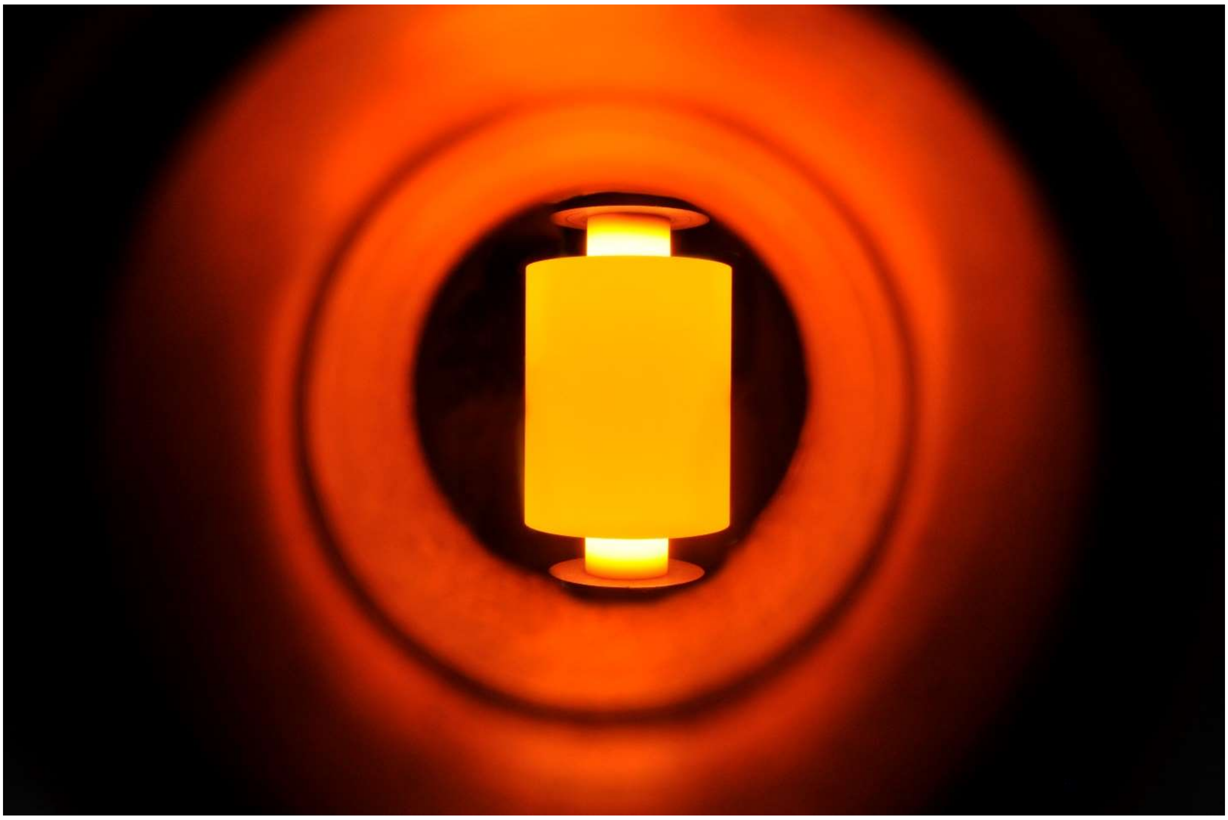
Introduction to

Field-Assisted Sintering Technology/Spark Plasma Sintering (FAST/SPS)

A guide for Designers and Engineers



Basic principle of FAST/SPS | Tool design and tool materials | Technical Guidelines | Case studies

FIRST EDITION (2022)

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1. Introduction

1.1 General

“Field Assisted Sintering Technology/Spark Plasma Sintering” (FAST/SPS) is a low-voltage, current-activated/assisted and pressure-assisted sintering process. It is preferentially used to densify materials or to synthesize new materials in a single processing step. FAST/SPS belongs to the group of “Electric current activated/assisted sintering (ECAS)” technologies.

FAST/SPS enables enhanced densification by superposition of external pressure and direct heating by applying an electric field. This technique results in higher heating rates and shorter cycle times when compared to conventional sintering, hot pressing or hot isostatic pressing. Furthermore, the sintering temperature can be significantly reduced.

FAST/SPS is preferentially suitable for ceramic powders, metal powders and composite materials, which are difficult to sinter due to

- Low sintering activity,
- High reactivity,
- Poor deformability,
- Unfavorable particle morphology,
- Unfavorable particle size distribution or
- Large difference of physical properties.

1.2 Vocabulary

The general term „Electric current activated/assisted sintering (ECAS)” includes more than 50 subcategories. Essential patents date back to the beginning of the 19th century (1). All ECAS technologies are based on an experimental setup similar to a hot press, but the specific characteristics are conductive punches and, usually, a conductive die, which are integrated into the electrical circuit of the equipment. The main difference between the subcategories is the type of current that flows through punches and die. The current can be applied as direct current (DC), pulsed direct current (pulsed DC), alternating current (AC), and combinations thereof.

Since 1990s, the term “Spark Plasma Sintering” (SPS) has become popular in scientific literature. It refers to the prevalent belief that a pulsed current in SPS devices generates sparks and plasmas in the powder bed, accelerating sintering kinetics, amongst others, by cleaning of particle surfaces. However, the formation of sparks and plasmas has never been proven without doubt (2) (3). Therefore, the term “Field Assisted Sintering Technology/Spark Plasma Sintering” (FAST/SPS) is used throughout this booklet, which refers to the review paper of Guillon et al. (4).

1.3 Principles of heating in a FAST/SPS device

In FAST/SPS devices, heating is conducted in specific tools consisting of two punches and a die. While the punches need to be electrically conductive, the die can be either electrically conductive or insulating. The dominating heating mechanism is Joule heating (= resistance heating).

Accordingly, electrical resistivity of the tool and the sample materials determines whether the powder or the die is heated. In general, there are three main distinguishable cases. In the case of non-conductive powders, heat is directly transferred from the heated tool to the powder by thermal conduction (**Figure 1a**). If conductivity of the powder exceeds a specific value, direct heating (self-heating) of the powder by dissipating energy within the powder becomes possible as additional heating mechanism (**Figure 1b**). The highest possible current density in the powder specimen can be achieved if a conductive powder is sintered in a tool with insulating die (**Figure 1c**). Electrical insulation can be achieved by either applying an insulating coating on the inner wall of the die or by making the die (or a suitable insert) from an insulating material.

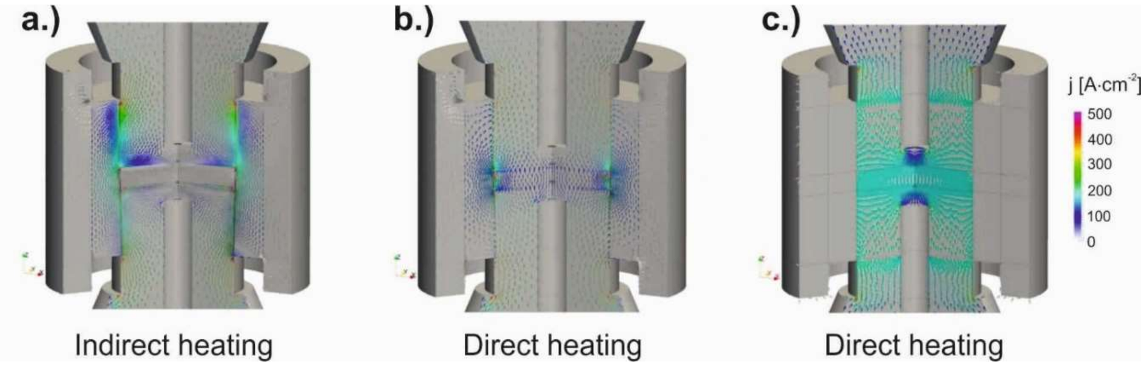

Figure 1: *Schematic current flow distribution in the case of* ***a.)*** *Non-conductive powder, conductive die, indirect heating by thermal conduction* ***b.)*** *Conductive powder, conductive die, direct Joule heating and* ***c.)*** *Conductive powder, insulating die, direct Joule heating. Adapted from* (4). *(Reproduced with permission of Wiley VCH).*

In the case of conductive powders, it must be taken carefully into account that specific electrical resistivity might drastically change by several orders of magnitude with increasing density and/or formation of conductive paths, e.g. in the case of composite materials (5) (6). For semiconducting materials, there might be even a significant change solely with increasing temperature. Resistivity changes during the FAST/SPS cycle have strong influence on the current and temperature distribution, and, therefore, on densification and sample homogeneity.

1.4 Mechanisms involved in FAST/SPS

FAST/SPS is based on superposition of **thermal**, **mechanical** and **electrical effects** contributing to densification of powders as discussed below (4). Due to direct heating of tool/sample, high heating rates are accessible, which enhance densification, inhibit grain growth, and reduce chemical interactions at interfaces. Vice versa, high heating rates promote temperature gradients and non-uniform temperature distributions. In addition to diffusion based sintering mechanisms, mechanical pressure activates additional densification mechanisms like particle rearrangement, plastic deformation, grain boundary sliding and creep. Current flow can be advantageous to break passivating oxide or carbonate layers. On the other hand, it might cause formation of percolating current paths (“Hot spots”) or temperature gradients due to the Peltier effect in DC and pulsed DC setups. In the literature, the formation of sparks and plasmas in the gap between particles (“micro spark/plasma theory”) is also discussed as an additional densification mechanism. Local overheating and melting of particle surfaces due to these effects is expected to support the formation of necks between the particles, especially at an early stage of sintering. A more detailed discussion is omitted here since formation of sparks and plasmas has not been proven without doubt so far (2) (3) (7).

1.5 Benefits and opportunities of FAST/SPS

There are specific benefits and opportunities of FAST/SPS (4) (8) (7) (9) (10), which can be summarized as follows:

- High heating rates up to 1,000°C/min
- Temperatures between room temperature and 2,400°C, in exceptional cases up to 3,000°C
- Complete time for transferring a powder into a sintered part in 2 to 25 min
- No binders needed
- No need for pre-compaction and handling of fragile “green” parts
- Accelerated sintering kinetics due to superposition of specific thermal and mechanical effects
- Reduction of sintering temperature by more than 200 °C
- High heating rates enhance densification over grain growth
- Limitation of grain growth, interfacial reactions and decomposition
- Possibility of bonding of dissimilar materials with low interdiffusion

These benefits make FAST/SPS attractive for the sintering of:

- Materials with low sintering activity like refractory metals and high-temperature ceramics (borides, carbides and nitrides)
- Powders with unfavourable powder characteristics regarding particle morphology and particle size
- Nanoscale powders, where the nano-sized grains are preserved in the sintered part, leading to unique mechanical properties such as high strength and hardness

- Materials and composites out of thermodynamic equilibrium = non-equilibrium materials (e.g. amorphous materials, metallic glasses)
- Composite materials and material composites combining materials with large difference of physical properties (e.g. melting points)

1.6 Benchmark of FAST/SPS with other sintering technologies

Conventional sintering (CS), hot pressing (HP) and hot isostatic pressing (HIP) are competitive technologies to FAST/SPS (10).

Due to the high automation potential, **conventional sintering** in batch or conveyor furnaces is the preferred choice when a large number of parts and high throughput production are the aim. Shaping can be done by pressing, ceramic injection molding, or additive manufacturing technologies, such as ink jet printing, whereby the shape complexity increases within this order. All of these technologies usually require binders to enhance stability of the "green" parts and, therefore, an additional debinding step before sintering is needed. In general, there are limitations with respect to large scaled parts for all of these shaping technologies. Depending on the furnace construction, conventional sintering can be done under oxidizing or protective conditions.

The experimental setup for **hot pressing** is quite similar to FAST/SPS. The main difference is the heating of the tool by an external heater leading to much slower heating rates. Heat transfer is based on thermal radiation and – if the cycle is done in a gas atmosphere – gas convection. Tools for hot pressing do not require electrical conductivity. Therefore oxide ceramics like alumina or zirconia can be used as tool materials, enabling the possibility of operation in air. Hot pressing in air is attractive for ceramics, which are prone to oxygen release and chemical expansion. Nevertheless, all other tool materials established for FAST/SPS are suitable for hot pressing as well, provided the right atmosphere is used (11).

For densification of powders by **hot isostatic pressing**, elaborated encapsulation including evacuation and gas tight sealing is required. Encapsulation of ceramic powders is particularly challenging since glass capsules are needed. In this case, complex shapes are not accessible. As alternative, complex shaped parts with closed porosity can be manufactured by pressing and sintering, powder injection moulding, or additive manufacturing technologies. Afterwards, hot isostatic pressing can be used to eliminate residual porosity. Here, it must be carefully considered that no gases that are insoluble in the bulk material are entrapped inside the pores. Hot isostatic pressing is done in a pressure chamber with an integrated heating element, which is filled with an inert gas like argon. Heat is transferred by radiation and convection. Pressure is applied by a compressor.

Main advantages of **FAST/SPS** compared to these technologies are higher heating rates, shorter cycle times, reduced sintering temperature and the possibility to manufacture large parts directly from the powder in a single processing step avoiding fragile “green” parts as intermediate processing stage. For protection of the tool materials from oxidation, inert or reducing atmospheres are typically used. Due to limitations regarding automation and complex shapes, FAST/SPS still serves niche markets and is applied for materials and composites which are difficult to process otherwise. On the other hand, FAST/SPS provides high safety, reliability and reproducibility.

2. Basic principles of FAST/SPS technology

2.1 Basic experimental setup

Figure 2 shows a schematic sketch of a FAST/SPS device. In principle, a FAST/SPS device consists of a mechanical, preferentially hydraulic, loading system, which at the same time acts as a high-power electrical circuit. Therefore, the mechanical and electrical load is applied via the electrodes. Densification in FAST/SPS is carried out in a tool consisting of two conducting punches and a conductive or non-conductive die. Two additional cone shaped spacers establish the contact to the electrodes. In larger FAST/SPS devices, the cones can be replaced by blocks with rectangular cross section. The standard material for punches, die, and spacers is graphite. Alternative materials are introduced in **Section 3.3**.

