Sintered Materials on the Way to Production by Means of Modern SPS Technologies

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Introduction

Spark Plasma Sintering (SPS) – also known as FAST (Field-Assisted Sintering Technology) – is an innovative sintering technology that is becoming increasingly important in the processing of numerous materials, e.g. nanostructured materials, composite materials and gradient materials. The process is based on a modified hot pressing process in which the electric current does not run through an external heater but directly through the tool and the component. Thanks to the application of a pulsed electric current and the resulting "spark plasma effect", very fast pre-heating times and therefore short process cycles are realized. As a result, grain growth and the development of equilibrium states can be suppressed, which enables materials with previously unachievable compositions and properties, materials in the submicron or nano-range and composite materials with unique/unusual compositions.

Building on decades of experience and the successful application of its classical hot pressing system, FCT Systeme GmbH began around eight years with the development of this sintering process, a process already considered extremely promising from the start. The basis was the idea of developing very fast sintering processes to open new routes to more cost-efficient production of improved sintered materials on the one hand and to enable the production of materials that had so far not been possible to consolidate with the usual consolidation processes on the other.

The SPS/FAST Technology

The basic theory of SPS/FAST heating depends on the pulsed current being introduced through the tool to the grain boundaries of the particles of starting powder and leading to partial heating and generation of an electric field with a plasma effect. Here the type and form of the electric pulse, as well as its duration and level adapted to the morphology of the starting powder play a crucial role in achieving the required SPS effect. In how far the theoretical assumptions can be transferred into practice on a 1:1 basis has not been conclusively clarified in all scientific details. Observations made during the consolidation process, however, have indicated very strongly the existence of the presumed cause-effect relationships. This applies particularly to materials that conduct or partially conduct electric current, but also to ceramic materials, which only exhibit electrically conductive properties in the high-temperature range. [1, 2]

The power component as used successfully in FAST plants today enables – despite the high available pulsed power – symmetrical loading of the supply network and prevents phase displacement at the mains supply (cos φ >0,98), which is a great advantage especially at the high operating powers like those needed for industrial application (up to 500 kW).

Already in the initial phase of the development work, it was soon realized that special attention had to be paid to the forecast narrow relationship between the tool design, the component material (compact) and the energy supply to the plant. Detailed knowledge of the influencing parameters – both process engineering and material-related parameters – is the precondition for selective and successful development of the FAST heating process. From the start, therefore, highest priority was given to the simulation (modelling) of the heating process, which will be discussed later. Even the initial work proved the expected and most important difference to classical sintering processes (especially to the hot pressing system that is related to FAST technology), resulting from the direct heating of tool and compact with pulsed direct current. With the use of homogeneous model materials and the help of the FEM method (finite element method) for simulating the heat distribution in the tool and test specimen during active heating, important findings concerning the SPS/FAST theory could be obtained, on the basis of which further work on the practical application of the system (larger components) could be pushed ahead. [3-6]

In the scope of a European Research Programme on the development of the FAST technology (2002 – 2006), the particular focus – also on account of the company's core expertise – was on the consolidation of ceramic materials and composite materials for processing temperatures to 2200 °C.

Already in September 2003, after a relatively short development time, the first test plant could be completed – as the first European proprietary development. It is equipped with a power element with 8000 A, current intensity at 60 kW and allows a maximum compact diameter of 80 mm.

SPS/FAST Plant Development

Based on the findings obtained from the two-year development phase, work started on the specific build-up of the company's own "FAST" family of plants, the core of which is oriented to the requirements from research and the user industry.

The SPS/FAST plants sold worldwide by FCT Systeme GmbH, which are

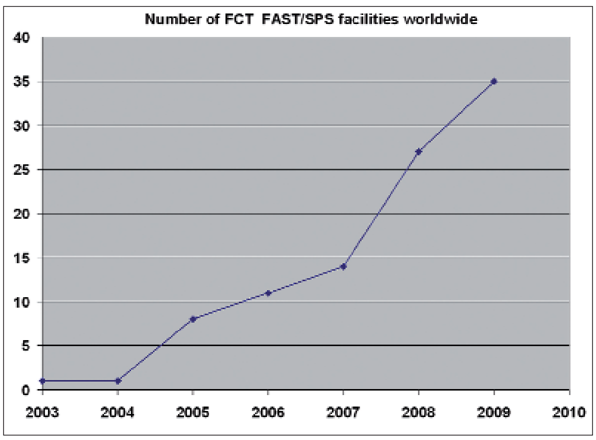


Fig. 1
Number of FCT-SPS plants worldwide

Fig. 2
FAST (SPS) production plant: HP D 250/C, semi-continuous operation

used very successfully in research and development as well as in industry (up to 400 t pressing force), are shown in Fig. 1.
Already after a short time, from industry came the call for more differentiated plants. First came a demand for smaller plants that enable a high output with a short cycle duration and secondly came a demand for large plants for application tests with large-sized components. Striking was also the interest of industry-oriented institutes in large plants with pressing forces up to 250 t for component-oriented development work.
Two key concept development directions have been systematically pursued by FCT Systeme GmbH:

- A quasi-continuously operating FAST plant for diameters to 300 mm and cycle times of 15 minutes with air lock technology for continuous operation, as well as
- High-speed plants based on TPA technology with cycle times from 10 seconds to 3 minutes.

