Chapter 13

Challenges and Opportunities for Spark Plasma Sintering: A Key Technology for a New Generation of Materials

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1. Introduction

Spark plasma sintering (SPS) or pulsed electric current sintering (PECS) is a sintering technique utilizing uniaxial force and a pulsed (on-off) direct electrical current (DC) under low atmospheric pressure to perform high speed consolidation of the powder. This direct way of heating allows the application of very high heating and cooling rates, enhancing densification over grain growth promoting diffusion mechanisms (see Fig. 1), allowing maintaining the intrinsic properties of nanopowders in their fully dense products.

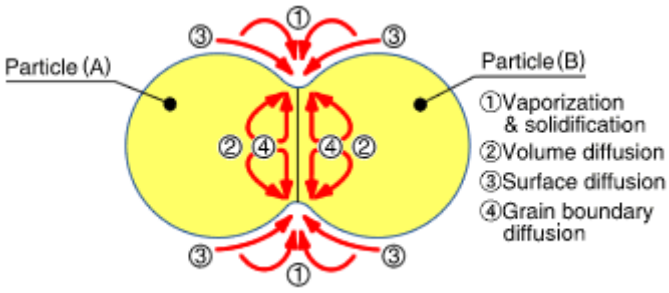

Figure 1. Material transfer path during sintering

It is regarded as a rapid sintering method in which the heating power is not only distributed over the volume of the powder compact homogeneously in a macroscopic scale, but moreover the heating power is dissipated exactly at the locations in the microscopic scale, where energy is required for the sintering process, namely at the contact points of the powder particles (see Fig. 2). This fact results in a favourable sintering behaviour with less grain growth and suppressed powder decomposition. Depending on the type of the powder, additional advantageous effects at the contact points are assumed by a couple of authors.

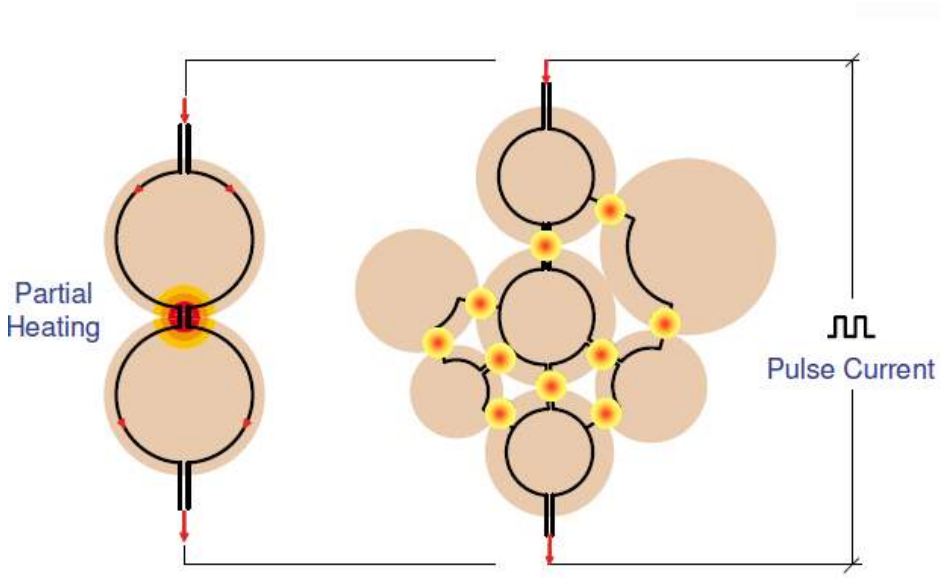

Figure 2. Energy dissipation in the microscopic scale

SPS systems offer many advantages over conventional systems 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. While similar in some aspects to HP, the SPS process is characterized by the application of the electric current through a power supply, leading to very rapid and efficient heating (see Fig. 3). The heating rate during the SPS process depends on the geometry of the container/sample ensemble, its thermal and electrical properties, and on the electric power supplier. Heating rates as high as 1000 °C/min can be achieved. As a consequence, the processing time typically takes some minutes depending on the material, dimensions of the piece, configuration, and equipment capacity.

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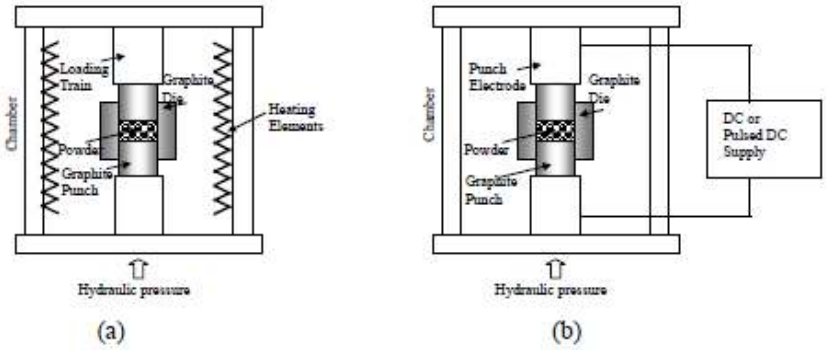

Figure 3. Schematic representation of (a) HP and (b) SPS

On the contrary, in conventional HP techniques, the powder container is typically heated by radiation from the enclosing furnace through external heating elements and convection of inert gases if applicable. Therefore, the sample is heated as a consequence of the heat transfer occurring by conduction from the external surface of the container to the powders. The resulting heating rate is then typically slow and the process can last hours. In addition, a lot of heat is wasted as the whole volume of space is heated and the compact indirectly receives heat from the hot environment. On the other hand, SPS processes are characterized by the efficient use of the heat input, particularly when electrically insulating powders is used and the pulsed electric current is applied.

It should be however mentioned that in SPS processes the problem of adequate electrical conductivity of the powders and the achievement of homogenous temperature distribution is particularly acute. In this way, the electric current delivered during SPS processes can in general assume different intensity and waveform which depend upon the power supply characteristics. In order to permit a homogeneous sintering behaviour, the temperature gradients inside the specimen should be minimized. Important parameters that are drastically determining the temperature distribution inside the sample are the sample material's electrical conductivity, the die wall thickness and the presence of graphite papers used to prevent direct contact between graphite parts and the specimen and used to guarantee electrical contacts between all parts.