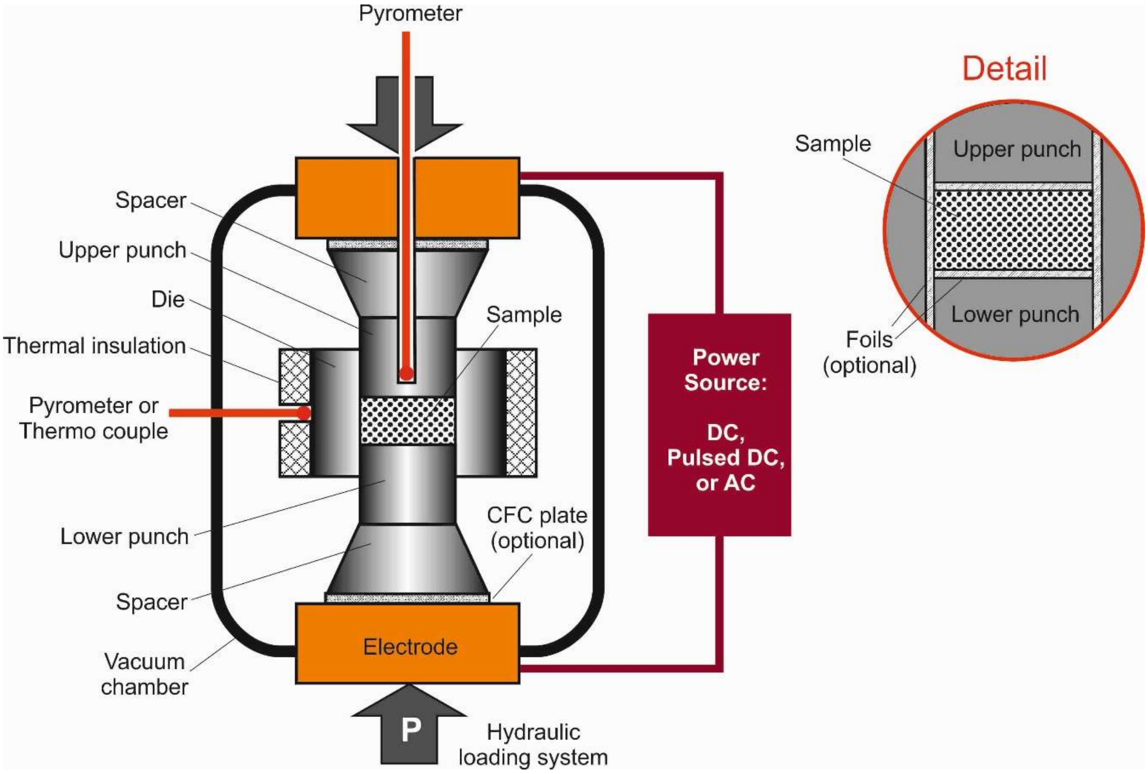

Figure 2: *Schematic sketch of the basic FAST/SPS setup. The detail sketch shows the sample area inside of the FAST/SPS tool, where foils can be inserted for improving the thermal and electrical contact while protecting the tools from abrasive wear and interface reactions.*

1) Main operational variables of a FAST/SPS cycle (9):

- Powder material properties
- Tool design, tool materials and functional coatings
- Heating cycle including heating rates, maximum temperature, and dwell times
- Mechanical loading cycle
- Vacuum and atmospheric conditions
- Power settings
- Cooling conditions

2) Control of FAST/SPS operation

FAST/SPS devices are usually operated via a control unit enabling to program the heating cycle, mechanical load, and atmosphere for automatically running the system. Usually, a PC logs data of all relevant operation parameters like temperature, load, displacement, chamber pressure, current, voltage, and others. Careful evaluation of these parameters enables conclusions on densification behaviour, optimum sintering and pressing conditions, and possible sample failure e.g. by fracture or by outgassing due to decomposition.

3) Applied load

In most FAST/SPS devices, uniaxial load is applied via a hydraulic piston. This piston limits the maximum load. Furthermore, mechanical strength and creep resistance of the tool material are other limiting factors, which both strongly depend on the applied temperature. It must be considered that misconstrued tool design might also limit the maximum load, e.g. in the case of a notch effect due to drilling holes for temperature measurement.

4) Atmosphere

FAST/SPS cycles are usually carried out in moderate vacuum (0.01 – 20 mbar) or under protective atmosphere (e.g. Ar, Ar/H_2 or N_2) to protect the tool and powder from oxidation. Fine vacuum is possible, but requires adapted chamber design. For generating the vacuum, electrodes and tool are placed in a vacuum chamber, which is evacuated by a vacuum pump. Optionally, the chamber can be filled with protective gas. There is a choice between static atmosphere control (= defined gas pressure) and dynamic atmosphere control (= continuous gas flow with controlled flow rate). Moderate over pressure up to 1.3 bar excludes contamination of the atmosphere by ambient air in the case of chamber leakages.

5) Thermal management

To avoid overheating of the FAST/SPS device, electrodes and chamber walls are water cooled. To reduce heat loss of the FAST/SPS tool by thermal radiation, the die can be optionally encapsulated with a graphite or ceramic felts. Furthermore, low thermal conducting carbon reinforced carbon

(CFC) plates are often placed between punch and spacer to decrease the heat loss via the water-cooled electrodes.

6) Optional debinding unit

Similar to sintering in especially equipped vacuum furnaces, FAST/SPS process can be combined with thermal or catalytic combustion of organic binders, e.g. when sintering of tape cast foils is aspired. For doing so, usually a separate debinding step at moderate temperatures is included in the sintering cycle. For controlled remove of the combustion products, chamber of FAST/SPS device can be optionally equipped with an external debinding unit like a cooling trap. Debinding requires special tool designs to ensure gas exchange between sample and surrounding atmosphere, e.g. through holes in the die.

2.2 Possibilities of resistance heating in FAST/SPS devices

Manipulation of wave form is a specific degree of freedom when designing FAST/SPS systems. **Figure 3** shows the three most important wave forms discussed in FAST/SPS literature, of which, pulsed DC is the most popular (3) (12). In first approximations, wave form has a negligible influence on sintering, assuming that the electric current mainly acts as heat source, based on the Joule effect. For most applications, this estimation might be true. Nevertheless, there are some effects which can arise by changing the wave form (3).

Under constant and pulsed DC, Peltier effect, electromigration, and electrochemical reduction of oxides will be at their maximum (if they appear at all) as current continuously moves in one direction. Switching to AC should minimize or eliminate these effects. Concerning the micro spark/plasma theory, pulsed DC is the favored operation mode. In general, it is difficult to design experiments that provide clear conclusions on the influence of the waveform on the resulting microstructural and mechanical properties. This is due to the fact that Joule heating dominates FAST/SPS, independent from the waveform.

Nevertheless, there is experimental evidence that pulsed DC and kind of pulse sequence change densification and microstructure evolution of materials, which are sensitive to electric field effects (e.g. oxide ceramics) (13). Amongst others, Grasso et al. (14) explains this effect by defining an electric field intensification factor, which becomes larger when the “off” time(s) τ_{off} during the pulse sequence increases. Background of this factor is the fact that – for achieving a specific power dissipation required for heating to the aspired temperature – the maximum current I_{max} must increase accordingly, which may amplify field effects.

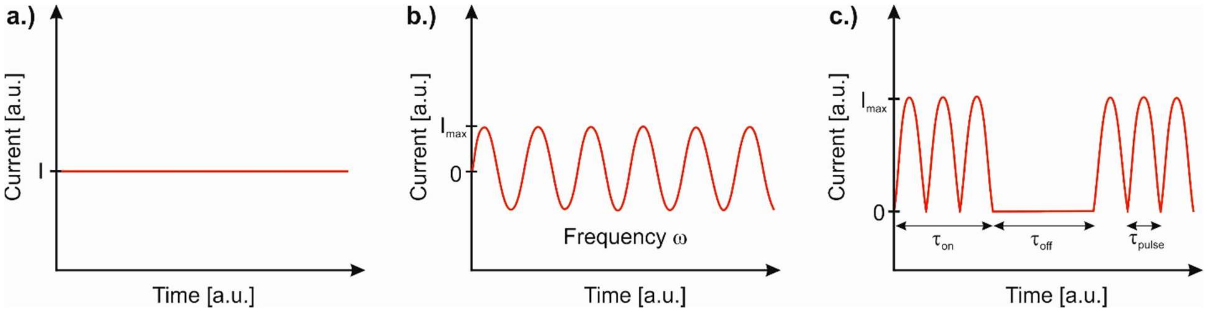

Figure 3: *Graphical sketch of typical wave forms in FAST/SPS devices* ***a.)*** *Constant direct current DC* ***b.)*** *Constant alternating current AC* ***c.)*** *Pulsed direct current DC, adapted from* (12).

When designing FAST/SPS systems, there are different concepts about how to transform high voltage, three-phase alternating current from the power grid into low voltage current for operating the FAST/SPS system. In general, the voltage of FAST/SPS systems is defined by the windings of the transformer and usually lies in the range of 3 – 10 V. **Figure 4** shows four concepts which enable the realization of the three main wave forms discussed before. **Table 1** summarizes the advantages and disadvantages of the different concepts.

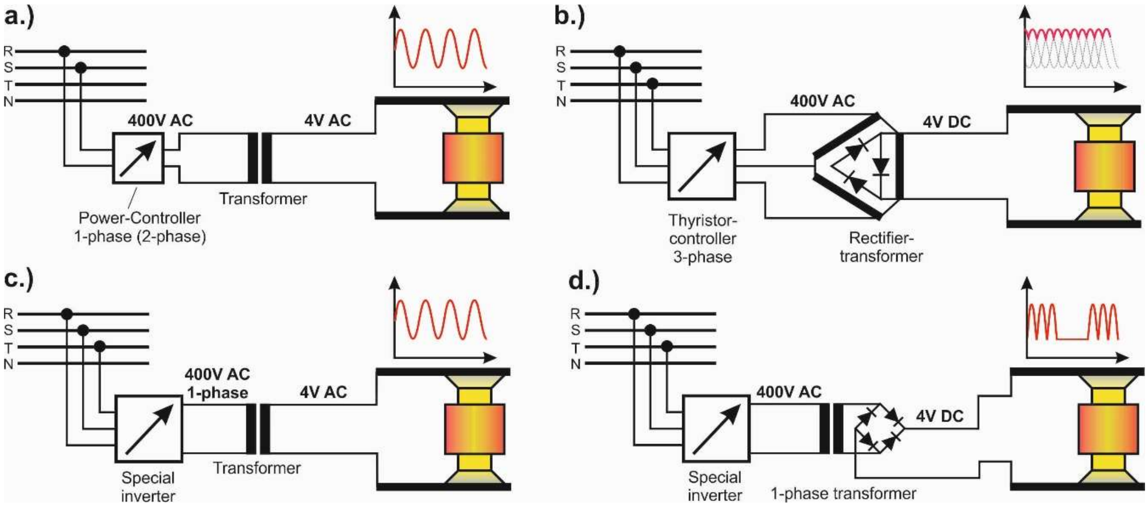

Figure 4: *Different kinds of FAST/SPS devices* ***a.)*** *AC – 2 phases* ***b.)*** *DC – 3 phases* ***c.)*** *AC – 3 phases to 1 phase (P+)* ***d.)*** *Pulsed DC – 3 phase to 1 phase DC* (15).

Table 1: *Concepts for transforming high voltage, three phase alternating current from the power grid into low voltage current for operating the FAST/SPS system.*

Transformation concept	**Advantages**	**Disadvantages**
AC – 2 phases	FAST/SPS devices easy to produce	Asymmetric load on power grid → only suitable for small devices, high operation cost
DC – 3 phases	Robust system Suitable for devices with high power Symmetrical load on the power grid	High efficiency loss at the diodes in the transformer (around 20 %) Risk of asymmetric axial heat distribution due to Peltier effect
AC – 3 phases to 1 phase (P+)	No need of diodes Small efficiency loss (around 5 %)	More complex and sensitive technology
Pulsed DC – 3 phase to 1 phase DC, typical pulse duration ranges from 1 – 999 ms	Symmetrical load on the power grid	Complex technology Risk of asymmetric axial heat distribution due to Peltier effect

2.3 How to conduct a standard FAST/SPS cycle

Figure 5 summarizes the different steps of a standard FAST/SPS cycle for sintering a sample with simple geometry. In addition, **Table 2** summarizes the main influence factors, which should be considered for each of these processing steps enabling to take full advantage of this technology. Often overlooked and inadequately described in the literature is the influence of starting powder characteristics on homogeneous sintering and densification.