Both types of plant are designed for industrial production.
For the industrial application of material-specific development work, today FAST plants with 2500 and 4000 kN press force, at 60 000 A max. pulsed current and 400 kW power are available. Such a plant (HPD 250) has been used for several years at the Fraunhofer IFAM, Dresden, for conducting component-oriented fundamental tests.
Fig. 2 shows a semi-continuous HPD250C production plant like that already used frequently in industry, for instance, for the economic production of sputter targets (Fig. 3) and composite materials.

Influence of Temperature Measurement

As in all thermal consolidation processes, temperature measurement is of the utmost importance. On account of the exceptionally high speed of the FAST processes, well-established temperature measurement processes are often no longer usable. So sometimes it may even be necessary to fall back on comparative measurements (energy input) – ultimately the only practicable solution at very high processing rates. Crucial for the efficiency of the temperature measurement is the positioning of the measuring points, to record useful measured temperature readings that are “physically clean” and can be correlated.
During the classical hot pressing process, with the introduction of Joule’s heat from outside (with induction or resistance heating), a considerable temperature gradient (spatial temperature difference) develops between the edge areas and the core of the compact, and this is especially so especially in the case of large-volume components. On account of this fact, the production of large-volume components by hot pressing is limited in terms of component homogeneity and material properties. In addition a hot pressing cycle requires a comparatively large amount of time as the actual pressure can only be usefully applied after an appropriate temperature balancing time.
These two weak points of the hot pressing system – inhomogeneity of the component and long cycle times – can be avoided with SPS/FAST processes. Here the force can be applied either in one stage (full pressure from the very start) or in more than one stage, the former being preferable providing process and press tool design is optimal (Fig. 4).
On account of the possibility of selectively conducting the flow of heat into the pressure ram and/or the surrounding press shell, it is possible both with electrically conductive and non-conductive materials to generate an almost homogeneous temperature distribution in the compact. However, this becomes increasingly difficult with increasing component size. For compacts with a high axial span, for a particularly thick-walled press shell is necessary, even this process reaches its limits. For components from around 100 mm axial span, the application of a new process should be considered, i.e. the SPS hybrid technology described later.
For the inevitably high preheating rates necessary to achieve the targeted material properties combined with shortened cycle times, an optimal temperature measurement

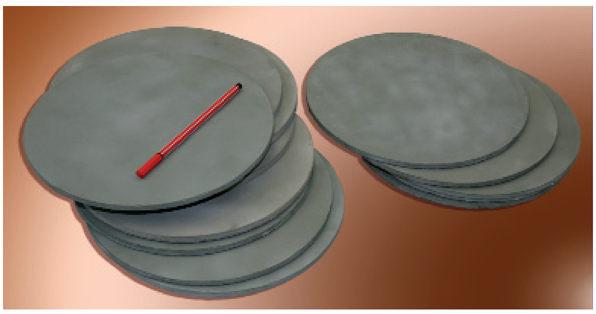


Fig. 3 Sputter targets (Ø 250 mm x 12 mm) sintered at 1800 °C and 35 MPa

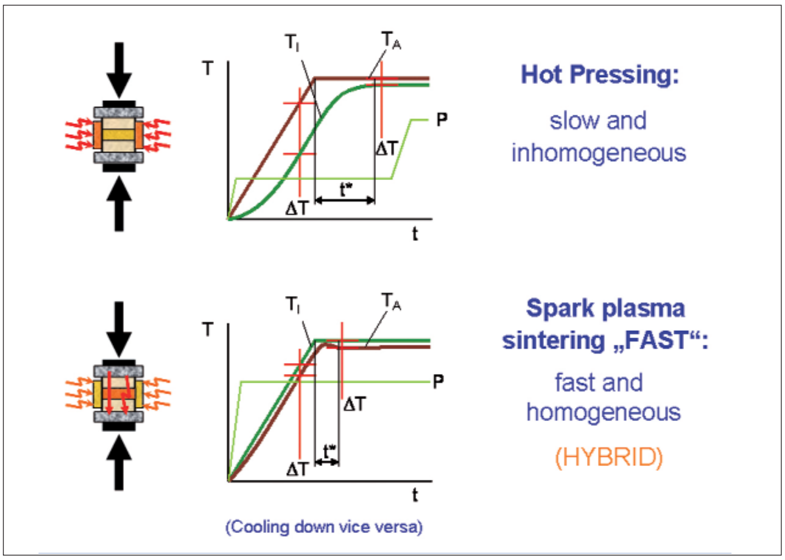


Fig. 4 Temperature homogeneity; hot moulding and FAST in comparison

is, as mentioned above, extremely important. So, derived from the company's own successful hot-pressing concepts, a measurement concept was developed, which enables measurement of the temperature directly at the workpiece to be consolidated. For the "fast SPS technology" (FASTSint®), this is the only obligatory route.