The application of external electric current to assist sintering was initiated by Taylor in 1933, who incorporated the idea of resistance sintering during the hot pressing of cemented carbides [Taylor, 1933]. Later, Cramer patented a resistance sintering method to consolidate copper, brass and bronze in 1944 in a spot welding machine [Cremer, 1944]. The concept of compacting metallic materials to a relatively high density (>90% of theoretical) by an electric discharge process was originally proposed by Inoue in the 1960s [Inoue, 1965]. Inoue argued that a pulsed current was effective for densification at the initial sintering stages for low melting point metals (e.g., bismuth, cadmium, lead, tin) and at the later sintering stage for high melting metals (e.g., chromium, molybdenum, tungsten). In the United States, Lenel al-

so used a spot welding machine for the sintering of metals [Lenel, 1955]. In addition to continuous pulses, some researchers also investigated a single discharge method, i.e., the powders were densified by a single discharge generated from a capacitor bank. In the late 1970s, Clyens et al. [Clyens et al., 1976], Raichenko et al. [Raichenko et al., 1973] and Geguzin et al. [Geguzin et al., 1975] studied the compaction of metal powders using electric discharge compaction (EDC) or electric discharge sintering (EDS). In all the methods cited, electrically conductive powders are heated by Joule heating generated by an electric current.

In 1990 Sumitomo Heavy Industries Ltd. (Japan), developed the first commercially operated plasma activated sintering (PAS) and spark plasma sintering (SPS) machines with punches and dies made from electrically conductive graphite [Yanagisawa et al., 1994]. One of the salient features of these machines was that, in addition to electrically conductive powders, high density was also achieved in insulating materials. In PAS process, a pulsed direct current is normally applied at room temperature for a short period of time followed by a constant DC applied during the remainder of the sintering process (see Fig. 4a). This procedure is often referred to in the literature as a "single pulse cycle process". In the SPS process, a pulsed DC is applied repeatedly from the beginning to the end of the sintering cycle (see Fig. 4b). In this case the procedure is referred to as a "multiple pulse cycle process".

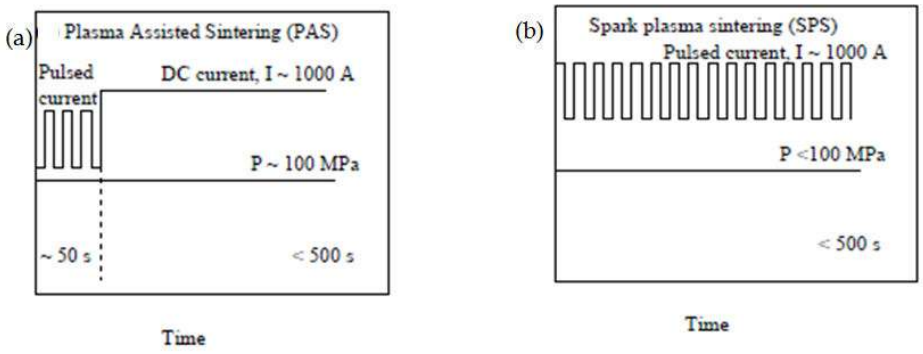

Figure 4. Sintering techniques

2. The basic SPS configuration and process

The basic configuration of a typical SPS system is shown in Figure 5. The system consists of a SPS sintering machine with vertical single-axis pressurization and built-in water-cooled special energizing mechanism, a water-cooled vacuum chamber, atmosphere controls, vacuum exhaust unit, special sintering DC pulse generator and a SPS controller. The powder materials are stacked between the die and punch on the sintering stage in the chamber and held between the electrodes. Under pressure and pulse energized, the temperature quickly

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rises to 1000~2500 °C above the ambient temperature, resulting in the production of a high quality sintered compact in only a few minutes.

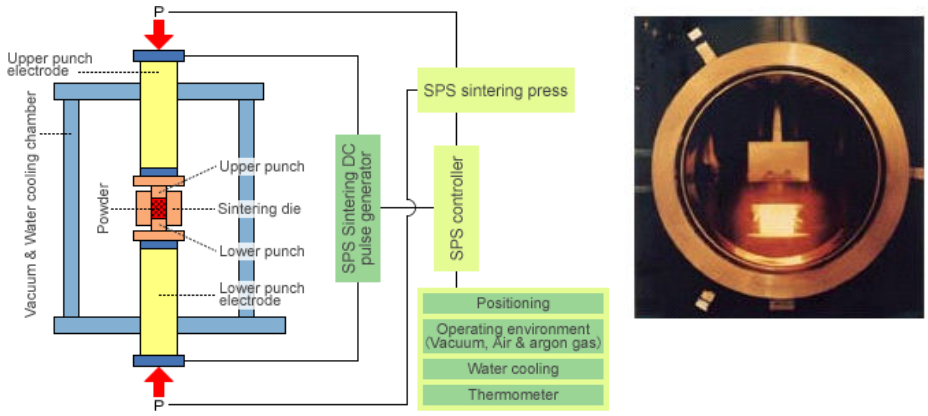

Figure 5. SPS system configuration and vaccum chamber

3. Principles and mechanism of the SPS process

The SPS process is based on the electrical spark discharge phenomenon: a high energy, low voltage spark pulse current momentarily generates spark plasma at high localized temperatures, from several to ten thousand ℃ between the particles resulting in optimum thermal and electrolytic diffusion. SPS sintering temperatures range from low to over 2000 ℃ which are 200 to 500 ℃ lower than with conventional sintering. Vaporization, melting and sintering are completed in short periods of approximately 5 to 20 minutes, including temperature rise and holding times. Several explanations have been proposed for the effect of SPS:

3.1. Plasma generation

It was originally claimed by Inoue and the SPS process inventors that the pulses generated sparks and even plasma discharges between the particle contacts, which were the reason that the processes were named, spark plasma sintering and plasma activated sintering [Inoue, 1965, Yanagisawa et al., 1994]. They claimed that ionization at the particle contact due to spark discharges developed "impulsive pressures" that facilitated diffusion of the atoms at contacts. Groza [Groza et al, 1999] suggested that a pulsed current had a cleaning effect on the particle surfaces based on the observation of a grain boundary without oxidation formed between particles. Whether plasma is generated or not has not yet been confirmed directly by experiments. Therefore, there is no conclusive evidence for the effect of a plasma generation in SPS. The occurrence of a plasma discharge is still debated, but it seems to be widely accepted that occasional electric discharges may take place on a microscopic level [Groza et al., 2000].