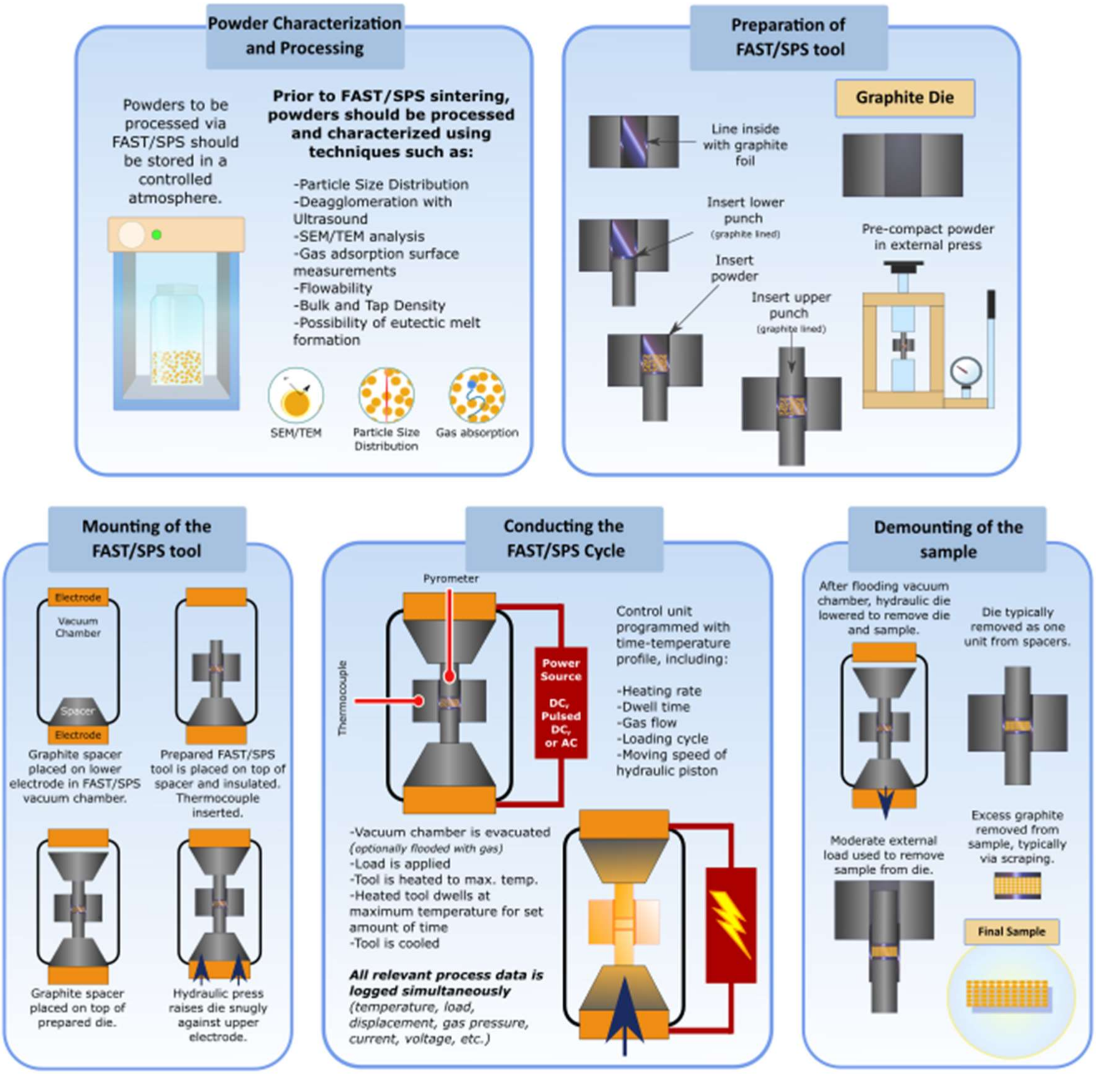

Figure 5: *Summary of the different steps of a standard FAST/SPS cycle on laboratory scale.*

Table 2: *Recommendations for successful conduction of a standard FAST/SPS cycle.*

Processing step	**Recommendation**
Powder processing	• Powder storage in uncontrolled atmospheres (e.g. ambient air) might drastically change the sintering behavior, e.g. due to formation of passivating oxide films on metal powders or diffusion inhibiting carbonate layers on ceramic powders. Therefore, storage and handling of powders under controlled conditions is recommended (e.g. in a glove box with Argon atmosphere). • If not necessarily required, powders should not contain any organic additives like binders or pressing aids.
Powder characterization	• **A comprehensive powder characterization is the basis of reproducible FAST/SPS cycles.** The following characterizations are recommended: 1) *Particle size distribution*: Laser diffraction, laser scattering or image analysis 2) *Powder agglomeration*: Measurement of particle size distribution with ultrasound as function of time. Hard agglomerates, which cannot be destroyed by standard ultrasonic treatment, might cause worse and inhomogeneous densification. 3) *Particle morphology and powder agglomeration*: Scanning electron microscopy (SEM) or image analysis (Camsizer) 4) *Primary particle size of nano-sized powders*: Transmission electron microscopy (TEM) 5) *Specific surface*: Gas absorption measurement according to Brunnauer-Emmet-Teller (BET) 6) *Flow ability and bulk density*: Hall Flowmeter according to DIN ISO 4490, DIN ISO 3923. High bulk density indicate high contact areas between powder particles, which in turn supports sintering neck formation at the early stage of sintering and accelerated densification. 7) *Tapping density* (DIN ISO 3953): High tapping density eases filling of the die. 8) *Risk of eutectic melt formation*: It must be carefully checked before FAST/SPS if there are possible eutectic melting phases between sample and tool material. It is recommended to check phase diagrams or to do preliminary sintering experiments of the respective material combination using the same atmosphere as in FAST/SPS.
Mounting of the FAST/SPS tool in the FAST/SPS device	• When mounting the tool in the FAST/SPS device, plane parallelism of punch faces must checked carefully to avoid fracture of punches and adjacent spacer components. • After positioning the tool, tool and spacers are clamped between the electrodes by moving the hydraulic piston. Thermocouple(s) and pyrometer(s) are placed at their position. Optionally, tool can be encapsulated by a porous heat shield (e.g. graphite felt) to reduce heat loss by thermal radiation. • Closing and evacuation of the chamber. • Optionally, the chamber can be flooded by protective gases like Ar, Ar/H_2 or N_2. FAST/SPS devices usually enable static atmosphere control (= defined constant gas pressure) and dynamic atmosphere control (= continuous gas flow with defined flow rate).

Programming the FAST/SPS device	• The control unit enables to program time-temperature profile including heating rate(s) and dwell times, gas flow, loading cycle and moving speed of the hydraulic piston. • FAST/SPS devices are usually operated by temperature control. The control unit defines the temperature and the system regulates the current and voltage in such a way that the measured temperature profile matches the programmed profile as precisely as possible. It should be considered that tools with different resistivity (e.g. graphite tool vs. steel tool) might require individual adjustment of PID control (PID = Proportional-Integral-Derivative) to reduce temperature fluctuations to a minimum.
Conducting the FAST/SPS cycle	• After programming the control unit, the individual program can be started and runs automatically. • **Figure 6** shows an example of a typical FAST/SPS cycle, which can be separated into four main stages. Depending on the specific application, variations of this cycle can be easily programmed. Stage I: Evacuation and optionally flooding the chamber with gas Stage II: Applying the load Stage III: Heating the tool, optionally a dwell time at maximum temperature enables to tune the microstructure by controlled grain growth after terminating densification. Stage IV: Optional dwell time at maximum temperature Stage V: Cooling • FAST/SPS devices are capable of logging all relevant processing data (temperature, load, displacement, gas pressure, current, voltage, and others). Careful analysis of these data is recommended as it gives important information on the sintering behavior of the powder. Unforeseen changes of the chamber pressure might indicate out-gassing of volatile species or decomposition of the sample. Abrupt change of displacement curve hints to sample or tool failure, e.g. due to formation of melting phases or component fracture.
Demounting of the part	• After cooling, the FAST/SPS tool is demounted, typically still in its completely assembled state. • In most cases, the sintered part is clamped in the die and can be removed by applying a moderate load via an external press. During ejection, sample failure can occur in the case that wall friction and radial pressure on the sample exceed critical values. Radial pressure on the sample remains after cooling when coefficient of thermal expansion (CTE) of the die is larger than CTE of the part. If this effect cannot be avoided, ejection of the part can be optionally done at enhanced temperatures, but this complicates the whole process, as it should, preferentially, be done prior in the FAST/SPS device. Furthermore, chemical reaction between sample and tool might hamper demounting as well. As stated previously, inserted foils or protective coatings might ease demounting of the part. Demounting can also be improved by using a separated die, which is held together by an outer ring.

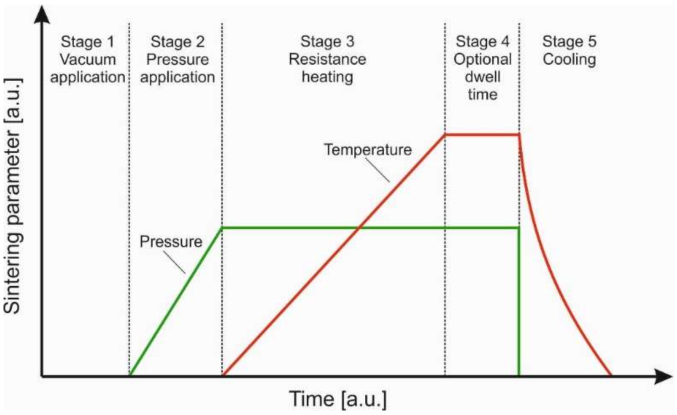

Figure 6: *Main stages of a standard FAST/SPS cycle. Adapted from* (16).

2.4 From laboratory to fabrication

Since the early 1960ies, FAST/SPS has been established in industry e.g. for the production of diamond reinforced tools, sputtering targets and braking pads (see **Section 6.2**). In the last years, there has been significant progress in the scaling up of FAST/SPS devices with respect to the sample size. In parallel, there are different solutions to increase the production capacity by applying multiple tools and/or manipulator systems for automated mounting of the parts in the FAST/SPS device. Furthermore, partly automated production lines, which contain separate pre-heating and cooling zones for pre-assembled FAST/SPS tools have been developed (17). Nevertheless, there is still the need for further improvement of FAST/SPS systems with respect to automated high through-put production. The state-of-the-art is shortly summarized here.

1) Scaling up of sample size

In industry, largest FAST/SPS devices enable production of parts with diameters beyond 400 mm. These devices also enable large scale production of parts by application of multiple tools. **Figure 7** shows two examples of FAST/SPS devices for large sample sizes.

__Figure 7:__ Examples of FAST/SPS devices for production of parts with diameters up to 450 mm __a.)__ KCE®-FCT H-HP D 400 from FCT Systeme GmbH (Courtesy of FCT Systeme GmbH) __b.)__ MSP5 & MSC5 from Dr. Fritsch GmbH (Courtesy of Dr. Fritsch GmbH).

2) Application of multiple tools

Multiple tools are standard for large scale production via FAST/SPS (**Figure 8**). One design concept are dies, which contain multiple inserts for punches in z-direction. In other concepts, insertion of intermediate spacers enables stacking of samples. Combinations of both are also possible (**Figure 8a**). Multiple tools can be also designed in form of a modular system of standardized elements, which are fixed by a frame (**Figure 8b**). Last not least, semi-finished parts can be stacked in multiple without the need of a specific die, if their mechanical stability is sufficient (**Figure 9**).

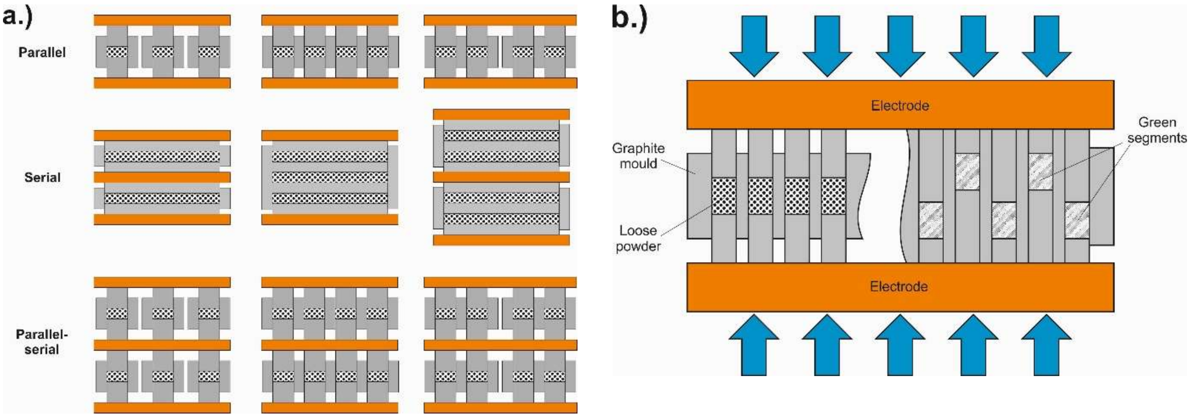

__Figure 8:__ Basic principle of multiple FAST/SPS tools __a.)__ Parallel and serial stacking, adapted from (4) __b.)__ Modular tool design, adapted from (18).

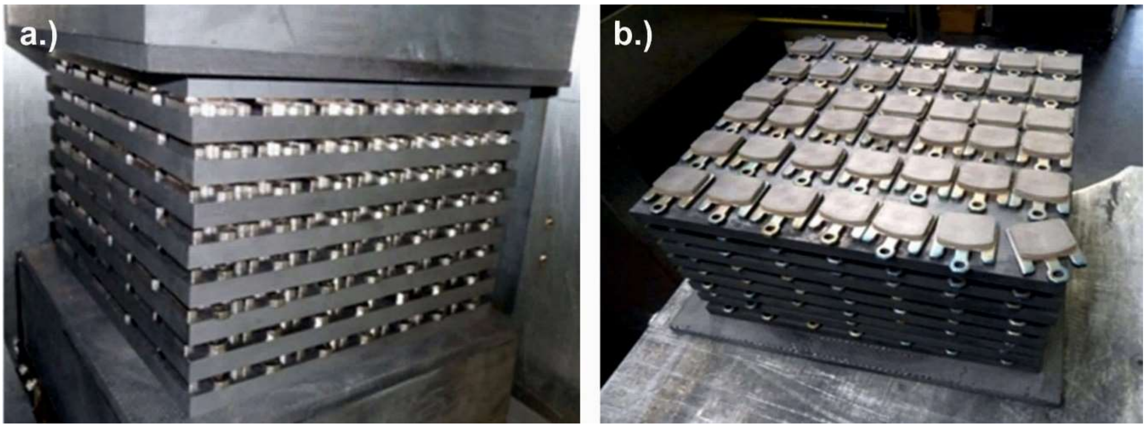

Figure 9: *Stacking of mechanically stable semi-finished parts (break pads) without specific dies around* ***a.)*** *Side view* ***b.)*** *Top view (Courtesy of Dr. Fritsch GmbH)* (18).

3) Manipulating systems for automated production

Fixing multiple tools by a standardized frame allows for them to bepositioned in the FAST/SPS by an automated manipulating system (**Figure 10**). **Figure 11** shows another example of an automated stacking and manipulating system based on a robot.