One essential potential advantage of the high-speed SPS technology is the possibility of producing very fine, dense microstructures and almost preserving the structure of the starting powder in the finished moulding. The reason for this is the short cycle times. It goes without saying that precise temperature measurement and consequently a sophisticated control system is indispensable for this.

High-speed process control taking into account the effective working temperature at the press tool also leads in SPS technology to much better hardness values, and this at significantly lower sintering temperatures. Important here are the very similar set-ups of the SPS and the fast hot pressing technologies. For the further development of the FAST concept, knowledge of the effective temperature at the workpiece is an important precondition for correct interpretation of the test results and therefore their transfer to field application.

Similar tests that confirm the above were conducted at the Fraunhofer IKTS in Dresden. Measured here was the influence of the temperature difference between the workpiece and press tool temperature on the microstructure and hardness of the material based on sliding abrasion.

Here the advantage of the SPS technology with regard to microstructure formation compared to commercial gas-pressure sintered materials becomes clear. And its potential too. All tests were conducted in relatively small pressing tools with diameters from around 50 mm. In the following, the temperature homogeneity in the compact is examined.

Tool/Material Interactions

The still relatively uncritical temperature distribution in small components – in the workpiece or tool – becomes a crucial criterion in larger components. Initial tests with roughly calculated tools on graphite basis have already revealed the problem of insufficient temperature homogeneity in the compact. Particularly striking is the influence of the temperature on the thermal and electric properties of the material to be consolidated. Great attention was consequently paid to this influence, specifically to the material changes during the consolidation process. This was very time-consuming and required the development of new measurement methods. Starting from the packing density in the precompacted state, these properties change by several powers of 10 during the compaction process, determined both by the temperature influence and the pressure as well as by the resulting density and microstructural state. Accordingly for "new materials", appropriate basic tests are necessary, to determine, at least roughly, the relevant properties and therefore to enable theoretical calculations concerning the course of the SPS process.

The theoretical work now available combined with the existing new measurement methods and the application of finite element simulation (Fig. 5) provide an important basis for the design of the component material/tool material system. The specification of tool geometry and material, especially the die and the plant die is of crucial importance here. The works so far refer to situations in which the filled mould is inserted in the press prior to the process and then removed again after the process. Work currently starting is aimed at optimizing permanently mounted FAST tools in respect of their thermal and electric properties so that a stable operating state is achieved in a repetitive process, leading to homogeneous compacts while at the same time not overloading the FAST press in continuous operation.

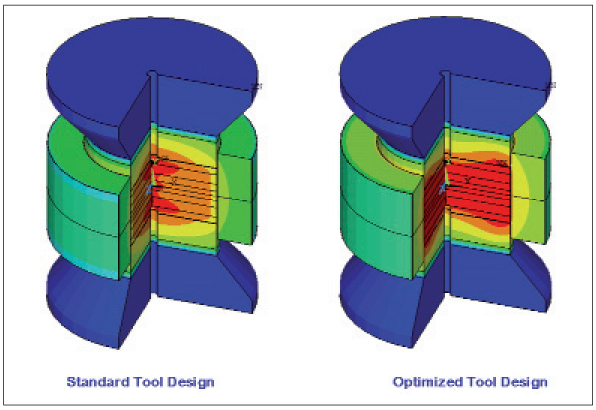


Fig. 5 FEM simulation of the FAST preheating phase

The use of graphite materials for the tool design is a logical and in principle correct conclusion. Nevertheless graphite should only be seen as a basis for further developments, for some properties of graphite, for example its creep behaviour, transition resistances and above all wear behaviour prompt the search for more suitable materials to ensure reproducible continuous operation of the FAST process.

The strong dependence of the process efficiency on the electric properties of the tool and the "compact" (e.g. see Fig. 6) necessitates precise coordination of the process parameters. The working range of the FAST presses tends to be limited with regard to the maximum voltage and maximum current intensity by the achievable power values that can be ultimately input into the workpiece. Accordingly, the electric properties of the tool and the compact have a major influence on the duration of the entire process.

The electric resistance of the die and the material to be compacted in turn influence the geometric design and the possibilities for ensuring optimal press operation.