3.2. Electroplastic effect

Metal powders have been observed to exhibit lower yield strength under an electric field. Raichenko et al. [Raichenko et al., 1973] and Conrad [Conrad, 2002] independently studied electroplastic phenomena.

3.3. Joule heating

Joule heating due to the passage of electric current through particles assists in the welding of the particles under mechanical pressure. The intense joule heating effect at the particle conducting surface can often result in reaching the boiling point and therefore leads to localized vaporization or cleaning of powder surfaces [Tiwari et al., 2009]. Such phenomenon ensures favourable path for current flow.

3.4. Pulsed current

The ON-OFF DC pulse energizing method generates: (1) spark plasma, (2) spark impact pressure, (3) Joule heating, and (4) an electrical field diffusion effect (see Table 1).

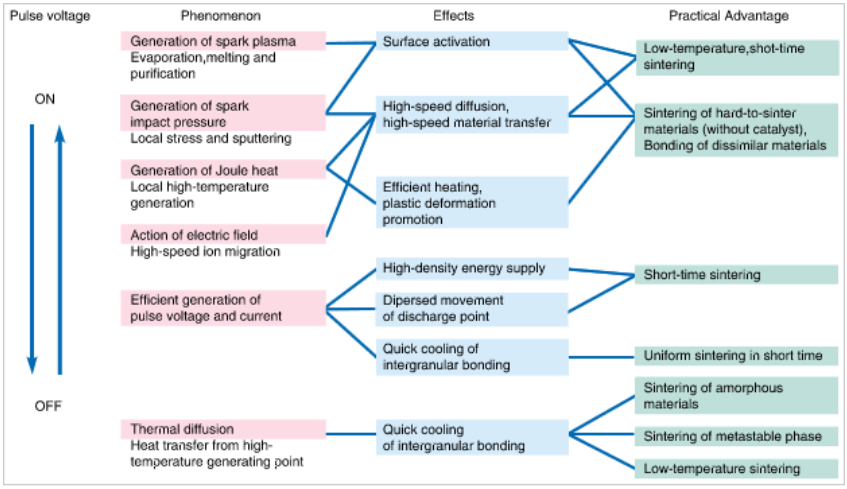

Table 1. Effects of ON-OFF DC

In the SPS process, the powder particle surfaces are more easily purified and activated than in conventional electrical sintering processes and material transfers at both the micro and macro levels are promoted, so a high-quality sintered compact is obtained at a lower temperature and in a shorter time than with conventional processes. Figure 6 illustrates how pulse current flows through powder particles inside the SPS sintering die.

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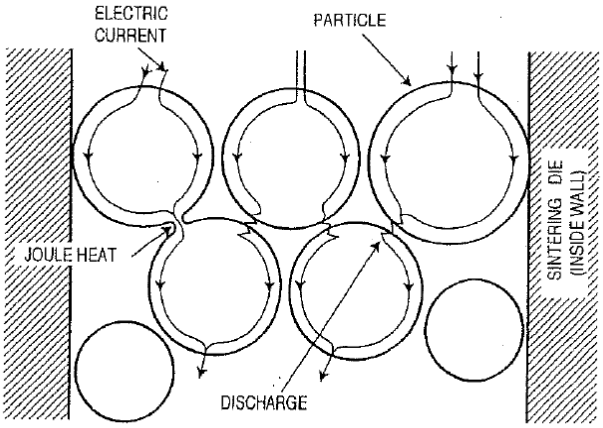

Figure 6. Pulsed current flow through powder particles

The SPS process is an electrical sintering technique which applies an ON-OFF DC pulse voltage and current from a special pulse generator to a powder of particles (see fig. 7), and in addition to the factors promoting sintering described above, also effectively discharges between particles of powder occurring at the initial stage of the pulse energizing for sintering.

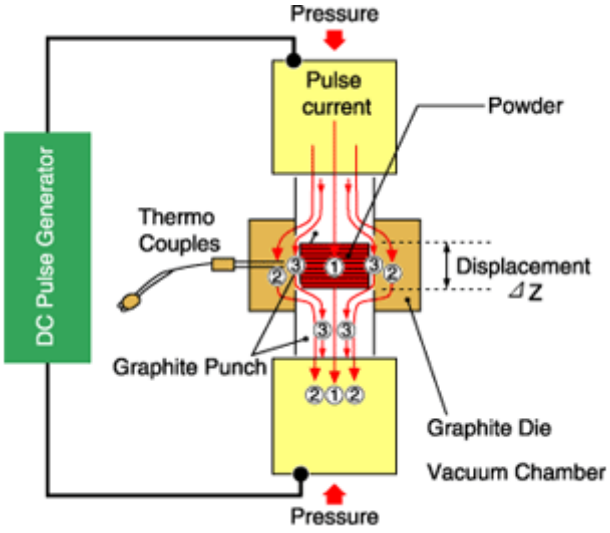

Figure 7. ON-OFF pulsed current path through the spark plasma sintering machine

High temperature sputtering phenomenon generated by spark plasma and spark impact pressure eliminates adsorptive gas and impurities existing on the surface of the powder particles. The action of the electrical field causes high-speed diffusion due to the high-speed migration of ions.

3.5. Mechanical pressure

When a spark discharge appears in a gap or at the contact point between the particles 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 melting on the surface of powder particles in the SPS process, and "necks" are formed around the area of contact between particles. Figure 8 shows the basic mechanism of neck formation by spark plasma.