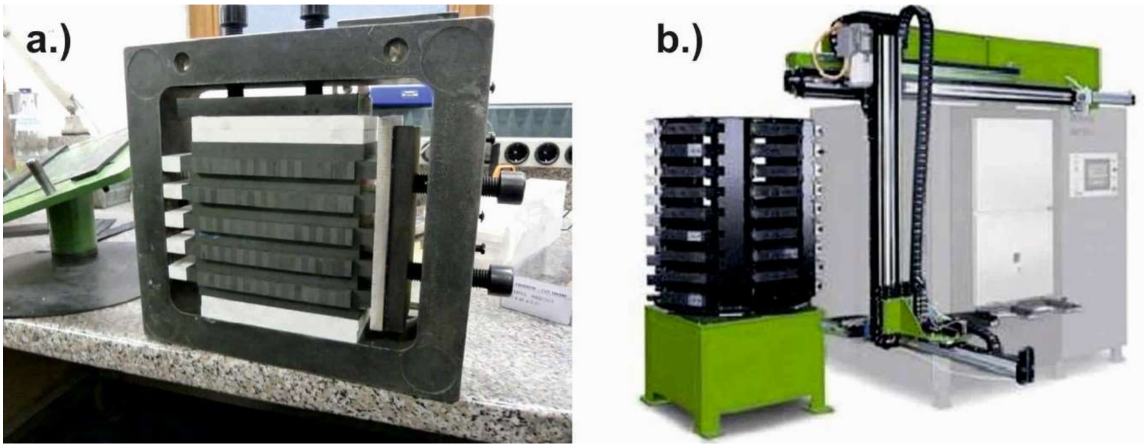

Figure 10: a.) *Frame for parallel sintering of 100 samples* ***b.)*** *Manipulating system for automated loading 54 frames in the FAST/SPS device. (Courtesy of Dr. Fritsch GmbH)* (18).

Figure 11: *Example of automated stacking and manipulating of a Dr. Fritsch FAST/SPS sinter press by means of a 6-axis robot. (Courtesy of Flertex Sinter).*

4) Production lines with separated heating and cooling zones

As alternative or in combination with the above-mentioned methods, the separation of the main processing steps (heating, consolidation, cooling) can enhance production capacity further. Well-proven in industrial applications are double chamber systems, decoupling the time-consuming cooling period from the heating and densification steps (**Figure 12**). After completion of the densification, transport of the hot tool into the cooling chamber takes place automatically. After closing the gate between both chambers, the main chamber can be opened again for charging with the next pressing tool. In order to provide enhancement of production capacity, next development stage comprises the implementation of a cooling channel instead of a chamber, as well as a pre-heating chamber or channel (17) (**Figure 13**). Recently, this principle was realized for the high-throughput manufacturing of large, rectangular ceramic plates at temperatures beyond 2000 °C. In this case, hot-pressing is used for densification. The system can operate two pressing tools simultaneously, each with max. 3000 kN and containing 14 tiles. Depending on the respective material, this results in an effective cycle time of 1 to 10 min, and a capacity of 40,000 to 400,000 tiles per year (19). It is obvious that the concept can be easily transferred to do the sintering by FAST/SPS.

Figure 12: *Example of a FAST/SPS device with separated heating and cooling zone (Courtesy of FCT Systeme GmbH).*

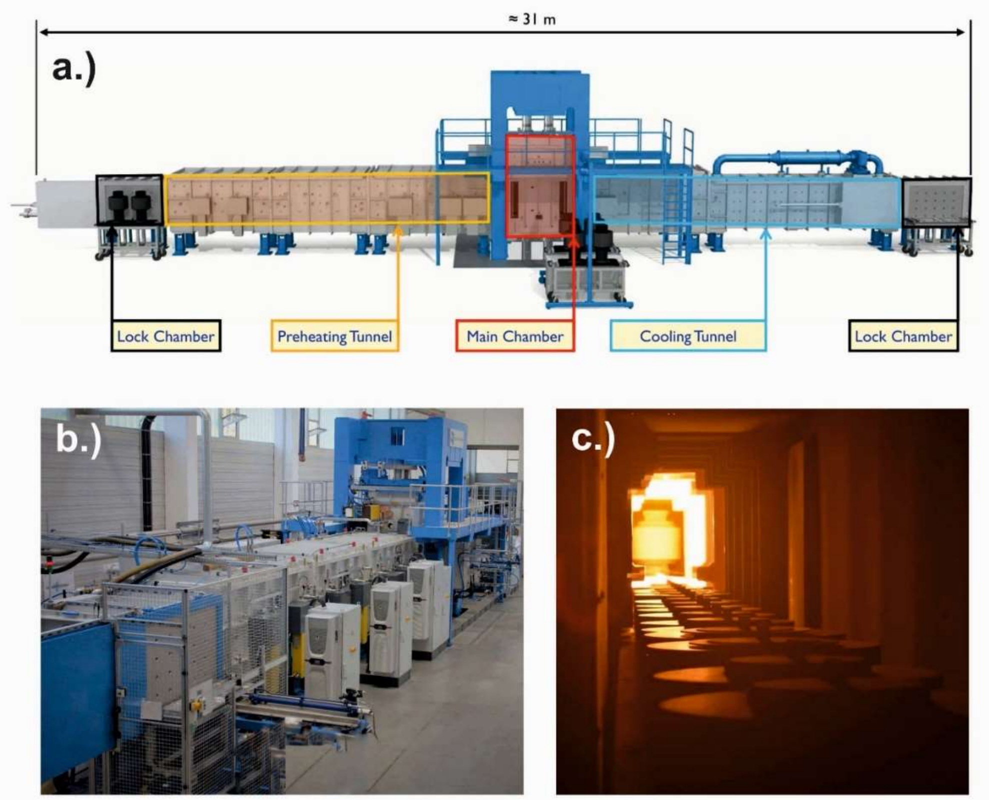

Figure 13: *High-troughput manufacturing of ceramic tiles by implementing a pre-heating tunnel and a cooling tunnel* ***a.)*** *Graphic sketch* ***b.)*** *View of pre-heating tunnel and main chamber* ***c.)*** *Look into the pre-heating zone (Courtesy of FCT Systeme GmbH).*

3. Technical guidelines

3.1 Temperature measurement

Since die and punch tightly enclose the powder sample, exact measurement and control of the temperature remains one of the biggest challenges for reliable use of FAST/SPS. The main requirements of temperature measurement are short reaction times, low time lag, high reproducibility, and measurement as close to the sample as possible. Either thermocouples or pyrometers can be used for temperature measurement in FAST/SPS devices. Both methods enable temperature measurement at distinct points and do not represent the overall temperature distribution of the sample. For predicting temperature distributions, finite-element modelling (FEM) is required (see **Section 5**). Direct measurement of sample temperature is described in literature (20) (21), but it is not practical in daily use.

Thermocouple(s) are usually placed in radial holes inside the die to measure the temperature as close to the sample as possible. The following thermocouples are in regular use: Type K (Ni-CrNi) for temperatures between room temperature (RT) and 1100 °C, type S (Pt-Pt 10% Rh) for temperatures between RT and 1450 °C and Type C (W5Re-W26Re) for temperatures between RT and 2200 °C.

Pyrometers measure the thermal radiation of the heated tool according to the Stefan Boltzmann equation:

$$j = \varepsilon \cdot \sigma_{SB} \cdot A \cdot T^4$$

j = thermal radiation, ε = emissivity, σ_{SB} = Stefan Boltzmann constant, A = area, T = temperature.

Temperature measurement via standard pyrometer is possible in the range between 300°C and 3000°C. As alternative, two-color pyrometers enable to broaden the measurement range to even lower temperatures (100°C – 2500°C) (22). Nevertheless, control of temperature near to room temperature remains difficult. Pyrometers can placed axially, measuring the temperature at the base of drilled holes in the upper or the lower punch. As alternative, they can placed radially measuring the temperature on outer surface of the die. However, this is coupled with higher inaccuracy.

3.2 Sintering atmospheres

The material of the tool used limits the atmosphere applied during FAST/SPS, more than the sintered material does. Atmosphere inside the tool is usually different from the atmosphere in the FAST/SPS chamber since tight tolerance of the gap between punch and die hinders unrestricted gas exchange. **Table 3** summarizes possible sintering atmospheres in FAST/SPS devices. Neutral or reducing atmospheres (Vacuum, Ar, N_2) are most relevant in daily use. Formation of passivating oxide or carbonate layers in the case of contamination of sintering atmosphere with H_2O or CO_2

must considered carefully. Furthermore, all of the atmospheres are critical for sintering of oxide ceramics, which are prone to evaporation, decomposition and/or oxygen release under lowered oxygen partial pressures. Contact to graphite tools or graphite foils might further aggravate the reducing effect.

Table 3: *Sintering atmospheres in FAST/SPS devices.*

Atmosphere	**Remarks**
Vacuum	• Vacuum in standard FAST/SPS devices in the range between 0.01 and 20 mbar depending on the chamber construction and vacuum pump • Better vacuum possible on demand • Vacuum recommended for materials sensitive to nitrogen or hydrogen
Argon	• Most commonly used protective gas in lab-scale FAST/SPS devices • Expensive for large scale devices • Argon enables to minimize evaporation or decomposition of sensitive materials • Entrapment of Argon in closed pores critical due to causing residual porosity difficult to remove
Argon/Hydrogen	• Application if targeting oxidation partial pressure below 10^{-6} mbar, which is not accessible with vacuum pumps • Argon/Hydrogen mixtures with hydrogen content below inflammation limit (2.9 Vol. % Hydrogen) are in regular use in lab-scale devices • Higher hydrogen contents are not accessible due to safety concerns
Nitrogen	• Most commonly used protective gas in large scale FAST/SPS devices • Oxidation partial pressure can be further lowered by addition of hydrogen below the inflammation limit • Possible reaction of nitrogen with tool materials forming e.g. nitrides or cyanides must considered carefully.
Technical air	• Operation of FAST/SPS devices with technical air (nitrogen/20 % oxygen) is possible with care • Limited oxidation resistance of most tool materials must considered carefully. The following temperature limits exists for the different tool materials: Graphite: 600°C Tool steels with low chromium contents: 300 – 400°C High temperature alloys with passivating oxide layers: 900 – 1000°C, electrical resistance of oxide layers might influence Joule heating of tools Refractory metals: Not suitable due to formation of instable oxide layers

3.3 Tool materials

The tool material play a key role in FAST/SPS. When choosing the tool material, the following influencing factors must considered:

- Reactivity between tool and sample

- Reactivity with the chamber atmosphere
- Mechanical properties such as ultimate strength and creep strength as function of load, temperature, and dwell time
- Failure tolerance when handled by untrained people
- Coefficient of thermal expansion related to the sample
- Machinability
- Material and processing costs
- Wear resistance in the case of multiple use
- Lifetime

Table 4 summarizes the physical properties of selected tool materials. Materials with electrical resistance clearly below the electrical resistance of graphite (10-3 Ωcm) might cause ineffective Joule heating due to the requirement of high current densities. Furthermore, exact temperature control during heating becomes challenging. **Table 5** summarizes advantages and disadvantages of materials used for FAST/SPS tools. Graphite based tools are most commonly applied. Depending on specific conditions, the application of composite materials or a combination of different materials for punches and die might become a promising alternative (23). If the sintered powder is conductive, electrically insulating dies are an option. In this context, literature describes a die concept containing a ceramic liner with a metal jacket (11).

Table 4: *Physical properties of selected tool materials. Partly adapted from (23) and complemented by referring to data sheets of related suppliers.*

Material	α [10^{-6} K^{-1}]	λ [$W\cdot m^{-1}K^{-1}$]	ρ [$\Omega\cdot cm$]	E [GPa]	σ_{crit} [MPa]
Graphite	4.0 – 6.5	80 - 110	$1\cdot10^{-3} – 2\cdot10^{-3}$	9 - 25	44 - 85
Si_3N_4	3.3	30	$> 10^{13}$	300	800
hBN	3.6	30	$> 10^{14}$	50	80
SiC	5.0	120	$> 10^{5}$	480	500
B_4C	4.5	30	$> 10^{5}$	450	350
AlN	4.5	150	$> 10^{11}$	320	250
WC	4.0	30	10^{-5}	700	500
TiC	5.7	20	10^{-5}	450	500
W360 Steel*	12 - 13	32	$5.9\cdot10^{-5}$	212	500
Inconel X750**	14 - 17	18 - 22	$1.3\cdot10^{-4}$	214	900 - 1000
Plansee TZM***	5.2 – 5.8	140	$5.6\cdot10^{-6} – 4.5\cdot10^{-5}$	300	800 - 1000
WC-Co	5.0 – 6.0	65 - 80	$2.0\cdot10^{-5}$	500 - 650	2000 - 3000

*Fe-4.5Cr-3.0Mo-0.55V-Mn,Si,C * Ni-based superalloy, ** Mo-based alloy.

α = coefficient of thermal expansion, λ = thermal conductivity, ρ = specific resistance, E = Young´s modulus, $\sigma_{crit.}$ = bending strength.

