

RAPID SINTERING OF NOVEL MATERIALS BY FAST/SPS – FURTHER DEVELOPMENT TO THE POINT OF AN INDUSTRIAL PRODUCTION PROCESS WITH HIGH COST EFFICIENCY

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ABSTRACT

FAST/SPS is the state-of-the-art spark plasma sintering technology developed by FCT. It offers rapid consolidation of a wide range of powders from all fields of ceramic and metallic materials, enabling significantly improved or even completely novel materials. This was shown very frequently in the last decades, resulting in a huge amount of scientific papers, which report promising results at almost all times. But the main part of reported FAST/SPS applications are still in the area of material development, although 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. After a short overview of the FAST/SPS technology this paper explains the most important industrial production requirements, which has to be fulfilled by the FAST/SPS equipment. Subsequently examples for industrial applications will be presented, concluded by a preview of future developments.

INTRODUCTION

In the last decade many scientific papers and presentations were published, dealing with the application of spark plasma sintering¹. The rapid consolidation of powders is the most important advantage of the state-of-the-art spark plasma sintering technology FAST/SPS, offering significantly improved or even completely novel materials. A wide range of material types was investigated, metals, alloys and intermetallics as well as Borides, Carbides, Nitrides, Silicides and Oxides, plus all imaginable composites and special material systems. Most of the results are from scientific investigations, but nevertheless very promising and demonstrating interesting opportunities for a transfer to industrial production. But industrial production is really different from scientific investigation and requires suitable and individually tailored equipment, in order to realize a manufacturing process with optimum cost efficiency. The industrial success of FAST/SPS will critically depend on the availability of accordingly optimized equipment.

FAST/SPS TECHNOLOGY FOR RAPID CONSOLIDATION OF POWDERS

Spark plasma sintering as well as its state-of-the-art version, called FAST/SPS, are sintering techniques, which are deduced from conventional hot pressing². Hence FAST/SPS systems likewise comprise a water-cooled vessel, a hydraulic pressing system, and a computer-aided process control by means of temperature and force measurement & control as well as a system for vacuum and atmospheric control inside the vessel respectively. The big difference compared to a conventional hot press is the absence of a heating element as well as conventional thermal insulation of the vessel. Instead a special power supply system feeds high current into the water-cooled machine rams, which that way act as electrodes simultaneously, feeding the high current directly through the pressing tool and the containing powder compact. Figure 1 presents the principle of FAST/SPS.

This special construction leads to a homogeneous volume heating of the pressing tool as well as the containing powder by means of Joule heat. Hereby only small thermal gradients are generated even at

high heating rates, whereas conventional sintering methods are hampered by thermal gradients, allowing only moderate heating rates and requiring long dwell time for subsequent (but mostly incomplete) homogenization. The advantage of FAST/SPS is visualized in figure 2 in terms of the