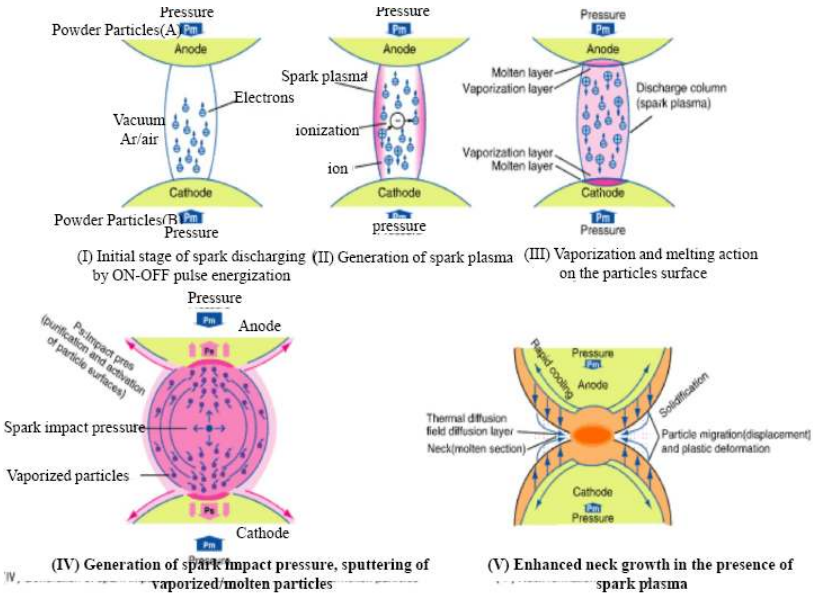

Figure 8. Basic mechanism of neck formation by spark plasma

Figure 9a shows 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 spark discharge column to the sphere surface and diffused so that intergranular bonding portion is quickly cooled. As seen in Figure 9b which show several necks, the pulse energizing method causes spark discharges one after another between particles. Even with a single particle, the number of positions where necks are formed between adjacent particles increases as the discharges are repeated. Figure 9c shows the condition of an SPS sintered grain boundary which is plastic deformed after the sintering has progressed further.

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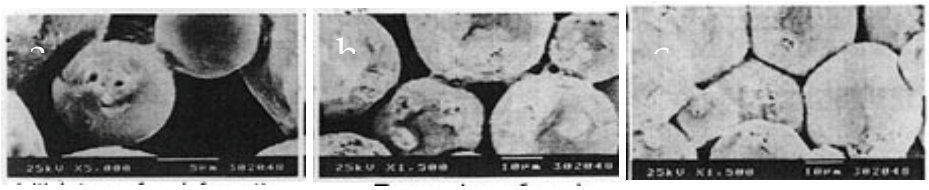

Figure 9. SPS sintering steps

4. Materials

The increasing importance of the electric current assisted sintering method as a tool for consolidation of powders is demonstrated by the large number of papers published in the recent years. It should be noted that references written in English language, at least at the abstract level, since 1922 to 2007 show an exponential growth since the late 90's [Orru et al., 2009]. While there are other reviews focused on a large list of materials sintered by SPS, we concentrate here on some relevant applications, mainly hard materials.

4.1. Cutting tools – Carbon nitrides (Materials Science and Engineering A 543 (2012) 173–179); hard materials

4.1.1. Titanium nitride

One of the most direct applications of SPS is the sintering of high melting point materials such as titanium nitride. This ceramic has received little attention in its bulk form due to its poor sinterability and inherent brittleness. Even more, all the works reported in the literature used the hot press technique to achieve densification. In particular, it has been shown [Yamada et al., 1980] that by employing extremely high pressures on the order of 1 to 5 GPa, it is possible to sinter pure titanium nitride to high density. In that work, they claimed relative densities of 98% for course powders and 95% for fine powders, respectively with a pressure of 5.0 GPa and a temperature of 1500 °C. More recent works showed [Graziani et al., 1995] that after 30 minutes at 1850 °C with a pressure of 30MPa, it was possible to obtain relative densities around 95%.

4.1.2. Transition metal carbonitrides

Similarly, transition metal carbonitrides are becoming increasingly important materials due to their excellent properties in the fields of superhardness, tribology, superconductivity and, electrical and thermal conductivities. Titanium carbonitride is the most widely employed material because it is the main component in commercial cermets. These cermets are prepared from a Ti(C, N) powder, or a combination of TiC and TiN powders, with Ni

as binder and are manufactured by sintering the compacted powder mixture at a temperature at which a liquid phase is formed. Again, due to the high temperatures required to sinter this material, Ti(C, N)-based cermets, hot pressing, hot isostatic pressing, or a combination of HIP'ing and sintering, under vacuum, nitrogen or argon atmosphere are currently used. One of the most common ways to prepare titanium carbonitride is to hot press blended mixtures of TiC and TiN powders in vacuum or argon atmosphere at 1700–2400 °C [Monteverde et al., 2002]. For this reason, SPS has emerged as a useful technology and has already been used to densify titanium carbonitride powders at a relatively low temperature [Borrell et al., 2012].

Titanium–silicon–nitrogen (Ti–Si–N) composites were SPSed from a mixture of Si_3N_4, titanium nitride (TiN), and titanium thus developing wear-resistant materials with good environmental and biological compatibilities in water and sea water [Hibi et al., 2002]. SPS compaction experiments of nanostructured titanium carbonitride powders, synthesized through rapid condensation from the gas-phase (high-frequency plasma), have been performed at 1600 and 1800 °C (sintering time = 1 min) [Angerer et al., 2005]. The sintering results were compared with data obtained by various conventional sintering techniques such as pressureless sintering, gas pressure sintering, and hot pressing. The experiments showed that the SPS method is capable of obtaining high densities (about 94% of theoretical density) combined with small grain size quotient d/d_0 of 5.4–6.5.

4.1.3. Al_2O_3-SiC

Al_2O_3–SiC composites are usually sintered by hot pressing of powder mixtures [Sun et al., 2005]. Although the simultaneous heating and uniaxial pressure applied facilitates the densification of these composites, high temperatures up to 1800 °C are usually required. Due to the high sintering temperatures needed, alumina grain growth and the subsequent final microstructure control is limited. Two temperatures were used for spark plasma sintering tests (1400 °C and 1550 °C) [Borrell et al., 2012]. In both cases, nearly fully dense samples (>99.0% relative density) were obtained for all Al_2O_3–17 vol.% SiC composites prepared through the different raw materials combinations tested. It was then shown that when similar composite powders (Al_2O_3–SiC) are sintered by hot press, at least 1650 °C and 1 h soaking time are required for total densification, even if the total SiC content is lower (5 vol.%) [Wang et al., 2000].