Table 5: *Tool materials for FAST/SPS including their advantages and disadvantages.*

Tool material	**Advantages**	**Disadvantages**
Graphite	• High operation temperature and excellent creep resistance up to 2,400°C in vacuum or inert atmosphere, standard material for temperatures > 1,200°C (9) • Electrically conductive from room temperature • Moderate resistivity ~ 10^{-3} Ω·cm suitable for Joule heating • Moderate coefficient of thermal expansion ($4.0 – 6.5 \cdot 10^{-6}$ K^{-1}) Ease ejection of materials with higher coefficient • Available in different qualities varying in grain size, density, purity -> best compromise for specific application • Ease of machining (11) • Self-lubricating surface prevents sticking and wear • Combination with higher strength tool materials (steel, TZM): Maintaining graphite environment by inserting graphite parts (9)	• Reaction with oxygen at temperatures above 600°C • Risk of carbon contamination and carbide formation • Graphite causes reducing conditions • Low flexural and compressive strength, limitation of minimum wall thickness • Low wear resistance, less suitable for high-throughput production, tools must be replaced regularly • Limited dimensional accuracy
Carbon reinforced carbon (CFC) (24)	• High stiffness and strength up to highest temperatures • Less sensitive to fracture • Resistant to thermal shock • Moderate coefficient of thermal expansion • Low thermal conductivity, can be advantageous to level thermal gradients and reduce heat loss via the water-cooled punches, but requires careful analysis (24).	• Reaction with oxygen at temperatures above 600°C • More expensive than graphite due to multistep processing • Anisotropic properties • Limited surface quality
Refractory metals (W, Mo, Ta, Nb) (11)	• Ease of machining • Moderate thermal expansion, similar range like graphite • Reasonable strengths even beyond 1000°C (vacuum or inert atmosphere) • Higher loads possible than with graphite, especially at temperatures < 1000°C • Alloys like Plansee TZM (= titanium-zirconium- molybdenum) deliver best compromise of properties, further improvement by functional coatings e.g. with $MoSi_2$ or B for increasing surface hardness and reducing wear	• Expensive • Susceptible to grain growth and creep • Susceptible to wear if particles reach the gap between punches and die • Reactivity, e.g. in contact with oxides, carbides, nitrides • High coefficient of thermal expansion in the range of 14 – 18 10^{-6} K^{-1}, higher than CTE of most ceramics -> risk of sample fracture during ejection, optional ejection at elevated temperatures

Superalloys (Rene 41, Udimet 700, Inconel X) (11)	• Ease of machining • Reasonable strengths (up to 900°C) • Higher loads possible than with graphite (up to 900°C) • Formation of protective alumina or chromia scales might enable operation in technical air at temperatures up to 900°C	• Expensive • Excessive creep and risk of stress rupture failure at temperatures beyond 900°C • Formation of passivating oxide layers might deteriorate Joule heating of the tools • Susceptible to wear in contact with abrasive particles • High coefficient of thermal expansion in the range of 14 – 18 10^{-6} K^{-1}, higher than CTE of most ceramics -> risk of sample fracture during ejection, optional ejection at elevated temperatures
Ferrous steels (11)	• e.g. Uddeholm QPro90 Fe-2.6Cr-2.25Mo-0.9V-0.3Si-0.38C • Ease of machining • Reasonable strengths (up to 600°C) • Higher loads possible than with graphite (up to 600°C) • Cheap compared to refractory metals and superalloys	• Creep and risk of stress rupture failure at temperatures beyond 600°C • Formation of passivating oxide layers might deteriorate Joule heating of the tools • Coefficient of thermal expansion in the range of 10 – 12 10^{-6} K^{-1}, higher than CTE of most ceramics
Ceramics (Carbides, Nitrides, Oxides)	• Ceramics are more wear resistant and less reactive than graphite • Ceramics maintain strength at higher temperatures than metal alternatives • Conductive carbide ceramics for punches and dies: WC, TiC • Conductive ceramics should be mainly used for punches (11): Here, mostly compressive loads, therefore clearly reduced failure probability, combination with other die materials recommended • Conductive ceramic punches: long life, resistance to creep, low reactivity, freedom with respect to environmental protection, higher loads than graphite	• Expensive • Machining of tools and especially of dies challenging (e.g. by diamond cutting, grinding) with exception of BN (machineable, but low strength) • Risk of brittle failure, especially at low temperatures • Limited thermal shock resistance
Composites	• Hard metal tools (WC-Co): higher load and wear resistance compared to graphite tools (25) • Carbide – graphite (TiC-C, WC-C, B_4C-C) composites for punches with enhanced conductivity (23) • Nitride – carbide composites (Si_3N_4+hBN, SiC+hBN, B_4C+hBN, AlN+hBN) for non-conductive dies (23)	• Expensive • Machining of tools and especially of dies challenging (e.g. by diamond cutting, grinding) with exception of BN (machineable, but low strength) • Risk of brittle failure, especially at low temperatures • Limited thermal shock resistance

3.4 Inserting foils and functional coatings (26)

Inserting foils or applying functional coatings on the inner surface of FAST/SPS tools is a helpful measure to separate tool and sintered part. Foils or coatings can be advantageous due to

- Avoidance or reduction of chemical reactions and contaminations resulting from the direct contact between the tool material and the sintered part, and avoidance of secondary operations to remove reaction layers
- Avoidance of the sticking of the sintered part to the die wall during ejection
- Protection of the tools from wear when sintering abrasive powders (e.g. diamond reinforced steel powders)

In an R&D environment, application of foils or coatings is quite common, and the time-consuming manual handling and risk of wrong application do not matter as much. In an industrial production environment, the situation is different. Robots cannot handle foils and sprays well and operators are often not paying attention to details when working under time pressure. Even small mistakes will result in low quality output or even cycle abortions. Therefore, even though using insert foils and functional coatings seems to be beneficial at a first glance, it is highly recommended to avoid them in industrial production wherever it is possible.

When inserting foils in a FAST/SPS tool, foils must tightly fill the gap between punch and die without wrinkles to ensure that powders do not enter the gap, which can cause tool fracture. Therefore, foils need to be cut in the exact size adapted to the tool design. Formation of foil fragments is critical with respect to severe damage and contamination of the sintered part. Furthermore, foil fragments can cause high temperature spots due to changing of the current flow within the tool. When applying conductive foils, areal contact is necessary to achieve a well-defined electrical contact between punch and die leading to a homogeneous current density in the sintered part. All of these issues become more pronounced with the increasing size of the sintered part. Functional coatings are an attractive alternative to foils. Usually, they are manually applied on tool surfaces, which are in direct contact with the sintered powder. **Table 6** summarizes specific issues of foils and coatings with respect to their application in FAST/SPS tools.

Table 6: *Inserting foils and functional coatings for FAST/SPS tools.*

Inserting foils	**Remarks**
Graphite foils	• Typical thickness ranges between 0.2 – 0.5 mm • Due to their layered structure, graphite foils usually have anisotropic electronic conductivity, which brings uncertainty to current and temperature distribution, especially in the case of varying thickness or gaps between foil and die wall. • On the other hand, flexibility of graphite foils improves electrical contact • High purity graphite recommended, graphite contaminations like sulphur might cause undesired chemical reactions
Metal foils (e.g. Mo, W, Ta)	• Typical candidates are refractory metals likes molybdenum, tungsten or tantalum • Mo and W are susceptible to form volatile, instable oxides • More resistant to damages and easier to handle than graphite foils • High electronic conductivity • Risk of chemical reaction with sample • Metal foils might be difficult to remove in the case of reaction or interdiffusion phenomena
Mica foils	• Mica is a natural, platelet shaped material usually applied as high temperature sealant • A typical representative is Vermiculite $(K, Mg, Fe)(Si, Al)_4O_{10}(OH)_2$ • Highly flexible • Electrically insulating, therefore not optimum for conducting powders • Relative inert material
Graphite spray	• Good alternative to graphite foils • Easy to apply • Electrically conductive, but less defined interface than in the case of foils • Contamination of the work space with spray particles • Common in diamond tool industry for protecting the graphite parts and reducing the wear of the die
Boron nitride spray	• Easy to apply • Electrically insulating material • Boron nitride not optimum for sintering of conductive powders. When applied on the face of the punches, direct Joule heating of the powder is not possible. • Boron nitride enables to insulate parts of the tool for guiding the current flow. • Less contamination of work space than in the case of graphite spray • Widely used in many industrial applications. • Regularly used for processing of diamond reinforced steels to avoid sticking on the die wall.
Suspension coatings, PVD, CVD (e.g. Al_2O_3, ZrO_2, Y_2O_3, TiN, SiC)	• Possible alternatives • Rarely used

3.5 Possible reasons for sample failure

Fracture of sample or tool is a common phenomenon when conducting FAST/SPS cycles. There are manifold possible reasons of sample failure, which **Table 7** summarizes. Some of the reasons are quite general for powder processing; others are specific for FAST/SPS technology.

Table 7: *Failure modes, which might appear during FAST/SPS cycles.*

Failure mode	**Remarks**
Inhomogeneous powder filling and friction between particles	• Powders with poor flowability and agglomerated powders might cause inhomogeneous filling of the die **Measures against:** • Breaking of agglomerates e.g. by milling • Characterization of flowability of powder e.g. by hall flow meter • Improving the flowability e.g. by spray drying of powder suspensions • Levelling of the filling height e.g. by application of a blade • Improving the bulk density by tapping or ultrasonic treatment • Pre-compaction of the powder before starting the FAST/SPS cycle • Avoiding that particles enter the gap between punch, foil and die by careful handling of powders
Wall friction of the die	• Inhomogeneous densification due to wall friction • Wall friction influenced by ratio sample diameter/sample height **Measures against:** • Application of foils or sprays (**Section 3.4**) • Optimization of tool design
Overloading of tools	• Exceeding the maximum strength of the tool material • Inserting drillings for temperature measurement reduces the maximum strength due to the notch effect • Penetration of powder into the gap between punch and die might cause overloading of the tool due to clamping effects **Measures against:** • Optimized tool design with improved dimensional accuracy • Inserting flexible foils completely filling the gap • Application of ductile jacket for brittle die materials
CTE mismatch between powder and die	• If CTE of die exceeds CTE of sample, clamping of the sample during cooling results, which might cause fracture of sample or die. Fracture can happen already during cooling or later during ejection • If CTE mismatch becomes too large, sample failure is difficult to avoid **Measures against:** • Careful consideration of CTEs • Application of separated die, kept by an outer ring. Ejection of sample and inserted parts in one step, afterwards opening of the die

Thermal gradients	• Indirect heating of the powder by thermal conduction and Peltier effect in the case of DC lead to thermal gradients in the sample. If these gradients exceed a critical value, thermal stresses might lead to inhomogeneous densification and sample fracture. • Effect more pronounced in the case of non-conducting powders and fast heating rates. **Measures against:** • Optimized tool design on the basis of FEM modelling • Thermal insulation e.g. by graphite felt or CFC plates • Hybrid heating with external heating element
Entrapment of gaseous species	• Gases like Ar, which are insoluble in the sintered material, might become entrapped in closed pores and avoid full densification. **Measures against:** • Application of protective gases, which are soluble in the material • Application of vacuum as protective atmosphere
Chemical expansion and decomposition	• In most cases, FAST/SPS cycles are done under reducing conditions (low oxygen partial pressure) • Reducing conditions become aggravated when using graphite tools, atmosphere inside the tool might differ from the surrounding chamber atmosphere • Materials, which are sensitive to low oxygen partial pressure, might decompose or show chemical expansion (abnormal expansion of oxide materials in the case of excessive formation of oxygen vacancies) **Measures against:** • Control of the oxygen partial pressure • Application of inert gases like Ar instead of vacuum
Chemical reactions	• Tight contact between tool and sample triggers the occurrence of chemical reactions, e.g. formation of carbides when using graphite tools • Surrounding atmosphere might also lead to chemical reactions, e.g. formation of nitrides when using nitrogen atmosphere **Measures against:** • Application of foils, sprays or coatings to avoid direct contact between sintered material and tool • Careful selection of atmosphere and tool material

4. Tool design

4.1 Standard tool design

In standard FAST/SPS configuration, only uniaxial pressing mode is available. Therefore, geometrical complexity of sintered parts is limited. **Figure 14** shows exemplary graphite tools for sintering of cylindrical parts with diameter between 12 and 100 mm. In industry, sample diameters beyond 400 mm are already established (27). The standard tool consists of two punches and a die. Optionally, two conus shaped spacers enable mounting in the FAST/SPS device and adaption to the diameter of the punches. These spacers also act as thermal insulator against the water-cooled electrodes. Insertion of additional plates made of carbon-reinforced carbon (CFC) between punches and tool, which have a lower thermal conductivity than graphite, can improve thermal insulation and homogeneity of temperature distribution further (24). However, when applying CFC plates, it must be considered carefully that in compaction direction CFC has a higher electrical resistance than graphite. Under unfavourable conditions, this might trigger intrinsic heating of the CFC plates counteracting the thermal insulation effect. CFC plates can bring advantages if FAST/SPS device is operated with high sample temperatures and low heating rates (= low current densities).

Figure 14: *Examples of graphite tools* ***a.)*** *Die, punches with holes for pyrometer, cones for adaptation to the electrodes* ***b.)*** *Tools in laboratory scale with diameter of 12, 20 and 30 mm and* ***c.)*** *Tools for larger FAST/SPS with diameter of 100 mm.*

4.2 Realization of complex shapes (28)

Limitation of directly manufacturing complex parts prevents wider application of FAST/SPS technology. Therefore, development of new tool designs and FAST/SPS concepts is aspired, with the goal of reducing finishing costs and material loss by secondary operations. Unfortunately, this is not an easy task. With increasing part complexity and varying cross sections, achieving uniform temperature distributions during heating and dwell time becomes more challenging. Areas in the component with smaller cross section and/or sharp edges act as heat sinks since heat losses are usually more dominant in those regions. Up to now, there are several approaches described in literature to increase part complexity, but most of these technologies are still in an early stage of development.

Thermal gradients are not the only reason to explain the density heterogeneity of complex shaped parts. Indeed, since densification is unidirectional, thin areas require only a small displacement for full densification as the final density depends on the relative displacement. Thus, once thin areas achieved full densification, the graphite punch can no longer move to densify the other areas of the part (**Figure 15**). In order to obtain a fully dense part with no distortion, it is therefore necessary to ensure that the stress field is uniform within the whole part. There are several approaches described in literature to increase part complexity.