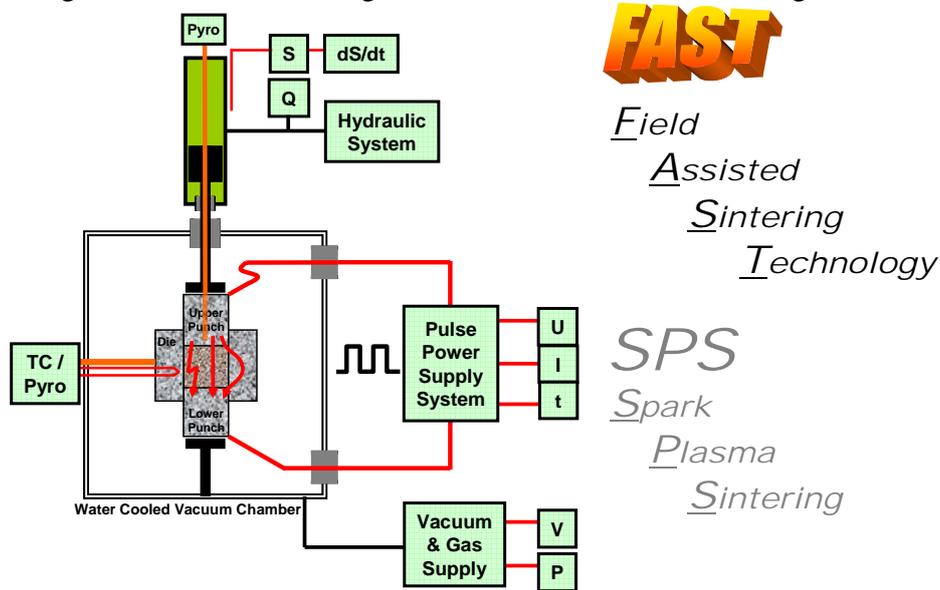


Figure 1. Schematic of FAST/SPS equipment

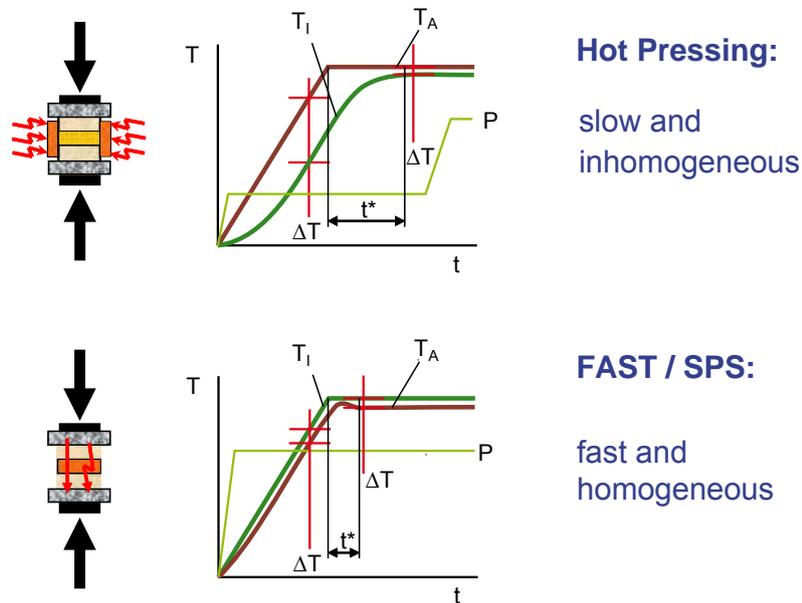


Figure 2. Comparison of FAST/SPS with conventional Hot Pressing

temperature of the center T_I compared with the edge of the powder compact T_A during the sintering cycle.

An additional advantage of FAST/SPS is shown in figure 3: 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. 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, e.g. electro-migration or microplasma-generation.

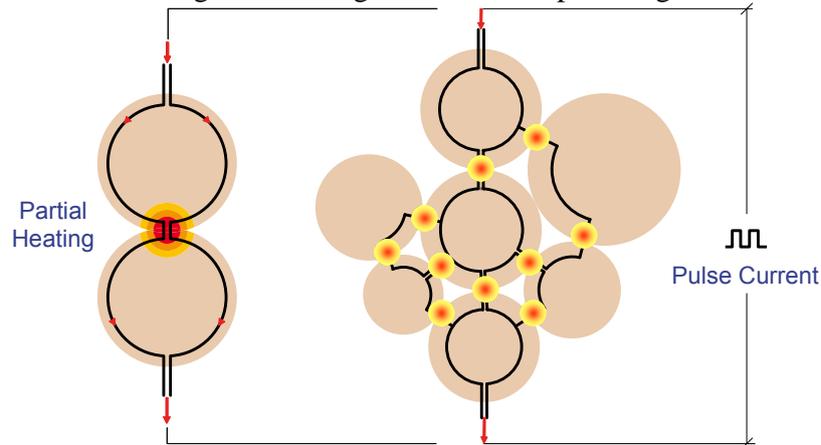


Figure 3. Energy dissipation in the microscopic scale

REQUIREMENTS FOR INDUSTRIAL APPLICATION OF FAST/SPS

As already mentioned 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:

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.