4.1.4. Transparent ceramics

4.1.4.1. Alumina

As in the previous case, grain growth is an important issue determining the mechanical performance of transparent ceramics. Even more, in the case of non cubic ceramics such as alumina or zirconia, the birefringence is a critical factor leading to a dispersion that is directly related to the grain size of the ceramics. For this reason, fine-grained transparent Al_2O_3 ceramics have recently attracted much attention due to their superior mechanical and optical properties [Apetz et al., 2003]. Conventional sintering requires very

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high temperatures and long holding time, which leads to a deficient mechanical performance [Wei et al., 2001]. Hot isostatic pressing reduces the temperature required, leading to high strength and translucent alumina [Mizuta et al., 1992]. It has been recently demonstrated [Jin et al., 2010] that it is possible to sinter to transparency common-grade commercial powders by spark plasma sintering. The powders, previously treated in HF, were sintered between 1300 and 1400 °C for 3 minutes under an applied pressure of 80 MPa and a heating rate of 100 °C min^{-1}. Another alternative reported in the literature is the use of the so-called self-doping process [Suarez et al., 2009] in which alumina powders are doped with aluminium ethoxide which will transform to alumina after calcination. These powders were sintered at 1200 °C, with maximum shrinkage velocity at 1000 °C, under an applied pressure of 80 MPa with a holding time of 20 min. In another doping process, the precipitation of CeO_2 nanoparticles (< 5 nm) on the surface of the starting alumina nanopowder using cerium(III) acetate as precursor has been studied [Alvarez et al., 2010]. It was then shown that the ceria nanoparticles strongly enhance the transparency of the spark plasma sintered compacts due to both the ceria nanoparticles acting as powder lubricant, increasing by around 15% the initial density of the powder in the SPS die and the CeO_2 nanoparticles, locating at grain boundaries, hindering alumina grain growth by pinning during SPS sintering at 1430 °C, under an applied pressure of 80 MPa for 2 min. This effect was found to be effective only under SPS vacuum conditions.

4.1.5. Yttrium aluminium garnet

Yttrium aluminium garnet (YAG) is a material showing a large transparency range from 250 nm (UV range) up to 5 μm. It is often produced in the single crystal form, but this limits the size and shape of the components produced. It was shown [Suarez et al., 2009b] that lyophilized YAG powders could be sintered to transparency (transmittance values of 82% in the infrared region and 56% at 680 nm) by SPS at 1500 °C for 3 minutes, keeping a constant pressure during the sintering cycle of 50 MPa and a heating rate of 100 °C min^{-1}.

4.1.6. Zirconia

As a hard-to-sinter ceramic, tetragonal zirconia polycrystal (TZP) samples are usually opaque even after high-temperature sintering because the existence of residual pores considerably scatters incident light and deteriorates the transparency. However, although it is possible to control the porosity through the sintering conditions (for example, using HP [Duran et al., 1989] SPS [Casolco et al., 2008] and HIP [Klimke et al., 2011]), it is still very important to keep the grain size as small as possible. Analogously to alumina, the non-cubic crystal structure leads to an optical anisotropy, birefringence in TZP is one order of magnitude higher than that of alumina, that causes diffuse scattering at grain boundaries. It has been shown [Zhang et al., 2011] that, compared to the method of pre-sintering plus HIP, high-pressure spark plasma sintering (HP-SPS) can be an extremely simple and effective route to obtain highly IR transparent tetragonal ZrO_2.

4.1.7. Magnesium aluminate spinel

Magnesium aluminate spinel is a ceramic material showing a large transparency range, covering from 0.2-5 μm. The densification to transparency (50% at 550 nm) by SPS of a fine-grained high-purity spinel was successfully demonstrated [Morita et al., 2008]. The temperature used in that study was 1300 °C with a holding time of 20 minutes and a low heating rate: 10 °C min^{-1}. Consequently, the versatility of SPS concerning heating rates can also be considered as a key factor to obtain transparent ceramics as it may cover a wide range of heating rates. Other works [Frage et al., 2007] showed very high optical transmissions in the visible range of spark plasma sintered powders of magnesium aluminate spinel powders doped with LiF. Undoped powders sintered at the same temperature of 1220 °C showed a smaller light transmission.

4.2. Biomaterials

Bulk hydroxyapatite (HA) compacts and composites with HA matrix may find a wide variety of applications in implantology [Martz et al., 1997]. However, HA shows a limited stability at high temperatures and will dissociate into tricalcium phosphate and tetracalcium phosphate at 1300 °C in air or 1000 °C in vacuum [Chaki et al., 1994]. On the other hand, high temperatures and long sintering duration required for consolidation of HA powders by conventional techniques often result in extreme grain coarsening or surface contamination, which can degrade the desired mechanical properties. These problems can be alleviated by using spark plasma sintering to avoid exposing the compacts to high temperatures for a long duration. It has been shown [Gu et al., 2002] that it is possible to sinter HA powders by SPS at temperatures as low as 950 °C while still keeping a good performance in terms of fracture toughness, microhardness and Young's modulus. Densities obtained at 950 °C after 5 minutes of holding time reached 99.6%. Also, these authors reproted that no phase change was detected at 950 °C, whereas HA started to decompose at 1000 °C. Also, no noticeable grain growth was observed at sintering temperatures between 850 and 1100 °C.

However, the intrinsic mechanical properties of HA, particularly its low strength and high brittleness, may still limit the applications of HA ceramics in loadbearing areas of the human body that HA should be strengthened. One attractive way to overcome these mechanical limitations is to use bioactive HA's ceramic/metal composites so as to achieve the necessary mechanical strength and bioactive properties at the same time [Lynn et al., 2002], which include that the incorporation of bioinert ceramics and the addition of biocompatible glass into HA matrix [Gautier et al., 1999]. However, due to the addition of a secondary phase, the phase changes of the composites at the usual sintering temperatures may still take place. For this reason, the sintering by SPS of TiO_2/HA composites was studied [Que et al., 2008]. These authors successfully prepared TiO_2/HA composites by combining high-energy ball milling with SPS. Their results indicate that the addition of TiO_2 had a positive effect on improving both the hardness and the Young's modulus of the HA, while still keeping a bioactivity as confirmed by in vitro tests.