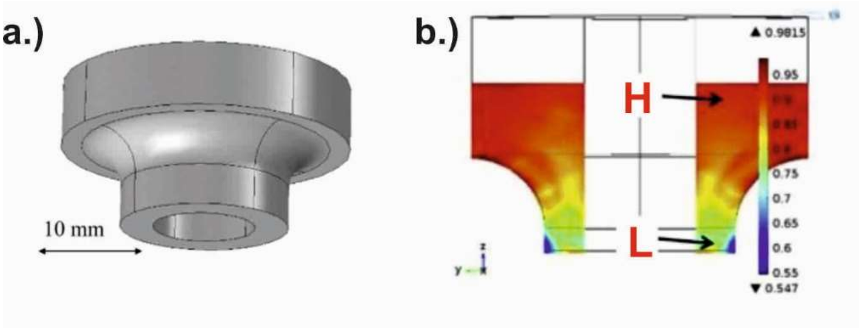

Figure 15: *Relative density distribution of complex part densified with usual graphite tool applying uniaxial load* ***a.)*** *3D image of the desired part* ***b.)*** *Relative density distribution, H = high density area, L = low density area* (29). *(Reproduced with permission of Elsevier).*

4.2.1 Net-shaped tools

Voisin et al. developed a mold that has several punches with different geometries (30). The pressure is applied in a progressive way depending on the thickness of the part's area thanks to the geometry of the different punch segments. This method allowed production of a turbine blade. **Figure 16** shows a schematic sketch of the tool design, **Figure 17** the final part and FEM modelling of temperature distribution.

These results are very impressive and show that the mold design could be the key to realize 3D shapes. Nevertheless, the high tool costs due to complex machining operations and the limited lifetime of the graphite parts remain problematic. The change of surface contact between the electrodes and the punches is also a specific challenge of this method for ensuring a homogeneous sintering during all the cycle.

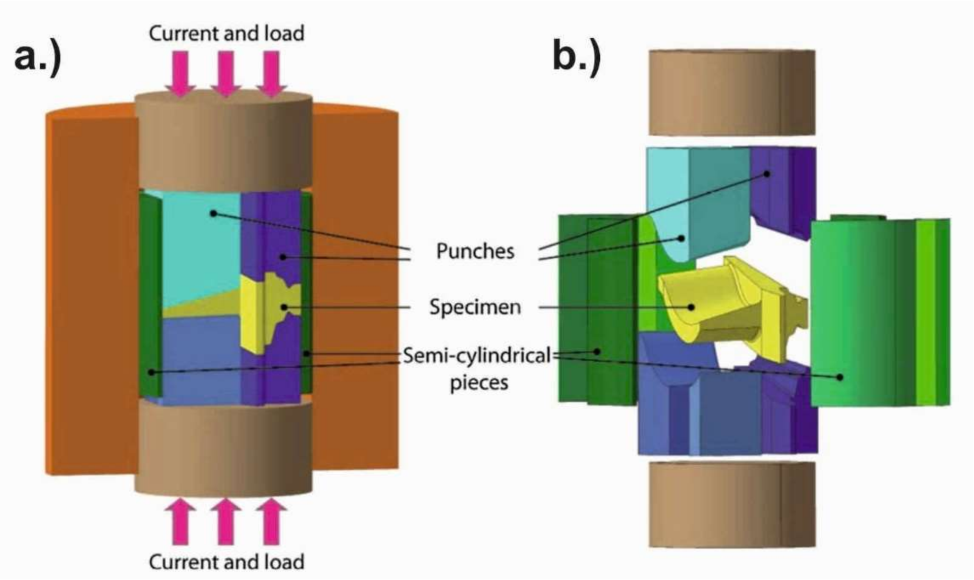

Figure 16: *Schematic drawing of the graphite mold.* ***a.)*** *Punches in their final position, fully densified specimen.* ***b.)*** *Exploded view of the mold, illustrating how the punches and the pieces with semi-circular sections give its shape to the blade* (30). *(Reproduced with permission of Wiley-VCH).*

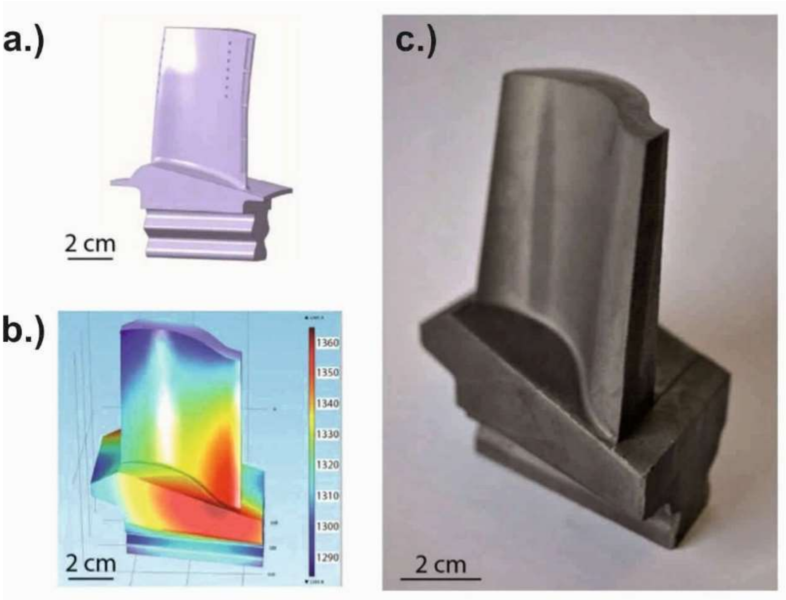

Figure 17: *Near-net shape blade obtained with a pre-alloyed 48-2-2 powder.* ***a.)*** *Blade to be near-net shaped.* ***b.)*** *Temperature map calculated by FEM.* ***c.)*** *Near-net shape blade obtained in a single SPS experiment* (30). *(Reproduced with permission of Wiley-VCH).*

4.2.2 Deformable interfaces method

Maniere et. al developed a method (WO2017099028A1) which allows the production of complex shapes by using a sacrificial part, which is separated from the sintered part by a mobile interface as shown in **Figure 18** (31). In this configuration, the sacrificial part is porous and sintered simultaneously with the part during FAST/SPS densification. The main advantage is – thanks to the porous sacrificial part – the homogeneous application of the pressure over the entire part during sintering enabling to obtain fully dense complex parts. Graphite sheet or spray (graphite or boron nitride) are applied as mobile interface.

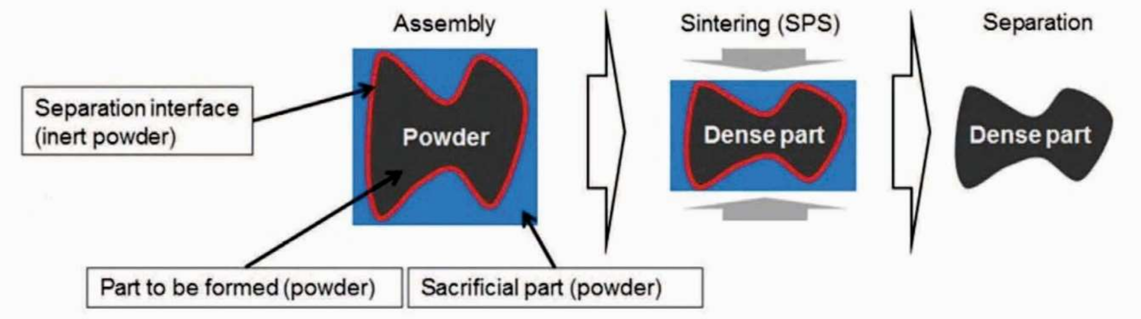

Figure 18: *Processing of complex shaped FAST/SPS parts by the deformable interfaces method* (31). *(Reproduced with permission of Elsevier).*

The main advantage of this technique is the high freedom of part design. The main challenge is the suitable shaping of the sacrificial part, as it directly determines the geometry of the final dense part. For densification of the assembly, standard graphite tools consisting of two punches and a cylindrical die - as established for conventional FAST/SPS cycles - can be used. Therefore, no extra cost for tooling arises, and the usual lifetime of graphite tools remains. However, design of the interface between the sintered part and the sacrificial material is crucial, since it must limit interdiffusion and lubricate the interface during the sintering in order to easily remove the sacrificial part after sintering. Another challenge is choosing a suitable combination of materials for the sintered part and the sacrificial part.

In principle, there are manifold possible material combinations and the method can be applied for metals, ceramics and composites, even reinforced with fibers or flakes. For demonstration, **Figure 19** shows a CoNiCrAlY turbine blade processed by this method in less than 1 h total sintering time.

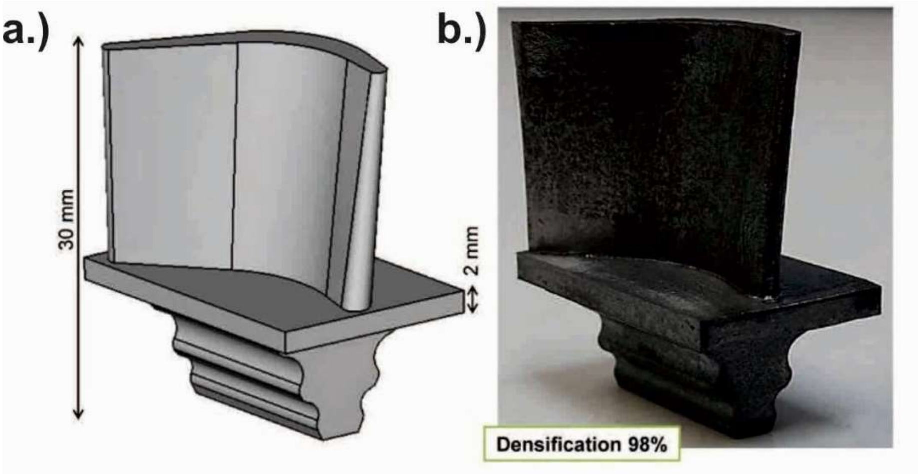

Figure 19: *CoNiCrAlY turbine blade made by the deformed interfaces method* ***a.)*** *3D image of the turbine blade* ***b.)*** *Sintered part (31). (Reproduced with permission of Elsevier).*

4.2.3 Hybridization of additive manufacturing and FAST/SPS

Norimat developed two methods for hybridization of additive manufacturing (AM) and FAST/SPS. The two methods combine the benefits of both processes, the production of complex shaped 3D parts by AM and the high-performance sintering by FAST/SPS. In this context, AM methods are preferred, which produce green parts from powders through methods such as fused deposition manufacturing (FDM), stereolithography (SLA) or binder jetting (BJ). General challenges are the suitable shaping of the green parts and the handling of organic binders in the FAST/SPS device.

The preform method: Figure 20 shows the processing steps of the preform method (Patent WO2020070107A1), a direct printing method. As a first step, a suitable cold printing process (FDM, SLA, or BJ) is used to produce porous green parts. Then, the green parts are placed in a graphite mold and are covered with a sacrificial powder as described in patent WO2017099028A1. Simultaneous densification of porous part and sacrificial powder via FAST/SPS follow. Finally, the sintered part is demolded.

Figure 20: *Schematic sketch of the preform method combining additive manufacturing and FAST/SPS for the production of complex shaped parts (Courtesy of Norimat SAS).*

For demonstration, Norimat SAS manufactured a series of complex parts from various kinds of materials such as stainless steel, Inconel, hard metal (tungsten carbide doped cobalt), boron carbide or alumina (**Figure 21**). At current state, the main constraint of this method is the limitation of materials already printable by cold printing methods. Consequently, process flexibility depends on the availability of suitable starting powders or requires the development of custom-made materials. To overcome this constraint, Norimat SAS developed an indirect method, which is based on a 3D printed counter-form.

Figure 21: *Parts obtained by the preform method (Courtesy of Norimat SAS).*

The counter-form method: Figure 22 shows the main steps of the counter-form method (Patent WO2020070133A1). The process starts with printing of a porous overmold by a suitable cold printing process (FDM, SLA, or BJ), which has the negative geometry of the final part. Then, the respective starting material (metal, ceramics, composite) is poured in this counter-form. The assembly is placed in a graphite mold. Simultaneous sintering of the material and the sacrificial overmold via FAST/SPS results in high densification of the sintered part. Finally, the sintered part is demolded.

Figure 22: *Schematic sketch of the counter-form method (Courtesy of Norimat SAS).*

This method allows for the sintering of almost all kind of materials with much fewer restrictions. For demonstration, Norimat SAS produced star-shaped prototypes starting from metal (titanium aluminide, aluminum alloy) or ceramic (zirconium boride or blue zirconia) powders (**Figure 23**).

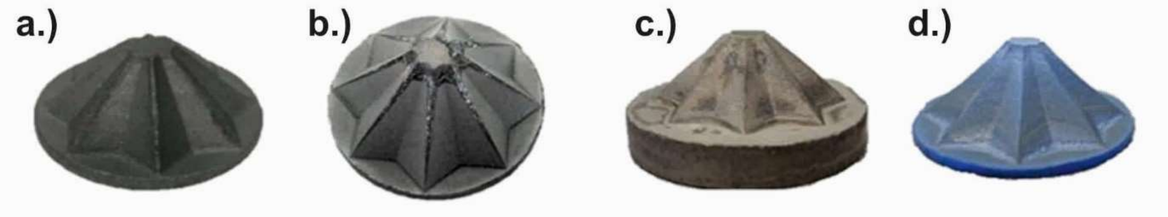

Figure 23: *Star-shaped prototypes sintered by the counter-form method. Demonstration for different materials:* ***a.)****TiAl* ***b.)*** *Al* ***c.)*** *ZrB_2* ***d.)*** *Y_2O_3 doped ZrO_2 (YSZ) (Courtesy of Norimat SAS).*

Hybridization of AM and FAST/SPS opens huge opportunities in the future. Both techniques are fully paired as AM opens the possibility to manufacture complex 3D parts in a FAST/SPS device. With this approach, FAST/SPS is capable of sintering custom materials directly in their final shape. Simultaneously, FAST/SPS could help overcome the limitations of AM, in regards to material performance and specific defects like residual porosity, which limits the use of AM for some applications. Finally, hybridization is an easy way to process materials, which are difficult to sinter, like high-performance composites or ultra-high temperature ceramics (UHTC), avoiding expensive secondary operation steps like machining or grinding.

