Spark Plasma Sintering

Sintering is the process of making objects from powder, by heating the material in a furnace below its melting point so that bonding takes place by diffusion of atoms. This leads to individual powder particles adhering to each other in a dense compact. The process is usually used for ceramics. Sintering can be done using various methods as Conventional Sintering, Spark Plasma Sintering (SPS), Microwave Sintering etc. Spark Plasma sintering is one of the processing routes to process biomaterials in laboratory.

Advantage over Conventional Sintering

Spark Plasma Sintering is a new technique which takes only a few minutes to complete a sintering process compared to conventional sintering which may take hours or even days for the same. This high sintering rate is possible in SPS since high heating rates can be easily attained due to internal heating of the sample as opposed to external heating seen in case of conventional sintering. Also, sintering time is reduced in SPS due to small holding time at sintering temperature, usually 5 to 10 minutes while in conventional sintering it may extend to hours. The heating rates normally attained in conventional furnaces are 5 to 8°C/min which can go maximum upto 10°C/min. So, to attain a temperature of 1200°C we usually require 2 to 4 hours or more whereas in SPS heating rates exceeding 300°C/min are easily obtained hence a temperature of 1200°C can be obtained in only 4 minutes.

Simultaneous application of temperature and pressure leads to high densification and hence a dense compact at sintering temperatures lower by 200 to 250°C than in conventional sintering is easily obtained. In SPS, since no coarsening and grain growth were allowed to occur, high relative densities were reached in very short time and nano sized powders can be sintered without considerable grain growth which is not possible in conventional sintering process. Hence, nano-structured ceramics or nano-composites can be easily prepared by SPS having more densification and fewer defects. These nano-structured composites exhibit excellent mechanical properties like high strength, high hardness etc.

For conventional sintering usually a green compact needs to be prepared externally using a suitable die and hydraulic machine for applying the necessary pressure. After this the green compact is sintered in a furnace. In SPS the powder is directly fed into the graphite dies and the

die is enclosed with suitable punches. This entire assembly is directly put into the SPS chamber and spacers are used if necessary. The chamber is now closed and the atmosphere (vacuum, Argon etc) in which sintering is to be carried out is applied in the chamber. The program is set into the control unit and sintering is carried out. Atmosphere control is much easier in SPS than in conventional furnaces. All types of materials, even those difficult-to-densify can be easily sintered in SPS. Due to advantage of high heating rate and less holding time, SPS can restrict the unwanted sintering reactions in highly reactive systems as opposed to conventional sintering and hence formation of undesirable product phases can be avoided.

Process:

Take known quantity of dried and milled powder sample in a cylindrical die, lined with graphite sheet which facilitates easy removal of the sintered compact. Switch on the Chiller – compressor and the machine. Place the die containing the powder sample inside the SPS chamber. Then, set the specific pattern (time-temperature data) needed for the experiment to follow and thereafter, maintain the required atmospheres as Vacuum, Argon etc inside the chamber. Set the power to maximum in Auto mode and the required load to be applied on the die. Also, set the z-axis position to zero. **ON** the timer and finally press **SINTER** to start the sintering process. For measuring the temperature in SPS two types of instruments are used: thermocouple for sintering temperatures below 1000°C and pyrometer for sintering temperatures in excess of 1000°C.

High DC Pulse is passed between graphite electrodes and axial pressure is simultaneously applied from the beginning of the sintering cycle. The sample is heated by the Joule-heating and the sparking among the particles of sintered material leads to the faster heat and mass transfer instantaneously. After the sintering, the power is turned off and the sample is allowed to cool.