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4.3. Materials for nuclear energy applications

The encapsulation of radioisotope materials such as $^{238}PuO_2$ and $^{241}AmO_2$ within tungsten cermets is of particular interest for the production of radioisotope heat sources for thermal management and radioisotope power systems [O'Brien et al., 2009]. The production of nuclear fuels for fission reactor systems based upon W–UO_2 or W–UN cermets is also of particular interest for increased operational safety and security. Conventional sintering techniques (such as hot isostatic pressing) required processing of materials at temperatures that were high enough to exceed the dissociation temperatures of the radioisotope oxides, which would lead to the formation of complete or partial non-cermet regions such as those described in the fuel element development summary in the General Electric report on the development of the 710 High-Temperature Gas Reactor [General Electric, 1967]. However, it has been shown in the literature that SPS is also able to densify materials at lower temperatures and shorter times than those required in conventional processes. Also, the presence of large grains observed in sintering processes such as hot isostatic pressing lead to a loss of mechanical properties. Spark plasma sintering minimizes grain growth and it is therefore ideal to be used in materials requiring a top mechanical performance. Other works also reported on the encapsulation of plutonium dioxide or americium dioxide within a tungsten-based cermet using the SPS technique [O'Brien et al., 2008].

4.4. Materials with low coefficient of thermal expansion

Materials with a very low coefficient of thermal expansion (CTE) are of great interest because of their many different applications, from cookware to aerospace applications. An interesting route to obtain this kind of materials is based on the combination of phases with positive and negative CTE [Garcia-Moreno et al., 2010]. The lithium aluminosilicate family (LAS) is the most studied system to prepare materials with very low CTE properties. Eucryptite and spodumene are the most used and studied phases with these characteristics [Moya et al., 1974] and it is well known that sintering of these materials to obtain dense ceramic bodies is quite complicated [Mandal et al., 2004], due to their narrow range of sintering temperatures and the easy formation of a vitreous phase. Therefore, by conventional methods of pressureless natural sintering, the ceramic materials thus obtained have usually low mechanical properties and Young's modulus. The fabrication of submicron LAS-alumina composites by spark plasma sintering has been suggested as a solution to this problem [Garcia-Moreno et al., 2011]. They found that it was possible to sinter the composites to the theoretical density, reaching higher strength values at lower temperatures than required by conventional sintering. In particular, composites comprising 15.65 wt.% alumina and 84.35 wt.% β-eucryptite gave the closest to zero CTE value in a wide temperature range.

5. Past, present and future of SPS process

5.1. Origin of spark plasma sintering

As previously mentioned, the technology related to current assisted sintering processes started in the late 1930s when a sintering process using electrical heating was introduced in

the United States. In Japan, a similar process based on the pulsed current applied sintering method was researched and patented in the 60s and is known as spark sintering [Inoue, 1963] [Inoue, 1961]. Nevertheless, the lack of application technology at that time limited fields where it could be applied. Unsolved problems associated with industrial production, equipment cost and sintering efficiency were key points. There was little literature on research into this process until the latter half of the 70s. The second generation was developed from the middle of the 80s to the early 90s. These units were small experimental systems with maximum sintering pressure of around 5 tons and pulse generators of up to 800 A, used primarily for materials research. The emerging of SPS process took place along the 90s when the third generation of this advanced technology was developed. These systems had large DC pulse generators of 10 to 100 tons and 2,000 to 20,000 A and more. They gained a reputation as new industrial processes for synthetic processing of gradient and composite materials [Omori et al., 1994] [Omori et al, 1994].

The evolution of interest in SPS process can be followed by analysis of fair indicators such as patents or scientific publications. The explosion of scientific research on this technology started less than two decades ago and it continues nowadays. In Figure 10, it can be observed the number of published items and citations in scientific papers with "Spark Plasma Sintering" as topic [Wok, 2012].

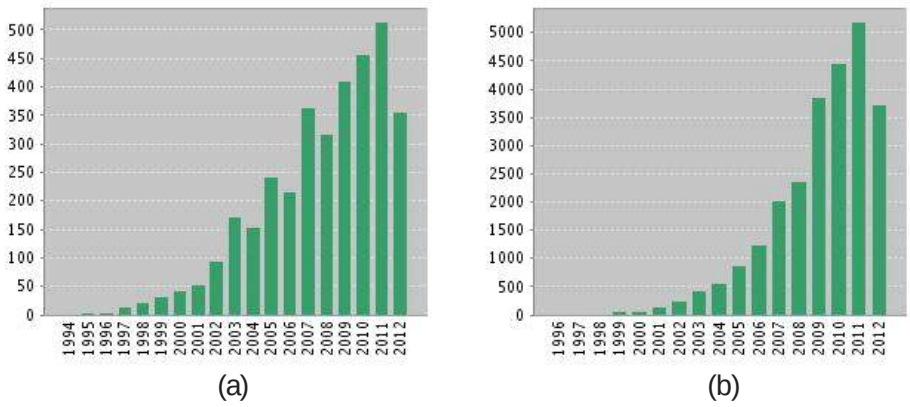

Figure 10. Published items (a) and citations (b) of Spark Plasma Sintering topic papers

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The excellent properties that can be obtained when this technique is used for consolidating materials leads to an exponential growing of research publications on this topic. It has passed from an exotic technique at the beginning of 90s to a fundamental tool for advanced material preparation, especially in the case of nanostructured materials. But, the interest in this technology is not limited to research area. Thus, 30% of patents related with SPS technology have priority dates of less than 10 years. Moreover, SPS has been progressively incorporated as one of the key steps in patents for obtaining advanced materials, especially for ceramic and metallic materials. The use of SPS for solving very diverse technological problems is a reality and first commercial products based on this technology are already in the market.

5.2. Industrial production requirements

As it was previously mentioned an enormous amount of reported FAST/SPS applications are still in the area of material development and more than enough opportunities for an industrial implementation were generated. Taking the next step forward to an industrial production of novel materials by FAST/SPS is currently highly dependent on the availability of suitable equipment. The industrial application of the FAST/SPS sintering method for the rapid consolidation of novel materials require special features, which have to be fulfilled by the equipment and are different from the requirements of scientific work to some extent. The main issues are discussed below:

5.2.1. Electrical output power

In order to assure a cost effective production, equivalent with high throughput (amongst other things), a sufficient electrical output power must be provided by the system. It is important, that the electric losses in the system are low in order to generate high heating power at the location, where it is needed. The actual value of the required power depends on the size and material of the powder compact and the pressing tool as well as on the intended heating rates and maximum temperatures.