5. Modelling of temperature and current density distribution (28)

For optimum tool design, with respect to homogeneous temperature distribution in the heated zone, reduced heat loss due to thermal radiation, and contact to the water-cooled electrodes, Finite Element Modelling (FEM) is highly recommended. Improvement of temperature distribution is based on optimization - e.g., of the wall thickness of the die, length of the punches, application of CFC plates, application of flexible foils between tool and sample, and the application of thermal insulation and additional heaters, as is the case in Hybrid FAST/SPS. Recently, a commercial software (Engemini) was introduced on the market (32), which can be used by customers to accurately estimate the temperature distribution for any material, and for a wide range of tooling setups. The software also includes a material database, so that the effort for the measurement of the input parameters is omitted. Engemini enables its users to reduce the effort required to develop new materials and perform tooling optimization significantly.

For accurate modeling, reasonable input parameters are required, or must be determined, in related experimental studies (21) (33). Most of the parameters, like electrical resistivity, are strongly dependent on temperature and density, and therefore are changing during the FAST/SPS cycle. Furthermore, there are some additional effects, which vary with the respective experimental set-up and are therefore difficult to consider in FEM. For example, the use of graphite foil can produce uncertainties due to anisotropic electrical resistivity and a less defined contact situation. The most important input parameters for the electrical-thermal-mechanical coupled FEM of FAST/SPS cycles coupling electrical, thermal and mechanical effects are:

- Tool geometry, including spacers, inserting foils or functional coating, and, optionally, thermal insulating felts.
- Temperature, atmosphere and density dependent material properties comprehending electrical resistivity, specific heat, thermal conductivity, bending and compressive strength, and emissivity.
- Sintering and pressure profiles.

Electrical and thermal contact resistances result from imperfect contact between adjacent components. In ideal case, perfect joining of the components enables the electric current to uniformly pass through the interface without current discontinuity. In real contacts, surface roughness of the contact area allows only partly current flow (**Figure 24**).

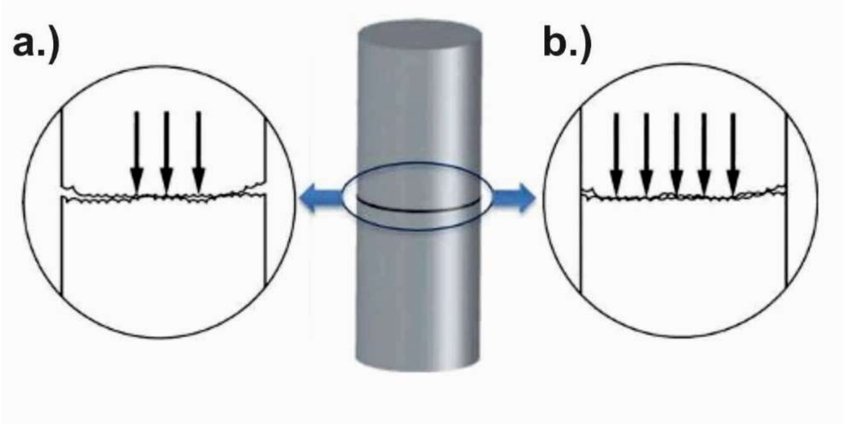

Figure 24: *Schematic sketch of a) bad contact b) good contact between adjacent components* (34)*. (Reproduced with permission of the American Ceramic Society).*

Contact resistances and their dependence on applied or resulting stresses are difficult to evaluate precisely. Nevertheless, due to their strong contribution to Joule heating, they require careful consideration to propose realistic predictions (35). For doing so, Manière et al. propose an experimental method (36), Van der Laan et al. a numerical one (37).

Most FEM studies neglect the influence of the specific sintering behavior of the starting powder. Instead, the material is estimated as a dense, elastic body. This assumption can cause large errors - in terms of the applied stress level - since a sintering material behaves like a viscous fluid. To overcome this restriction, specific constitutive sintering laws have been recently implemented in FEM codes.

Other attempts to reduce thermal gradients

It is obvious that the electrical resistivity of the tool assembly – consisting of two punches, die and spacers – dominates the temperature distribution within the tool and the sintering part. This resistivity can be modified in a controlled way by changing tool dimensions and/or tool material. Usually, the punches are the regions with the highest current densities during FAST/SPS processing. Axial temperature gradients can be controlled (e.g. by the length of the punches) while radial gradients are more difficult to handle. In a fundamental study done on a conductive metal alloy (48Ti–48Al–2Cr–2Nb), Voisin et al. (38) demonstrated that increasing the length of punches and die and, in parallel, decreasing the diameter of the spacers and the wall thickness of the die is a promising approach to reduce thermal gradients in the hot zone (**Figure 25**).

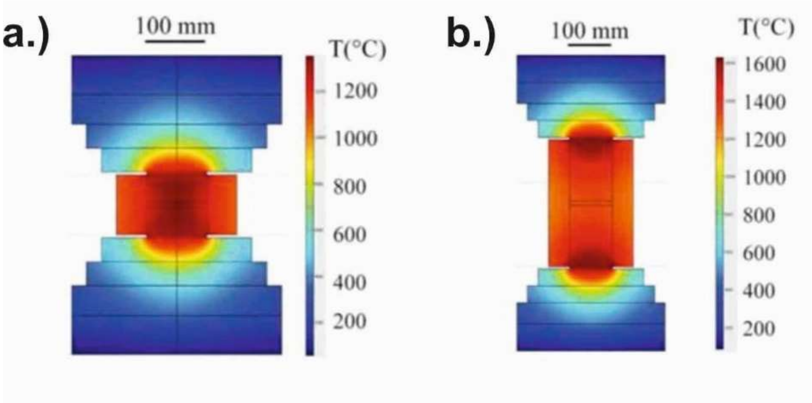

Figure 25: *Improving the temperature distribution in the hot zone of a FAST/SPS tool* ***a.)*** *Standard tool* ***b.)*** *Improved temperature distribution by increasing the length of punches and die, while reducing diameter of the spacers and wall thickness of the die (38). (Reproduced with permission of Elsevier).*

Giuntini et al. proposed another approach to handle temperature gradients in radial direction, when sintering non-conductive powders like Si_3N_4 (39). By removing material from the punches (drilling holes or machining concentrically rings, **Figure 26**), the current pathways were directed from the punch center leading to a more homogeneous current and temperature distribution. FEM showed a larger effect in the case of the ring configuration resulting in significantly reduced radial and axial thermal gradients when compared to normal, unaltered punches.

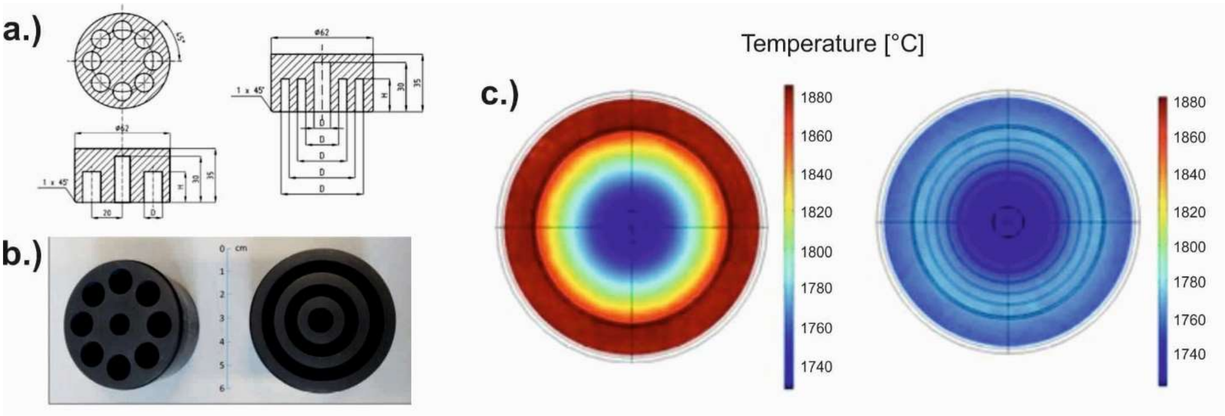

Figure 26: *Alternative punch designs for more homogeneous current and temperature distribution* ***a.)*** *Technical drawing of hole and ring design* ***b.)*** *Top view of both designs* ***c.)*** *FEM of the temperature distribution, comparison standard punch and punch with ring design (39). (Reproduced with permission the Ceramic Society of Japan).*

6. Case studies

6.1 Diamond tools (26)

Diamond tools are one of the oldest industrial applications of FAST/SPS and, as of today, this sector is also considered to be the largest in terms of volume and value. The main use of diamond tools is for cutting hard materials in the construction industry, such as asphalt, concrete, marble, granite and other stones. The tools consist of a metal bond matrix with embedded diamonds. The metal matrix holds the diamonds in place, while the diamonds cut the materials. Since diamonds are one of the hardest existing materials, they are able to cut other hard materials, and thus they are ideal for such cutting tasks. However, diamonds have a distinct disadvantage. When sintered at temperatures of 800 °C or beyond, diamonds tend to carbonize and consequently lose their cutting performance.

In order to prevent the diamonds from carbonizing, the sintering process for hardening the metal bonds has to be as short as possible. This was the main motivation behind why the German company Dr. Fritsch came up with the first industrial FAST/SPS sinter press in 1953. At that time, diamonds from natural repositories were being used, which made the diamond tools very expensive. For the diamond tool manufacturers, it was a huge loss if the diamonds carbonized. Furthermore, the short sintering cycles of FAST/SPS increased the productivity drastically and decreased the production costs at the same time – another huge motivation for the manufacturers to switch from traditional sintering to FAST/SPS. Today, synthetic diamonds are used for diamond tools in the place of diamonds mined from natural repositories. However, the carbonization risk remains, and the very high production volumes still require highly productive and economical production routes.

Nowadays, FAST/SPS is the standard production route in the diamond tool industry worldwide. Thousands of FAST/SPS devices are in operation worldwide in this industry sector. In recent years, many of the production operations moved to China, and the cost pressures have forced diamond tool manufacturers to increase their production volumes and to automate their production lines. **Figure 27** shows two examples of diamond tools.

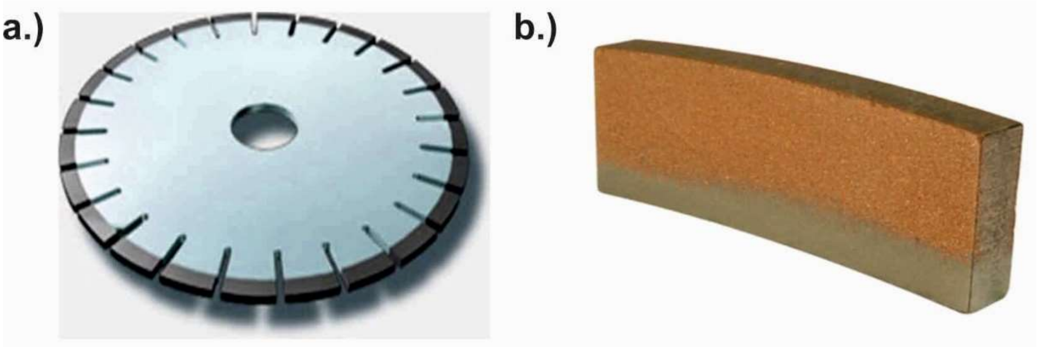

Figure 27: *Diamond tools manufactured by FAST/SPS* ***a.)*** *Diamond tool saw blade* ***b.)*** *Diamond tool segment with diamond-less area (Courtesy of Dr. Fritsch GmbH).*

6.2 Friction materials like brake pads (26)

FAST/SPS has been in use for the production of sintered friction materials for more than 15 years, and the number of producers - as well as applications - increase constantly. The main applications of sintered friction materials are for heavy-duty and racing vehicles as well as emergency braking systems. While conventional automotive brakes contain organic friction materials optimized with respect to drivers comfort and noise reduction, sintered friction materials are characterized by strong braking performance, low fading and resistivity to high temperatures. These characteristics make them the preferred solution for the abovementioned applications. One of the first markets was high-performing motorcycle brake pads and, up to now, this market still dominates. However,high-speed trains like the German ICE, French TGV and Japanese Shinkansen (and their derivates) are also relying on sintered brake pads in one of their braking systems. Likewise, even mountain bikes, e-bikes, rollercoasters, elevators and wind turbines are equipped with sintered brake pads.

Friction materials are not limited to brake applications. Highly loaded clutches are sintered parts as well, especially for heavy-duty applications like trucks and agricultural machines like tractors. So called “clutch-buttons” are part of every transmission gear. In countries like India, the aftermarket sales of sintered clutch-pads exceeds the number of OEM products (OEM = Original Equipment Manufacturer) by far, with many local producers. This is primarily due to the limited lifetime of OEM parts due to bad road conditions and stop-and-go-traffic.

FAST/SPS for sintering friction materials brings several advantages:

- Improved homogeneity of the brake pad microstructure due to the avoidance of or decrease of undesired grain growth.
- High production rates due to short sinter cycles, coupled with reduced production costs.
- Direct sinter bonding of the friction material on the brake´s backing plate without need of soldering and calibration. Skipping of production steps further reduces costs.

Compared to sintering friction materials in belt or bell furnaces, the advantages of sintering by FAST/SPS are obvious. Therefore, almost all large manufacturers operate FAST/SPS devices. **Figure 28** shows large-scale production of brake pads and clutch buttons by using multiple tools.

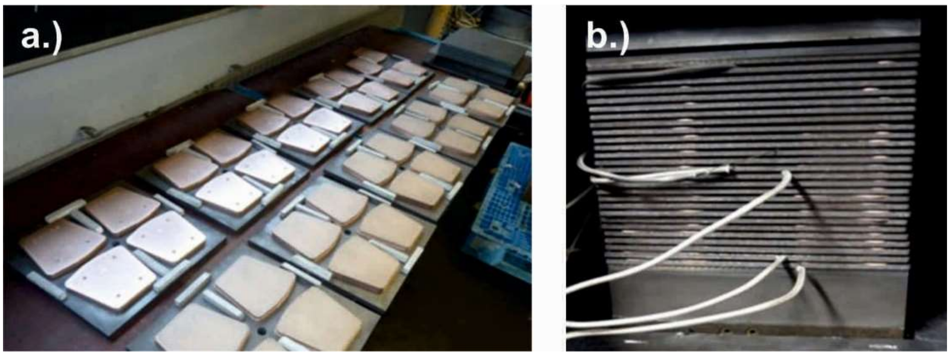

Figure 28: ***a.)*** *Brake pad samples* ***b.)*** *Sintering of 500 clutch buttons in a stack configuration (Courtesy of Dr. Fritsch GmbH).*

6.3 Sputter targets (26)

Functional coatings made by physical vapour deposition (PVD) have a broad spectrum of applications, e.g. for surface protection or improved aesthetic. In PVD, a thin film is deposited on the substrate by vaporizing a solid material in a vacuum chamber. Vaporization of the so-called sputter target is achieved by electron beam or by ion bombardment in a plasma environment. Atoms or molecules of the target break loose, diffuse through the chamber, and accumulate on the surface of the substrate. The PVD technique is used for coating tools, microchips, semiconductor devices, optical lenses, solar panels, medical devices, tools, perfume and liquor bottles, LDC-screens, food packaging and many other everyday products.