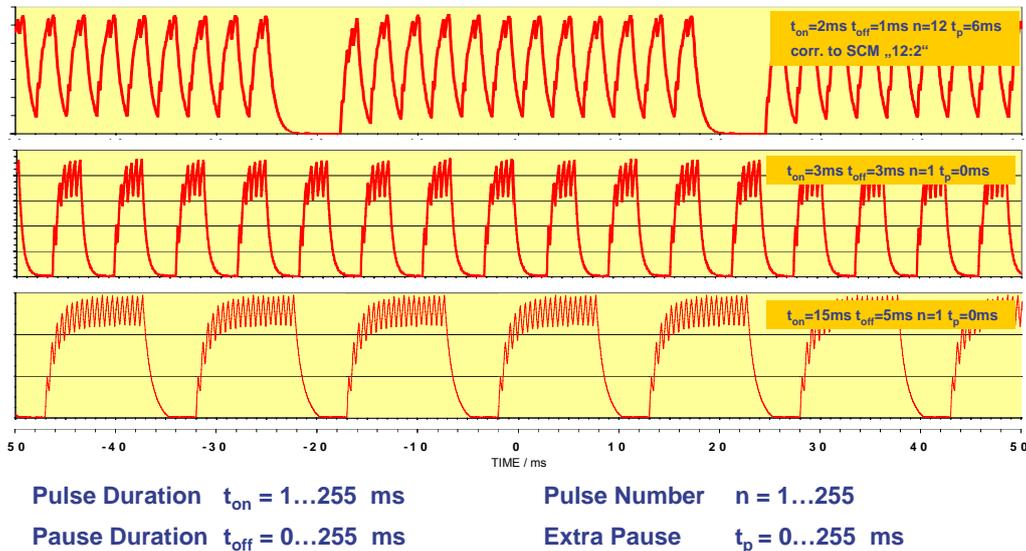


Figure 4. Flexible output of pulsed DC current

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 are 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 figure 4).

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 center, which gives a much more significant value than the measurement of the die temperature³.

Optimized Pressing Tool Systems

As pointed out before, 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 significant lower than for conventional sintering methods, e.g. hot pressing (see figure 2), a design optimization is advantageous anyhow, especially if highest 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 compact⁴. As an example figure 5 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).

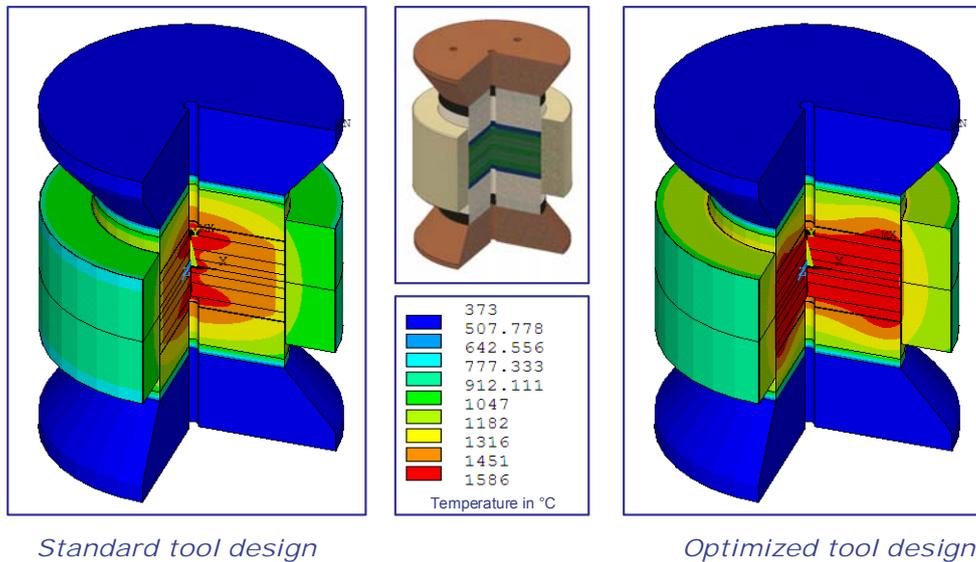


Figure 5. Temperature distributions in standard and optimized pressing tool systems
The benefit of optimized pressing tool systems is a superior material quality and homogeneity, e.g. reflected by an even distribution of high hardness values across the diameter of a 200 mm circular disk compared with the standard pressing tool situation (figure 6). 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 significant longer sintering cycles.