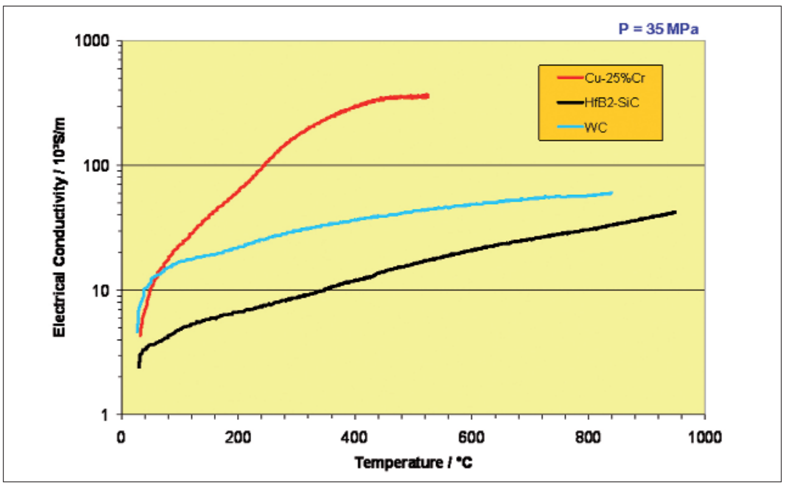


Fig. 6 Specimen resistance $Cu_{25}Cr = f(T)$

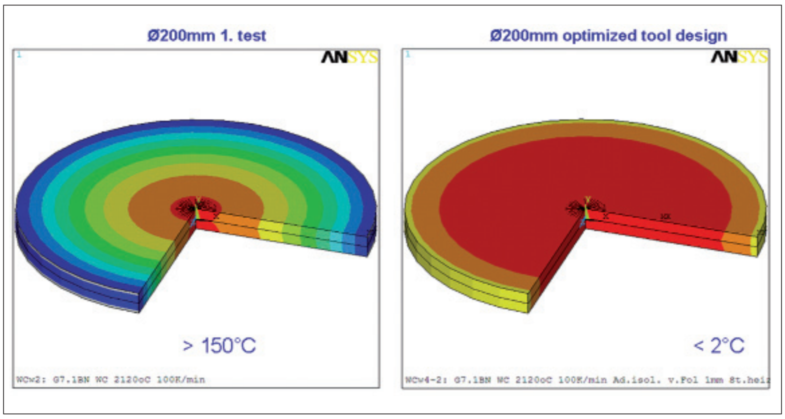


Fig. 7
Temperature distribution in a tool Ø 200 mm, 2100 °C, 100 K/min

The producing industry is increasingly calling for components with special properties, e.g. nanostructured microstructure or extreme fine-grainedness or extreme component homogeneity combined with full density. Extreme component properties naturally place extreme demands on the operating parameters, the tool design and material preparation.
The first tests with compacts (Ø 200 mm) made of submicron tungsten carbide (cobalt-free) resulted in – despite the high absolute density values (>99 % theoretical density) achieved - pronounced fluctuations in the density distribution which were consequently intolerable for the planned application. Workpieces with high homogeneity were mainly achieved based on the tool design. Today it is possible with tools to Ø 300 mm to achieve a good temperature homogeneity (deviations of less than 20 °C) combined with low density and hardness deviations (Fig. 7).
It should be emphasized again here that every component material requires the development of its own tool concept. Even slight variations in the raw material properties, which might well be tolerable with the use of other production methods, may in individual cases necessitate significant changes in the tool concept for the SPS/FAST process.
With the exception of hard metals, transition metal carbides and many borides, the direct passage of current through the material is limited. This has two consequences: for these materials, particularly with fast preheating rates and large specimen dimensions, thermal gradients in the material will result and secondly the actual SPS effects can hardly be expected [7].
The effects of the drastic reduction in the sintering temperature reported in the literature must be critically questioned. They are sometimes caused by the type of temperature measurement. Real effects are, however, the influence of the pressure on consolidation (similar to hot moulding) and prevention of the reduction of the sintering activity by surface diffusion and grain growth in the preheating phase.
In spite of this, we see great potential in this method for wide-ranging application with ceramic materials too. These are specifically:
materials that are currently hot-pressed (e.g. BN, TiB_2/BN composites, SiC-whisker-reinforced Al_2O_3, B_4C). Thanks to the achievable short cycle times, possibilities result for near-net shaping, with potential finishing savings. Initial tests at the IKTS have shown that the homogeneous consolidation of BN/TiB_2 composites is possible at least to specimen diameters of 80 mm and even non-rotationally symmetrical components can be compacted to form homogenous microstructures.
The short cycle times and the pressure, which can reach up to 100 MPa or more, allow the consolidation of nanomaterials with minimal grain growth. This enables relatively low-cost production of nanomaterials and components. Besides the production of the nanomaterials, the method can be applied to produce ceramic materials with defined structure by means of sinter forging (superplastic forming) [8-10].
The short cycle times in the minute range allow new material concepts for a wide range of applications in different fields (e.g. cutting and wear materials, biomaterials, multifunctional materials). For example, preliminary work is underway on the compaction of diamond or cBN/WC/Co composites [11-13], which cannot be produced with conventional methods. In connection with biomaterials, for example, there have been reports of ZrO_2-HAP composite materials with high strength [14]. These components cannot be sintered with conventional methods.
High-density functional materials [15] and even transparent ceramics (Al_2O_3, spinel) can be produced effectively with the method.
The method allows the sintering of components with built-up structures, e.g. multilayer systems or even spark plugs for diesel engines.

