. conclusion

Spark Plasma Sintering (SPS) is a newly developed process sintering – a synthesis and processing technique – which makes possible sintering and sinter-bonding at low temperatures and short periods by charging the intervals between powder particles with electrical energy and effectively applying a high temperature spark plasma generated momentarily. This paper reviews the application and mechanism of SPS process is expected to find increased use in the fabrication of porous media with high strength, which are difficult to sinter by conventional sintering methods.

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