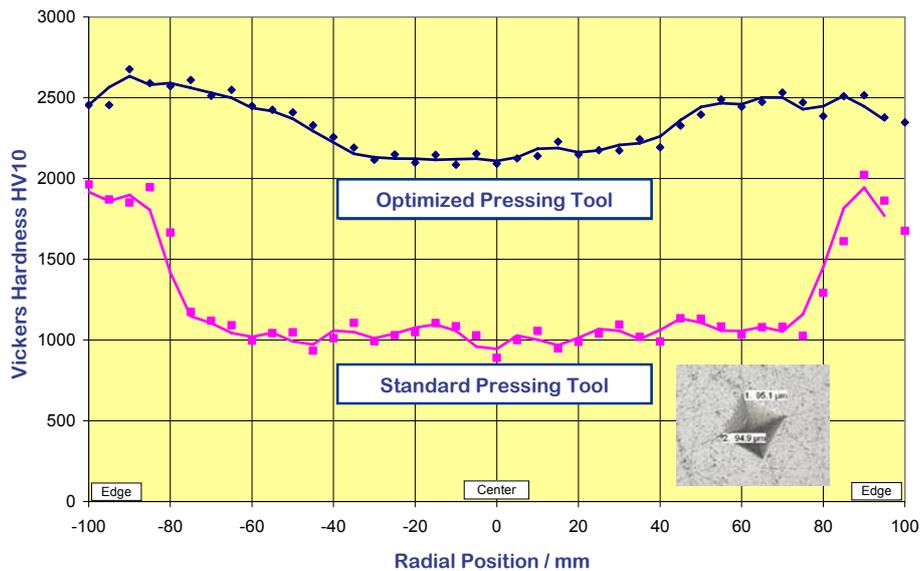


Figure 6. Hardness distributions generated by standard and optimized pressing tool systems

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 7. Thus the thermal gradients of FAST/SPS, which are directed from the interior to exterior typically can be compensated by the inversely directed gradients of the additional heating system. As shown in figure 8 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 9, 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 / densification by use of FAST/SPS, whereas the dark grey curves show the enhanced sintering behaviour by use of hybrid heating.

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 (see figure 7). 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.

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 10 shows as one alternative a 250 ton hybrid FAST/SPS production system according to figure 7, but additionally equipped with two ABB industrial robots for charging and discharging. A further example can be found in the next paragraph.

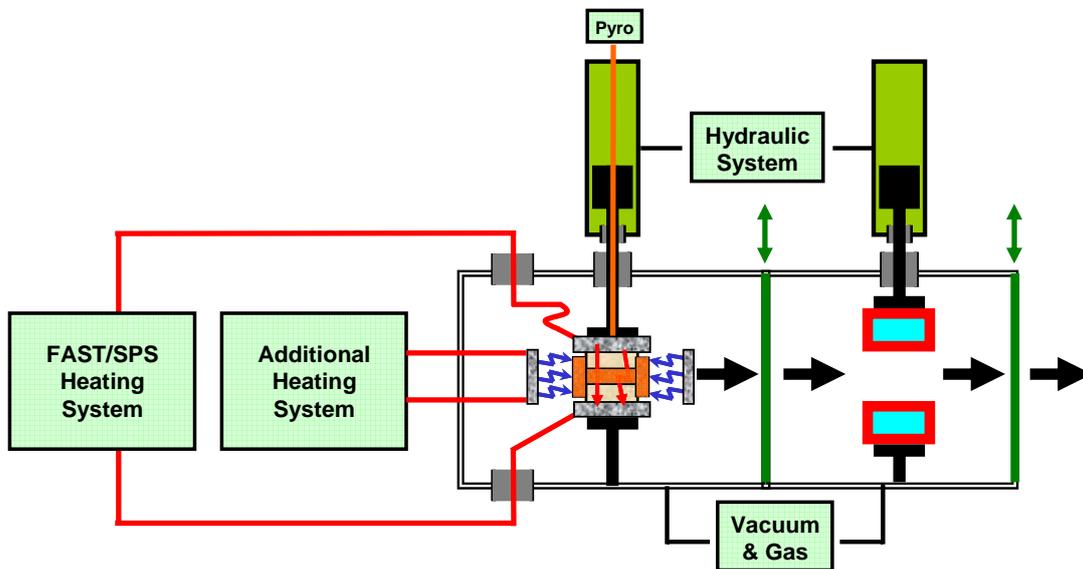


Figure 7. Schematic of an industrial high throughput sintering system with hybrid heating, separate cooling chamber and semi-continuous operation facility