Other Examples of "FAST Activities"

Two essential core objectives formed the focus of the further development of the FAST plant concept: first reliable technical instruments had to be designed with the help of which the required maximum temperatures can be achieved without overloading the system. Secondly, the sintering cycle times were to be drastically shortened, to just a few seconds in some cases.
Besides the classical metallic and ceramic materials, functional ceramics are becoming increasingly important – for SPS/FAST a still largely unexplored area in respect of the use of this process engineering to obtain specific material properties. Current examples of this are electrooptical, piezoelectric and magnetic materials. Extensive research work in recent years has led to a sheer flood of publications on spark-plasma sintering, indicating the potential of this technology. In this context, readers are referred to current studies and work at the Fraunhofer Institutes IKTS and IFAM (Dresden).
The advances of recent years in thermoelectric functional materials have given rise to the hope that with the help of the FAST technology and the use of nanoscale starting materials, an important step can be made towards the practical application of this technology in the field.
Nanotechnological approaches have realistic chances particularly for functional materials, as much improved properties can be achieved with them. For thermoelectric materials, which convert current directly and reversibly into temperature differences, efficiencies were achieved with these that hardly seemed possible in the last five decades.
Composite materials on the basis of titanium or titanium alloys are inter-

esting structural materials for lightweight construction applications. As the titanium alloys exhibit outstanding specific strength, high ductility, toughness, corrosion and creep resistance, they are suitable as a matrix for composite materials with application temperatures of more than 500 °C. Best-known today are long-fibre-reinforced Ti-based materials.
Particle reinforcement has the advantages of a comparatively low-cost, powder metallurgy production route and isotropic properties. In addition, after the sintering process, the materials are easier to process further, e.g. by forging.
For the production of particle-reinforced titanium-based materials, commercially available TiAl6V4- (<100 µm) and SiC powder (F400) were used. The powder blends were prepared in the ratio TiAl6V4/15 vol. % SiC in a turbular mixer, These blends were filled – without undergoing any further pretreatment – into the graphite tools (Ø interior = 100 mm und 200 mm, 30 MPa) of the SPS system and steadily heated in a vacuum at 100 °C/min to sintering temperatures between 700 °C and 1100 °C.

Tungsten Carbide-Cobalt

High potential is assigned to FAST technology for influencing the hardness/toughness ratio of tungsten carbide-cobalt materials. Various research projects have shown that SPS/FAST technology can be used to achieve higher fracture toughness with the same Co content.
Another, very promising application of SPS technology is the production of sputter targets, which are needed as starting material for a wide range of coating processes, like, for example, PVD, laser or ion beam technology. Targets made of metals/metal carbides, etc., of ceramic materials or compositions that cannot be produced by smelting are usually produced by a powder metallurgical route by pressing powders or powder blends and subsequent sintering or hot pressing. The advantage of the FAST process is shown in the excellent homogeneity and high density of the achieved targets combined with very much shorter process times.
The following Fig. 8 shows the process sequence for the FAST consolidation of a Co-free tungsten carbide material for sputter targets with a diameter of 200 mm. With optimization, a special heating system (hybrid system) was developed, which can effect a considerable reduction of the temperature gradients in the component by means of an additional, supplementary heating system that uses an induction coil for additional heating of the – frequently thick-walled – press mould from the outside ("hybrid heating system").