5.2.2. Flexible power supply

Depending on the type of the powder, several different sintering mechanisms are possible. Some of them can be influenced by the type of the heating current. Therefore a power supply with high flexibility is important in order to achieve optimum sintering results in terms of throughput and material quality. FAST/SPS systems are capable of generating a wide range of pulsed DC current with computer controlled, arbitrary pulse parameters to the point of pure DC current (examples see Fig. 11).

5.2.3. Precise temperature measurement & control

The correct sintering temperature is the most important process parameter besides time and heating rate. Due to a special design FAST/SPS systems are measuring the temperature in the vicinity of the powder compact centre, which gives a much more significant value than the measurement of the die temperature [Vanmeensel et al., 2005].

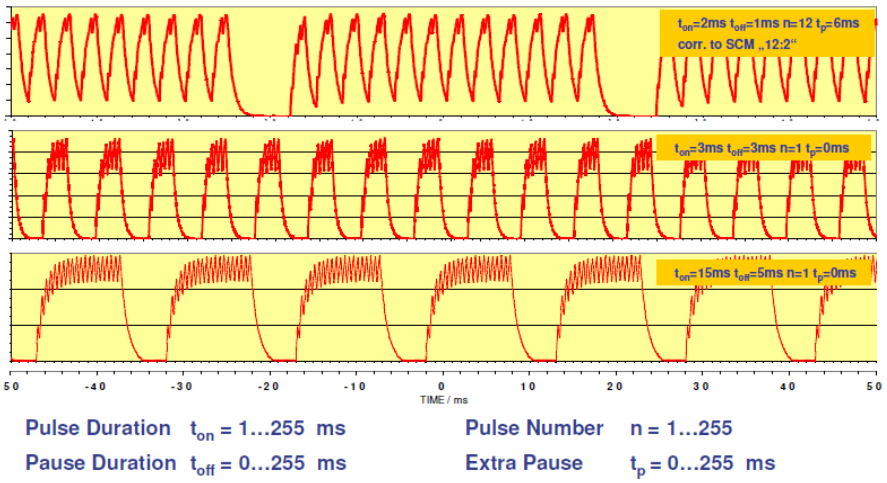

Figure 11. Flexible output of pulsed DC current

5.2.4. Optimized pressing tool systems

Due to the special construction of FAST/SPS systems, the pressing tool system, consisting of the two pressing punches, the die and other auxiliary components, is the “heart” of the system, because it not only contains the powder compact but also acts as the “heater” (in interaction with the compact). Even though the temperature gradients in the system are significantly lower than for conventional sintering methods, e.g. hot pressing (see Fig. 12), a design optimization is advantageous anyhow, especially if higher heating rates, minimized dwell time and optimum material quality are desired. A helpful tool for design optimization is the numerical simulation (finite element method “FEM”) of the heating behaviour, taking into account the temperature dependent thermal and electrical properties of the applied tool materials as well as the powder compact [Vanmeensel, et al., 2006]. As an example Figure 12 shows the temperature distribution in a pressing tool system containing two powder compact circular disks of 200 mm diameter after heating to 1500°C within 12 min and 5 min dwell time. With the standard tool design (left) the remaining temperature difference in the compact amounts to 160 K, which can be reduced to 60 K by design optimization (right).

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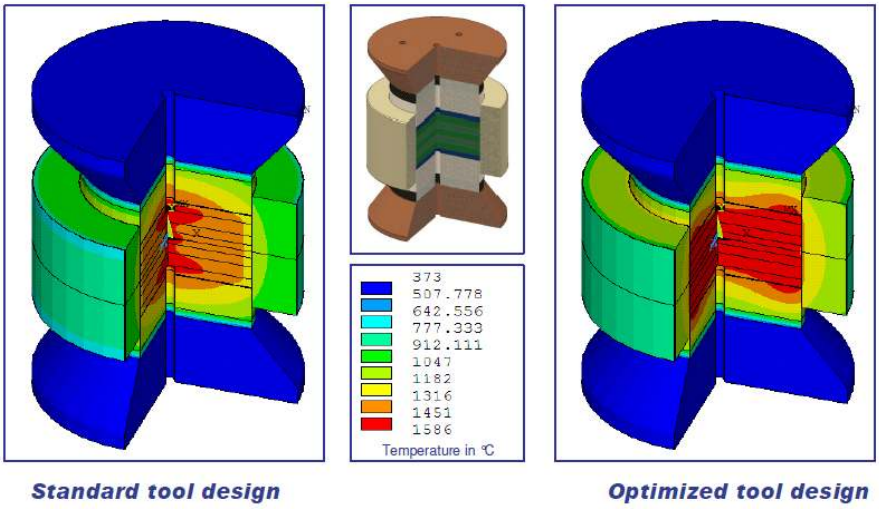

Figure 12. Temperature distribution in standard and optimized pressing tool system

The benefit of optimized pressing tool systems is a superior material quality and homogeneity, e.g. reflected by an even distribution of high hardness values across the diameter of a 200 mm circular disk compared with the standard pressing tool situation (Fig. 13). Furthermore the highest heating rates made possible that way are an essential condition for the realization of nano-structured materials, which are often impossible to sinter by conventional methods due to significantly longer sintering cycles.

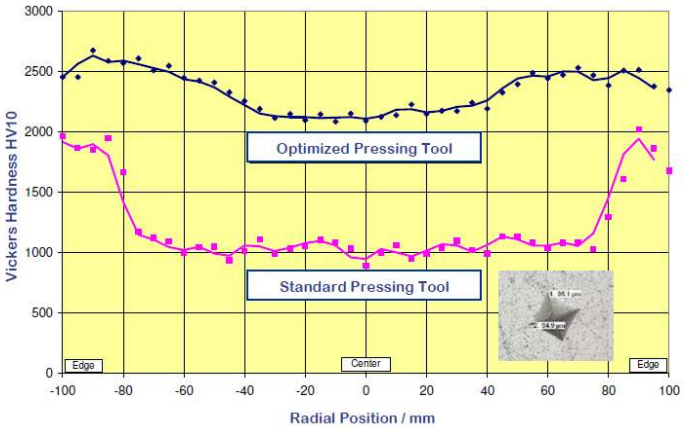

Figure 13. Hardness distributions generated by standard and optimized pressing tool systems

5.2.5. Hybrid heating

The so called "Hybrid Heating" is a combination of the FAST/SPS method with one or several additional heating systems, which act usually from the outside of the pressing tool systems, as illustrated in Figure 14. Thus the thermal gradients of FAST/SPS, which are directed from from the inside outwards typically, can be compensated by the inversely directed gradients of the additional heating system. As shown in Figure 15 the superposition of the gradients (left side) results in an extensively minimization of these gradients (right side). This allows further enhancement of the heating rates at simultaneously optimized homogeneity with all the advantages pointed out before.