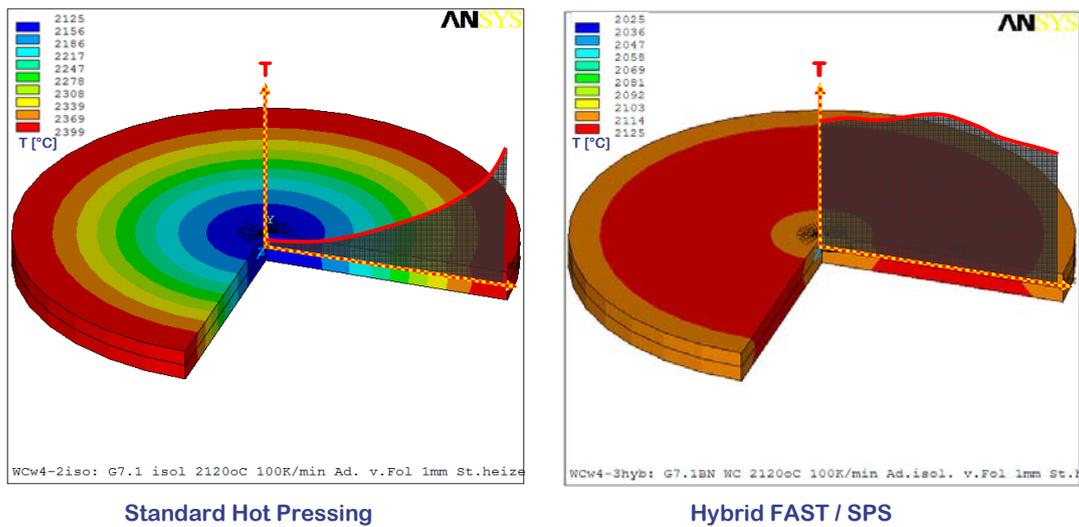


Figure 8. Compensation of residual temperature gradients by hybrid heating

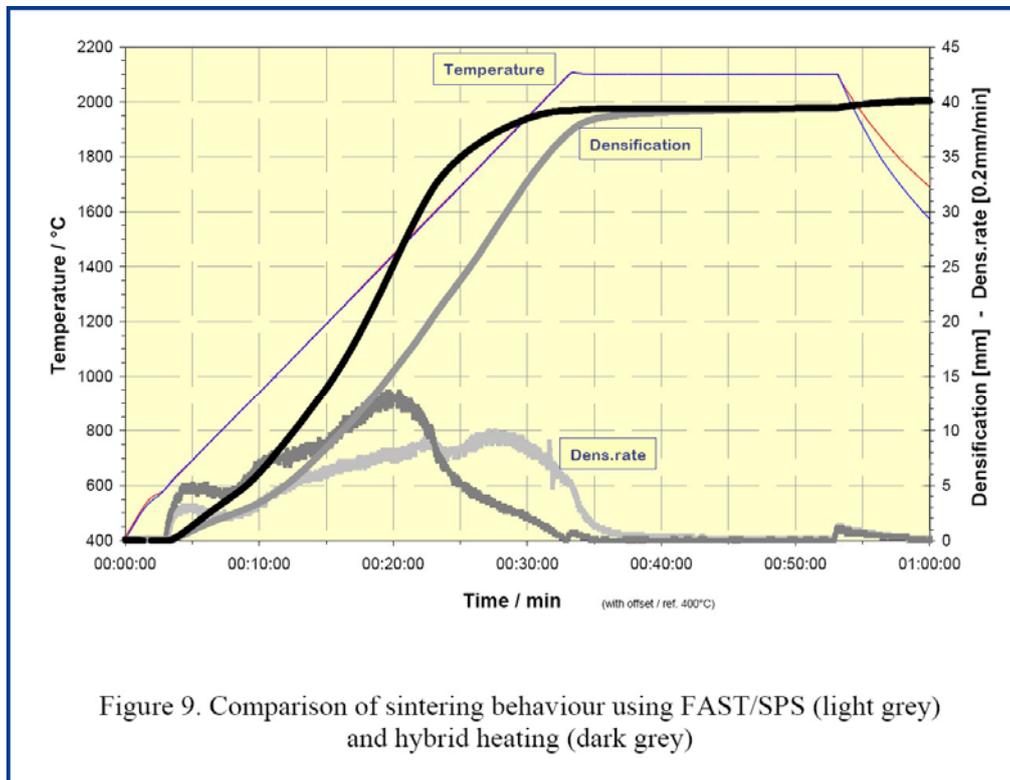


Figure 9. Comparison of sintering behaviour using FAST/SPS (light grey) and hybrid heating (dark grey)

EXAMPLES OF HIGH-THROUGHPUT FAST/SPS INDUSTRIAL APPLICATION

Hybrid - FAST/SPS Production of Large Components

One of the first industrial applications⁵ of the FAST/SPS sintering technology is the manufacturing of plate-like, large-area articles (figure 11), e.g. sputtering targets for the coating of goods with a wide range of functional surface layers. The above mentioned hybrid FAST/SPS systems, shown in figure 10, are ideally adapted for the high throughput manufacturing of such parts.