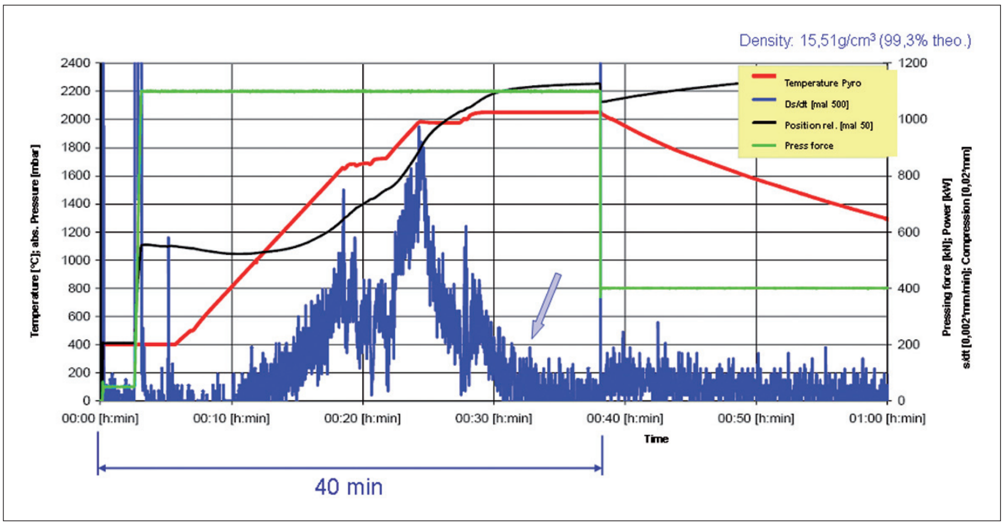


Fig. 8
Material: WC (Co-free), dimension: Ø 200 mm x 13 mm ("Twin"), SPS-sintered in the HPD 250

As in SPS system too, the realization of required component properties, such as hardness, wear resistance, etc. is possible even with a short residence time of the compact at an elevated temperature, the cycle times being minimized with the application of a special cooling system (cooling chamber). The prin-ciple is shown schematically in Fig. 10.
The users of this technology especially appreciate the advantages of the economic production derived from the time saving and the specific realization of required component properties even in larger size components.
Key focuses are the production of ceramic components for semi-conductor systems (e.g. bond heaters, etc.), large-size sputter targets as well as highest quality bulletproof materials (body armours). Several plants of this type to 400 t press force are currently in production.

The new "FASTSint®" Fast Sintering Process

The motivation for the development of FAST-Sint® was the need for a fast sintering process that is suitable for (e.g. injection moulded) components with relatively complex geometry. For SPS can generally only be applied to produce simple component geometries, i.e. only cylindrical, square and annular components with constant axial span.
FAST-Sint works with an extremely low thermal mass, so that extremely short sintering times are possible, which can be essential especially for nanostructured or imbalance materials. As the process works without the use of a moulding die, no tool costs are incurred and problems with tool wear for high unit numbers are avoided. Even chemical and thermomechanical incompatibilities between component and press tool (contact reactions, thermal expansion effects) are of practically no matter. FAST-Sint can be automated based on the introduced dry press mechanics/hydraulics, as shown schematically in Fig. 1. One test of the actual core technology is possible in a conventional FAST press of the HPD series so that corresponding sintering tests can be performed without the need for protracted development of special mechanics/hydraulics.
Suitable here are naturally materials in which the short sintering time leads to extraordinary microstructural properties, the starting powder being either in the submicron or nano range, without significant grain growth, so that excellent properties can be expected (Fig. 11).
First test series with SSiC and WC/10%Co materials have returned good results. Hard metal blades could be sintered in a heating time of just 100 s and 60 s holding time to exhibit material properties that are only matched in conventional production processes after two hours holding time. Fig, 11 shows in tabular form the sintering conditions applied and the material properties achieved for several test cycles in

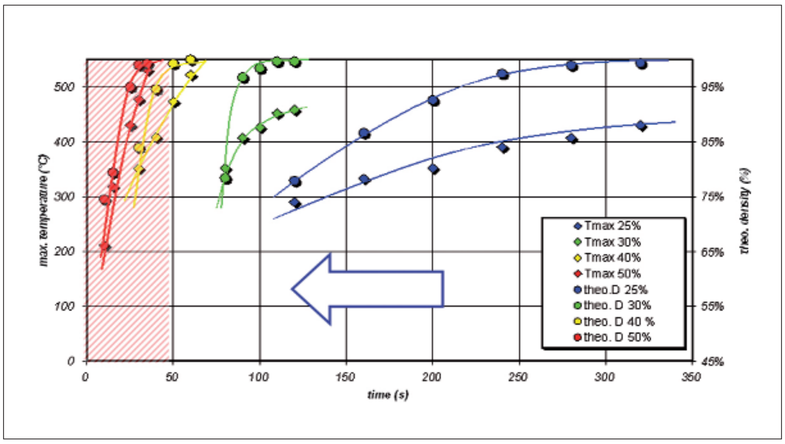


Fig. 9 Temperature and theoretical density as a function of time and heating current [19]

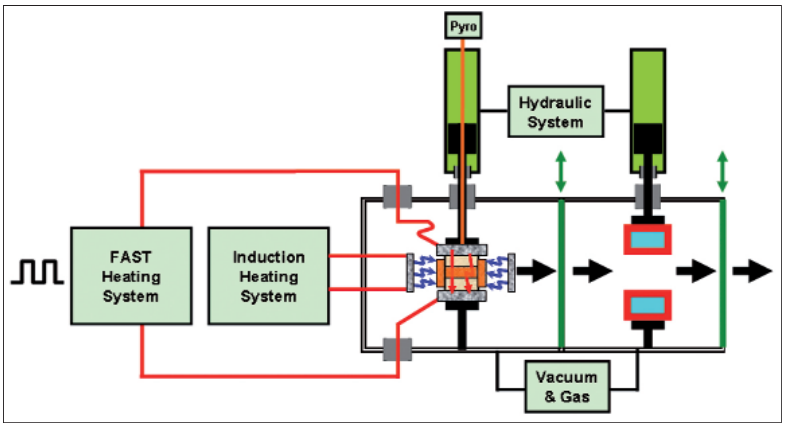


Fig. 10 Principle of the Hybrid-FAST technology for the production of large-volume components (from electrically conductive starting powders).