A practical example showing the positive effect of hybrid heating can be found in Figure 16, which compares the sintering behaviour of rectangular plates made of binderless tungsten carbide (size 150 x 175 mm). The light grey curves show the densification byuse of FAST/SPS, whereas the dark grey curves show the enhanced sintering behaviour by use of hybrid heating.

5.2.6. Fast cooling system

The production capacity of an industrial FAST/SPS system is not only governed by the maximum possible heating rate and a minimized dwell time, but also by a fast cooling facility, which allows early discharge of the completed pressing tool. This is realized by an additional cooling chamber, separated from the actual sintering chamber by a gas/vacuum-proof, gate and equipped with special fast cooling rams. An automatically working handling system shifts the hot pressing tool system from the sintering chamber to the cooling chamber. After automatic closing of the gate the sintering chamber is ready for charging the next sintering cycle during cooling of the previous pressing tool.

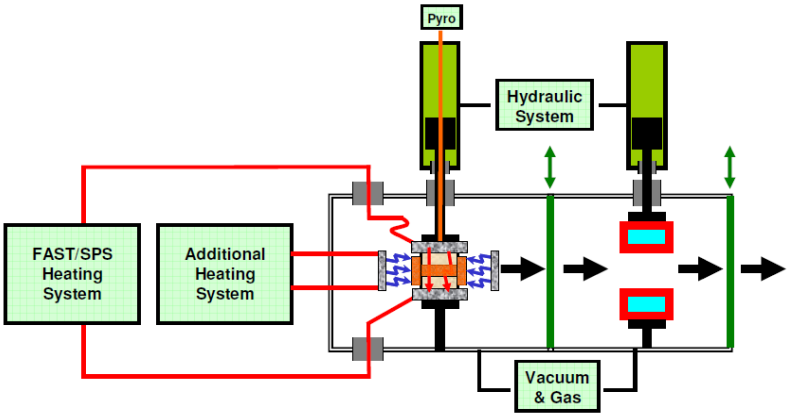

Figure 14. Schema of an industrial high throughput sintering system with hybrid heating, separate cooling chamber and semi-continuous operation facility

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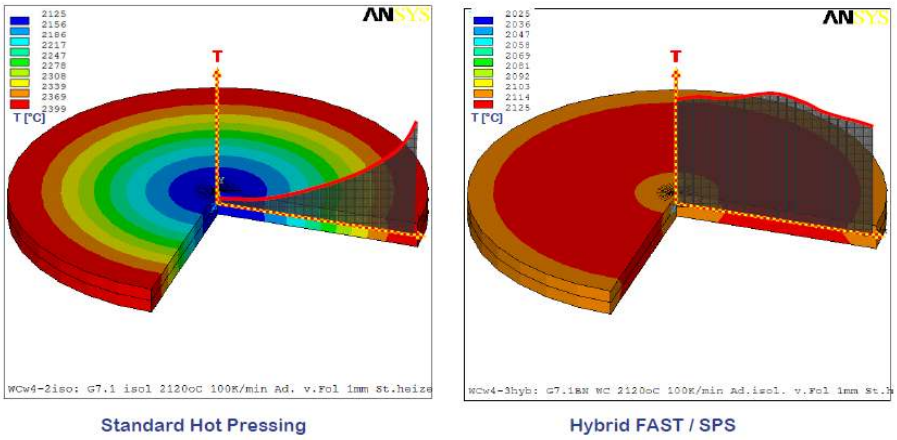

Figure 15. Compensation of residual temperature gradients by hybrid heating

5.2.7. Automatic operation

In order to realize a cost efficient industrial application of FAST/SPS sintering systems, the automation is an essential prerequisite. An important step is the semi-continuous operation mode mentioned above in conjunction with the fast cooling system. Due to a combination with robots and manipulators a fully automatic operation can be realized.

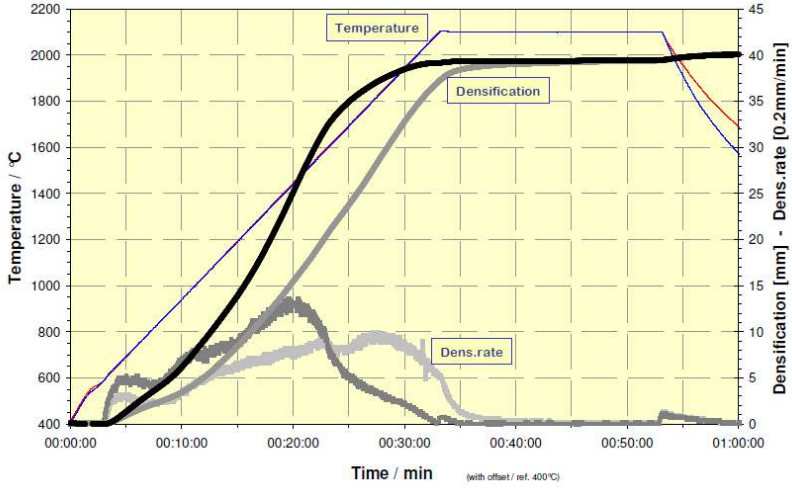

Figure 16. Comparison of sintering behaviour using FAST/SPS (light grey) and hybrid heating (dark grey)

Since 2010, the Nanomaterials and Nanotechnology Research Center (CINN-CSIC), in collaboration with FCT Systeme, the SPS technology leader in Europe, are working in the development of new advanced multifunctional materials obtained by using hybrid heating equipment. The potential applications of the components developed are very diverse covering fields such as space and aeronautics, automation, mechanical engineering or biomedicine. As examples of technical prototypes already developed, they can be mentioned ultrastable components for satellite mirrors and high precision and accuracy optical devices, ultrahard protection components, nanostructured cutting tools or biomaterials.

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