Large Scale Production by FAST²

For the mass production of small parts (5 to 25 mm) a special series of FAST/SPS systems was developed called "FAST²" (FAST square = fast FAST), which is based on state-of-the-art powder pressing technology combined with the FAST/SPS sintering method, realizing rapid and fully automatic operation including powder handling, filling of the integrated pressing tool and discharge of the readily sintered parts. The throughput of such systems (figure 12) can be as high as six pieces per minute, depending on the sintering characteristics of the actual material as well as the size of the parts.



Figure 10. 250 ton hybrid FAST/SPS production system according to figure 7, optionally equipped with two ABB industrial robots for charging and discharging.

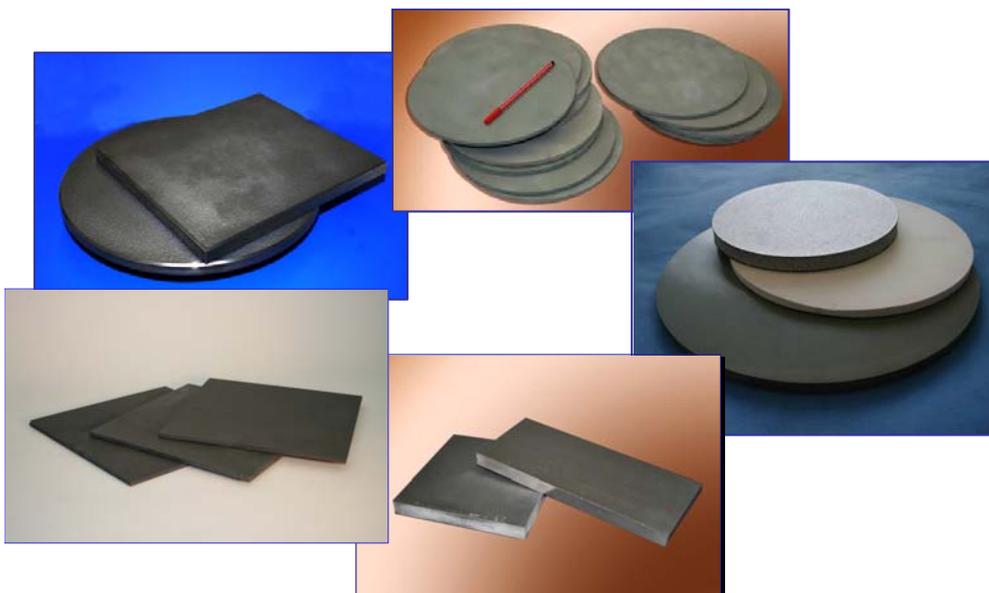


Figure 11. Plate-like, large-area articles (e.g. sputtering targets) made of binderless tungsten carbide, noble metals, and several ceramic materials



Max. Speed 6 pcs./min
Pressing Force 10 t
Part Diameter 5 – 25 mm
CNC Control



Figure 12. FAST² system for the mass production of small parts



Figure 13. Pressing tools for multiple powder compacts

Further Examples for Cost Efficient FAST/SPS

Besides all the possibilities mentioned above in order to realize an industrial production with high cost efficiency, figure 13 demonstrates one other method: The use of multiple pressing tools for single or manifold powder compacts can provide a number of parts by one sintering cycle, enhancing the effective throughput of the FAST/SPS system significantly.



Figure 14. Currently running development from simple shapes to complex geometries

CONCLUSION

It has been shown, that the promising results of FAST/SPS reported by numerous scientific papers can be transferred to a cost efficient industrial production, if the equipment meets the respective prerequisites. These requirements have been discussed and examples for successful industrial applications have been shown. Current developments are related to the industrial production of more complex geometries (figure 14) as well as further optimization of quality and costs.

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