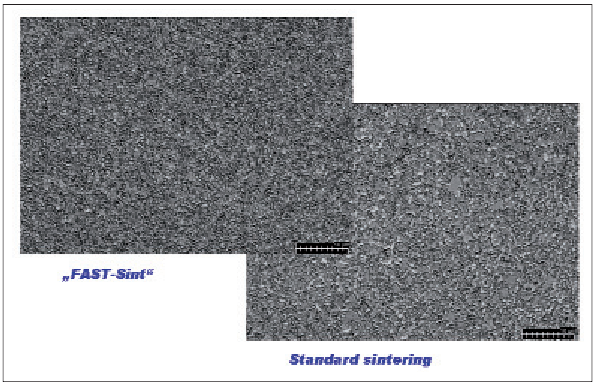


Fig. 11 (top) Microstructural comparison based on a WC/Co material (10 % Co) between FASTSint® (AO2, BOO, COO) and a "standard sintering process"

mized process control and an active sintering time of 40 minutes, a final density of >99 % of the theoretical density is achieved.

There is further potential for process optimization. Today, with optimal tool design, two compacts (152 mm x 175 mm x 12 mm) can be sintered in one cycle. With further development of the plant engineering (higher power), another shortening of the cycle time is probable.

The capability of the FAST technology is currently shown in the successful application of the findings so far obtained (component diameter 80 mm) to large-volume components, for example made of aluminium alloys, which can only be produced by means of powder metallurgical processes – as a starting compact for extrusion or forging forming.

So in an exceptionally short cycle time almost full consolidation of the compacts can be achieved (Fig. 9). [16-18]

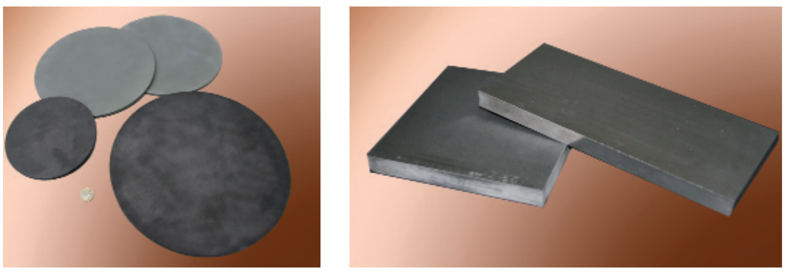
The most important argument in favour of SPS/FAST is, as mentioned earlier, the short cycle time combined with optimal material properties. The energy input in the compact is crystallizing as the main influencing factor. For the plant technology this means that the energy supply in particular must be even more flexible and more powerful.

The current cycle time for the production of forged compacts made of aluminium-silicon alloys (density >99 %) is below 90 seconds, with the use of a special method for "energy portioning" (energy input in the compact) with exact recording of the input energy.

The latest research project at FCT Systeme in the field of spark plasma sintering is termed "FAST²" with the objective of realizing even shorter cycle times (<10 s), on the basis of the dry pressing system (TPA-FAST). Here too the achieved material properties, which are far superior to those of cast components, encourage the application of laboratory findings in the field.

One example of the production of composite or mixed crystal materials is a titanium carbon nitride/ aluminium oxide, a material established today for cutting ceramics for machining, for example, hardened steel. By means of appropriate material and process optimization, it has been possible to realize compon-ent densities >99 %, with an active cycle time of less of three minutes. Further studies on this very promising approach are under way, especially in respect of the use of even finer starting powders.

The spectrum for further tests for the compaction of metallic and non-metallic materials by means of FAST technology is very broad. [20-25] Detailed descriptions of the individual possibilities would, however, far exceed the bounds of this paper.



