Spark Plasma Sintering

➤ The spark plasma sintering process offers significant improvements over conventional hot press and hot isostatic press sintering.

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Spark plasma sintering (SPS) is a high-speed powder consolidation/sintering technology capable of processing conductive and non-conductive materials. Theories on the SPS process vary, but most commonly accepted is the micro-spark/plasma concept, which is based on the electrical spark discharge phenomenon wherein a high-energy, low-voltage pulse current momentarily generates spark plasma at high temperatures (many thousands of °C) in fine local areas between particles.

SPS' operational or "monitored" temperatures (200-2400°C) are commonly 200 to 500°C lower than with conventional sintering, classifying SPS as a lower-temperature sintering technology. Material processing (pressure and temperature rise and hold time) is completed in short periods of approximately 5 to 25 minutes. The relatively low temperatures combined with fast processing times ensure tight control over grain growth and microstructure.

Process

SPS utilizes uniaxial force and ON-OFF DC pulse energizing. The ON-OFF DC pulse voltage and current creates spark discharge and Joule heat points between material particles (high-energy pulses at the point of intergranular bonding). The high frequency transfers and disperses the spark/Joule heat phenomena throughout the specimen, resulting in a rapid and thorough heat distribution, high homogeneity and consistent densities.

The initiation of the spark discharge in the gap between particles is assisted by fine impurities and gases on and between the surfaces of the particles. The spark discharge creates a momentary local high-temperature state of up to 10,000°C, causing vaporization of both the impurities and the surfaces of the particles in the area of the spark. Immediately behind the area of vaporization, the surfaces of the particles melt. Via electron draw during ON TIME and the vacuum of OFF TIME, these liquidized surfaces are drawn together, creating "necks." The ongoing "radiant" Joule heat and pressure causes these necks to gradually develop and increase. The radiant heat also causes plastic deformation on the surface of the particles, which is necessary for higher-density applications.

During the SPS process, heat is concentrated primarily on the surfaces of the particles. Particle growth is limited due to the speed of the process and the fact that only the surface temperature of the particles rises rapidly. The entire process—from powder to finished bulk sample—is completed quickly, with high uniformity and without changing the particles' characteristics.

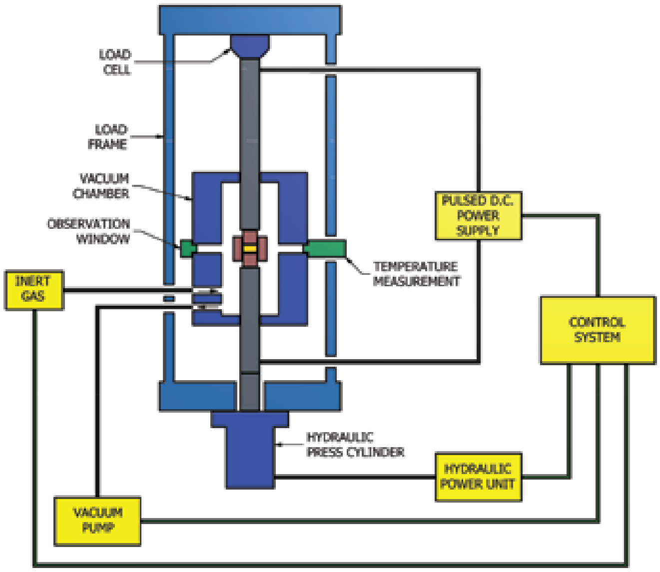

Figure 1. Basic configuration of an SPS machine.

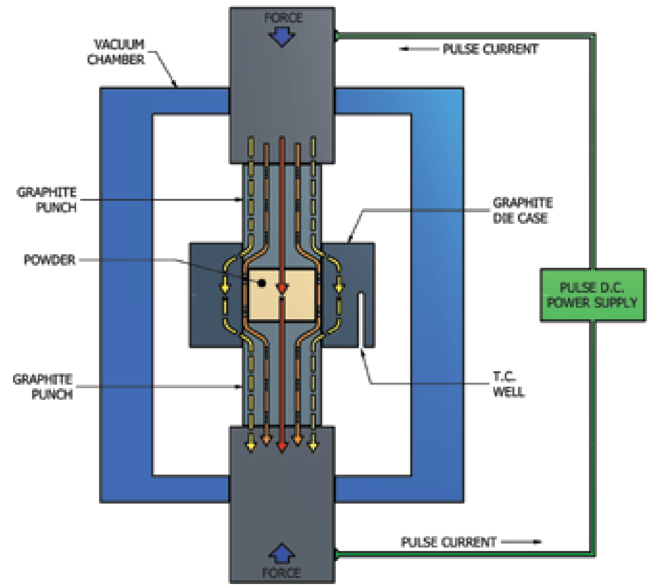

Figure 2. ON-OFF pulsed current path through the machine.

Force (pressure) plays an important and predictable role in curbing particulate growth and influencing overall densities, but in the SPS process, accurate manipulation of force can actually enhance the process. Force multiplies spark initiation (diffusion) throughout the sample as the material moves under pressure, especially during critical out-gassing stages. Both too much and too little pressure can negatively influence the process. In large samples where high density is required, force is commonly increased in stages to enhance out-gassing and electrical diffusion.

Applications

SPS is effective for any powder material application, but interest is especially high for nanocrystalline structures. Generally, superfine materials have more surface area per volume than the same material made with larger particles. This, along with the way particles interact once compacted, "amplifies" material characteristics. In theory, high-strength materials show an increase in strength, highly wear-resistant materials show higher wear resistance, highly magnetic materials show higher magnetism, and so on. Suitable applications include advanced lightweight armament, guidance optics and ultra-high-strength tooling.