Figs. 12 Typical components with "simple" geometries Ø to 400 mm

The New Hybrid-FAST Technology

Spark plasma sintering has essentially two fundamental limitations in respect of its applicability: component size and the electric properties of the material. A homogeneous microstructure is only achieved in comparatively small-sized components, as during sintering of larger components – from around 100 mm diameter – temperature gradients result, and the materials must be electrically conductive. To overcome these limitations and realize large components in a much shorter cycle

comparison with the conventional production method.
Current R&D projects on this process concern its transfer into field application and the influences on the material properties. [26, 27]

Outlook

The results so far obtained in connection with the FAST technology have aroused great interest in the continuation of this work, both in respect of fundament research with regard to new materials and material properties, as well as the field application of the research findings.
Applications for SPS/FAST technology can be found mainly in electric engineering, mechanical engineering, automotive and medical systems.
On international level, an extensive R&D project to last several years is currently commencing under the title "Nanoker", in the scope of which the FAST technology plays an important role especially in connection with the consolidation of nanoscale materials.
Generally, the most important research projects on German and European level concern nanomaterials with special properties. Material focuses are aluminium materials, composite materials and graduated materials as well as carbon nanotubes (CNT) such as B_4C-CNT, W-CNT, etc.
More and more industrially supported research programmes are launched with clear specifications for the targeted materials and components.
Besides the still important fundamental work on material development, the realization of more complex component geometries is at the forefront of future considerations. Up to now, only simple geometries (disks, rings, cylinders, and similar) have been realized (Fig. 12). First projects on the production of angular (rectangular and square) shapes have been successfully concluded. A solution must still be found to meet the important requirement for the realization of more complex near-net shape components (Fig. 13).
As industrial users of FAST plants most, above all, work cost-oriented, besides appreciating the improved material properties and the improved homogeneity, they attach great importance to shorter cycle times. As in mass production or mass use of the components in question, appropriate capacity rates per unit of time are necessary, the FAST technology is currently being transferred to TPA basic technology. This is in terms of the technology involved the most difficult part of the development work so far, but also the most forward-looking (Figs. 14/15). First successes have already been registered, but a few more years of intensive development work must still be expected. Such a system, with the help of which a faster changeover from low-temperature levels (500 °C) to temperatures above 2200 °C can be realized in several seconds, is currently being built. The main focus lies on the reproducibility of the processes, tool wear (service lifetime) and overmeasure.
Here too, on account of the required mechanical and electric properties of the press tool, tool optimization is a central interest, especially in respect of the high temperatures required.
Another system with high expected potential, which is currently in the development phase, concerns the fast consolidation of liquid-phase sintered materials, like, for example, Si_3N_4/YAG and similar. With the objective of obtaining a pore-free and homogeneous microstructure, the preforms obtained in the near-net shaping process are exposed to an additional gas pressure of 100 bar during reduction of the second phase. This technology is currently in the development phase.
The bottom line is that with all the considerations demanded by this process it should be noted that successful developments can only be expected, when apart from the already identifiable possibilities, the production costs can be lowered and/or the material properties significantly improved.
Further possibilities for the practical application of this very promising process in future can be keenly awaited.

Fig. 13 Complex near-net shape components

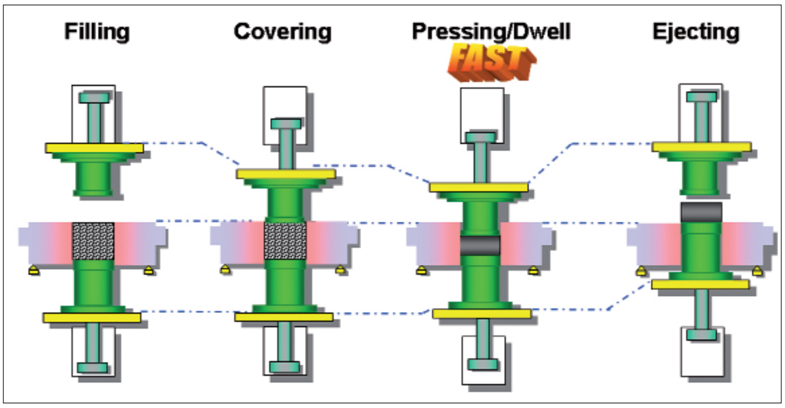


Fig. 14 Implementation of FAST system in the TPA system

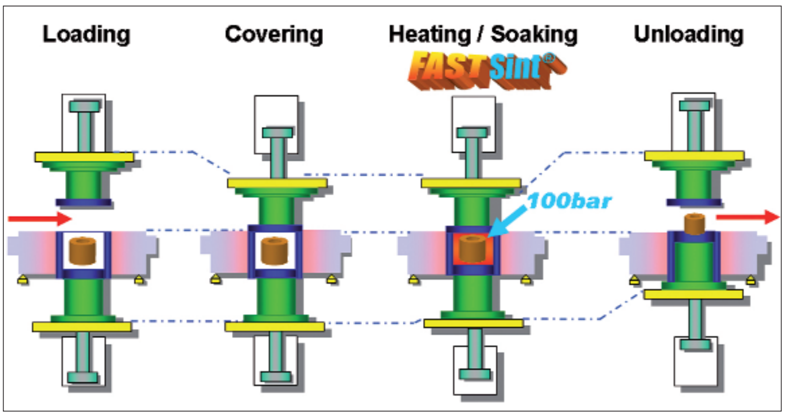


Fig. 15 Principle of the FASTSint® fast sintering technology

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