Nanomaterials haven't seen much industrial commercialization to date because conventional sintering technologies cause substantial particulate damage and growth. However, since SPS technology can sinter nanocrystalline materials with very little grain growth and negative particulate effect, the door is now open to test and study new ideas in powder material applications. Many common materials may well find new applications with substantially improved characteristics.

SPS technology can sinter materials without the use of binders. Most conventional powdered material sintering technologies require pre-forming and binders, and in many cases, expensive binder removal processes. Binders weaken the part due to their susceptibility to chemical wear, reduced hardness and strength, and oxidation breakdown. The availability of binder-less material could be valuable in numerous applications, including cobalt-less tungsten carbide, high-purity ceramic fuel cells and ceramic optics.

Part Characteristics

The rapid spark diffusion and the consistent heat throughout the part produces very little internal stress within a part made with SPS. Many part failures occur due to internal stress and microcracks caused by heat migration during conventional sintering. SPS technology binds particles with electrical discharge energy evenly throughout the part. The heat is simultaneously consistent at the outside of the part and the center. Along with the relative speed of the process, this eliminates much of the internal stress commonly seen in conventionally sintered parts.

SPS technology is capable of achieving nearly 100% theoretical density in almost any metallurgical or ceramic material, including composites. When ultra-high densities are required, some grain growth is necessary. Some materials require binders that are added to facilitate density with less grain growth, while other applications require very specific and uniform porosity. By accurately controlling compression and temperature, SPS technology can control material porosity while maintaining strong particle bonds throughout the shape.

"Net" and "near-net" shapes are also possible with SPS, going directly from powder to finished part in one step. Currently, these shapes need to be symmetrical and relatively simple; a complex shape would require secondary machining.

SPS technology can produce true seamless bonding (dry and liquid phase bonding). Because the SPS process draws particles together and offers the ability to reach nearly 100% theoretical

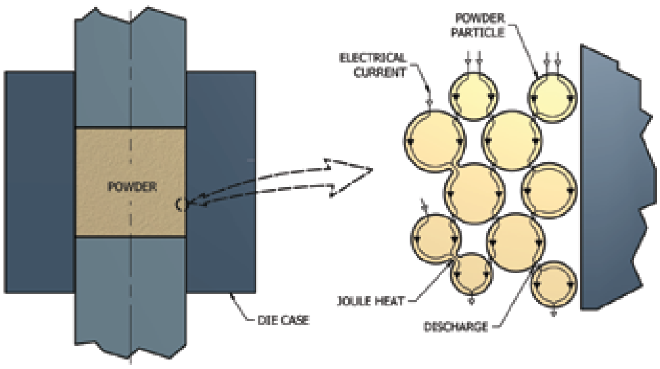

Figure 3. ON-OFF pulsed current path through the powder.

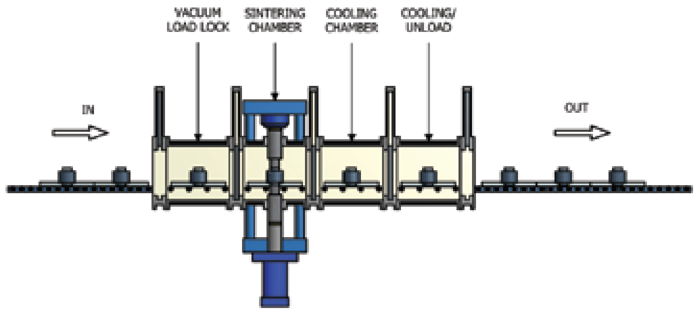

Figure 4. Example of a production concept.

density, the process can produce bonded parts that have no seam. This bonding phenomenon is possible between like and dissimilar materials, though radically dissimilar materials require progressing layers between the initial materials to account for different thermal stresses. In the case of bonding rough surfaces, loose powder can be applied between the surfaces to ensure a solid bond.

Benefits

As mentioned previously, the SPS process utilizes high-amperage pulse DC current to generate spark plasma energy between each particle. Physical compression can be up to 300 tons and the chamber is under negative atmospheric pressure (vacuum) with or without inert gas. Heat is concentrated on the surface area of each particle, and every particle is equally and completely bonded with the surrounding particles. Under high pressure, mild plastic deformation of the particles ensures ultra-high density values, while high-porosity, fully bonded materials can be achieved with lower pressure and less heat and time.

Pre-existing oxidation and contaminates are vaporized from particle surfaces during sintering, providing for higher-purity materials and stronger bonding between particles. SPS is capable of sintering dissimilar materials without going to a liquid phase on the lower-temperature materials. In the case of composite materials, high homogeneity is possible even with lower densities. In the case of layered materials known as functionally graded materials (FGMs), preformed layers remain consistent in density and shape even if the materials have radically different sintering properties.

Consideration of a powdered metallurgical "recipe" requires metallurgical expertise, but the actual operation of the machine is quite simple. Once the die set is loaded and the temperature feedback system (thermocouple or pyrometer) is in place, the operator programs the temperature and pressure ramp-up and hold settings. The atmosphere (vacuum and/or inert gas) is set, the electrical settings (ON-OFF times and frequency strategy) are programmed and then the data feedback graphics are set up. SPS technology can also be combined with various forms of production and automation systems, including multi-head, rotary, batch, and conveyor systems. Robotic interface is also possible.

Finally, SPS operational expenses are consistently 50 to 80% less than conventional sintering technologies, due primarily to speed. In some applications, SPS technology has been more than 20 times faster than conventional sintering technologies. Most SPS applications only require minutes in comparison to the hours needed with conventional systems.

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