The key component of the sputtering process is the sputter target. The material used for the target defines the type of material that will coat the desired surface.. Sputter target manufacturers use a wide range of materials for many different applications. Additionally, specific additives help to align sintering temperatures of materials or to stabilize sputter targets in the case of harsh coating conditions. Today, most of the sputter targets are still hot pressed with rather long sintering times. However, manufacturing of sputtering targets by FAST/SPS is becoming more and more popular due to the following reasons:

- Reduction of the sintering cycle time to a minimum clearly reduces or even eliminates grain growth of the sputtering target, resulting in higher homogeneity, higher density, improved microstructure and, finally, improved coating performance.
- Compared to hot pressing, cycle time are reduced from 24 hours to only a few hours, which includes cooling. Reduction of cycle time is the main motivation for many sputter target

manufacturers for replacing hot pressing with FAST/SPS, especially for the production of small to medium batch sizes. While large quantities of standard targets are mainly produced in Asia with long shipping times, the production of specialized target requires much shorter delivery times. As a side effect, such products tend to have much higher margins when compared to sputter targets manufactured by standard mass production.

Manufacturers such as Dr. Fritsch developed FAST/SPS production machines which are optimized for sputter target manufacturers. Adapted chamber design and pressing forces enable to manufacture sputter targets with diameter of up to 400mm in stacks. After sintering, targets are automatically transferred into a cooling chamber without breaking inert gas atmosphere or vacuum. Separating the cooling cycle from the sintering cycle increases the productivity significantly. **Figure 29a** shows a sputter target mounted on a copper plate, **Figure 29b** a production line with separate FAST/SPS and cooling chamber.

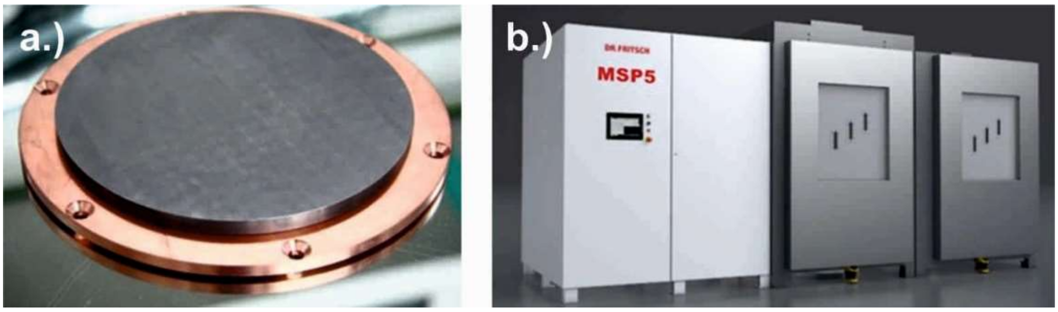

***Figure 29: a.)** Sputter target (Picture courtesy of RHP Technology GmbH) **b.)** FAST/SPS device Dr. Fritsch MSP-5 with MSC-5 (Courtesy of Dr. Fritsch GmbH).*

6.4 Functionally Graded Materials (26)

Even if it is not a specific product category, functionally graded materials are typical applications of FAST/SPS in several industries. The term “functionally graded” generally refers to alloys, which combine characteristic properties of several materials in an advantageous way. Often, quite different material properties must be handled, e.g. when combining materials with high and low sintering temperatures. Here, adapting the powders themselves or applying a sintering technique which enables to reduce the sintering temperature (as is the case for FAST/SPS), or a combination of both, comes into play.

A successful business case for functionally graded materials made by FAST/SPS are tungsten carbide-based wear parts used for caterpillars and cultivators in the lumber industry and in agriculture. The tracks of these heavy-duty vehicles are covered by wear-resistant tungsten carbide parts which need to be changed regularly. Up to now, a caterpillar or cultivator must brought to a garage for the exchanging of and mounting of new tracks. This costly maintenance service is aggravated by the fact that track mounting and exchanging keeps the machines out of operation for a long time. To replace this maintenance service, a weldable tungsten carbide containing material composite was developed using FAST/SPS as its main production route. Since tungsten carbide cannot be welded itself, a functionally graded composite consisting of several layers of steel materials with a tungsten carbide layer on the top and a soft, easily weldable steel at the backside. FAST/SPS is an ideal sintering technique for such types of composite materials due to the capability to monitor and adjust temperature and pressure with high precision, provided that the device is equipped with adequate sensors and software. The intermediate phase causes a strong bonding between the wear resistant layer and the welding seam. **Figure 30** shows a related functionally graded composite material.

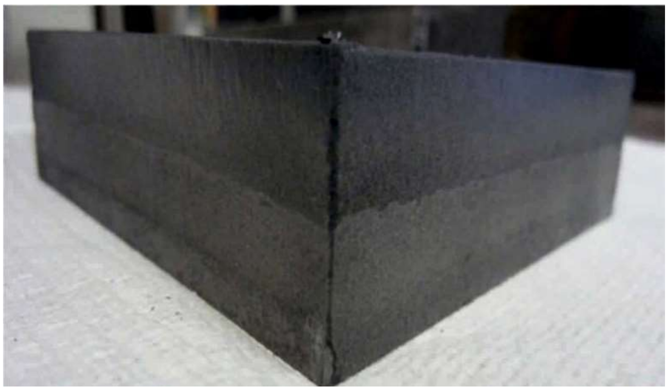

Figure 30: *Functionally graded composite material connecting a wear resistant tungsten carbide layer with a weld able steel support (Courtesy of Dr. Fritsch GmbH).*

6.5 Filter element (28)

Norimat has developed an Inconel 718 filter using the hybrid method that combines binder jetting and FAST/SPS (**Figure 31**). The advantage of using this technology compared to conventional processes, such as casting and machining, is the ability to limit the production time of the parts while still obtaining parts with high mechanical performance. The geometry obtained is more accurate than what can be achieved with a machining process, simultaneously avoiding a material loss of nearly 60%. Wall thickness is around 500 µm with very limited deformation due pressure applied along the z-axis during the FAST/SPS process.

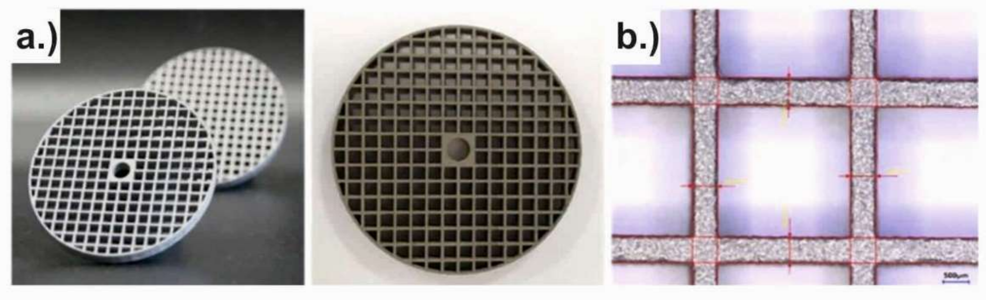

Figure 31:* *a.)* *Filter made of Inconel 718* *b.)* *Photo of the filter wall (Courtesy of Norimat SAS).

6.6 Colored ceramics for watches (28)

Norimat has developed colored ceramics for the watch market. The implementation of colour pigments in ceramics was limited by the process conditions needed for the densification of the parts. The high temperatures and firing times of conventional sintering processes are too high and deteriorate the pigments. As result, parts emerge with a pastel or burned color. The kinetics of FAST/SPS sintering considerably reduce the time the pigments are exposed to heat. The color pigments are therefore preserved, giving the ceramic its full brilliance and shine (**Figure 32**). The combination of heat and pressure makes it possible to obtain not only colored ceramics, both at the core and on the surface, but also to achieve mechanical properties passing the most demanding tests in the watch making industry, whether in terms of impact resistance or machinability.

Figure 32: *Watch bezel made of coloured ceramics (Courtesy of Norimat SAS).*

6.7 Multicolor materials for jewelry and watch applications (40)

"Tiger Metals" and "Tiger Ceramics" are powder technological manufacturing concepts where powders are prepared into a proper granule size. After blending two or more materials, rapid densification of the mixture via FAST/SPS follows. Ideally, materials with a different optical color are combined resulting in a unique macrostructural appearance (**Figure 33**). Depending on the specific properties of the granules, a large variety of pattern becomes possible. In these patterns, it is very important to create a clear boundary between the elements or alloys. This requires a careful selection of processing conditions in order to not only minimize diffusion or reaction, but to also ensure a full density and well bonding of the individual phases. Various combinations have been realized such as silver-gold, silver-titanium combinations as well as various mixtures between red gold/white gold or yellow gold. Additionally the direct bonding with other precious metals is possible as shown in the **Figure 34**.

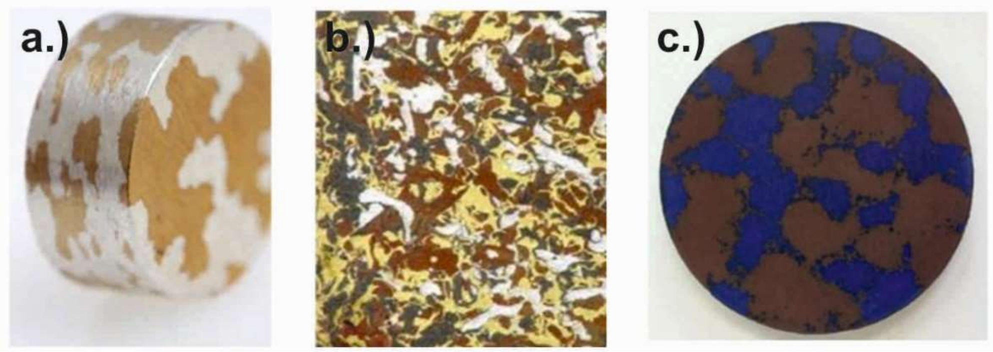

Figure 33: ***a.)*** *"Tiger Metal" realized in silver-gold* ***b.)*** *"Tiger metal" as multi-material patterns (consisting of four different metals)* ***c.)*** *Characteristic pattern of a "Tiger ceramic" based on oxide ceramics (Courtesy of RHP Technology GmbH).*

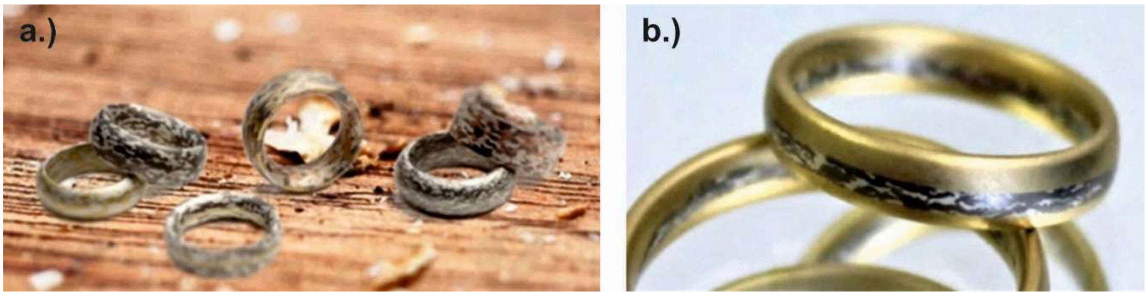

Figure 34: *Rings made from "Tiger Metals"* ***a.)*** *Design 1* ***b.)*** *Design 2 (Courtesy of RHP Technology GmbH).*

6.8 Multi-layer materials and sandwich structures (40)

FAST/SPS technology also enables the diffusion bonding of different materials. Applications for such kind of multilayers once again include the jewelry and watch making industry, where, for example, multilayers of various precious metals are used to create the starting material for the so-called Mokumegane. In this case, sheets are prepared and bonded by a pressure-assisted technique, followed by extraction of elements, which are deformed subsequently in order to create a "wood" like pattern. Additionally, the same concept can be also applied for the manufacturing of a material with tailored thermophysical properties. The combination of metal sheets of copper and molybdenum or copper and tungsten creates material composites with low coefficient of thermal expansion and high thermal conductivity. Such composites are suitable for heat sinks in electronics. **Figure 35** shows selected examples.

*Figure 35: **a.)** Multilayer structure **b.)** Sandwich structure of silver-copper **c.)** Combination of copper and molybdenum in multilayer arrangement (Courtesy of RHP Technology GmbH).*

6.9 Heat sink materials for electronic applications (40)

The increase of the power density in electronics requires materials with tailored thermophysical properties. W-Cu or Mo-Cu are material composites which allow for the tailoring of the coefficient of thermal expansion in a range of $6 - 10 \cdot 10^{-6}$ K^{-1} while keeping a high thermal conductivity of 200 – 300 $W \cdot m^{-1}K^{-1}$. For applications where even higher thermal conductivities (>400 $W \cdot m^{-1}K^{-1}$) are required, it is necessary to include fillers in metallic matrices such as diamond. Copper-diamond or silver-diamond composites offer a new generation of thermal management materials. In order to achieve a high surface finish or a machined structure, it is necessary to prepare these materials as sandwich structure consisting of metal/metal-diamond/metal layers, which are machined subsequently to their final contour (**Figure 36**).

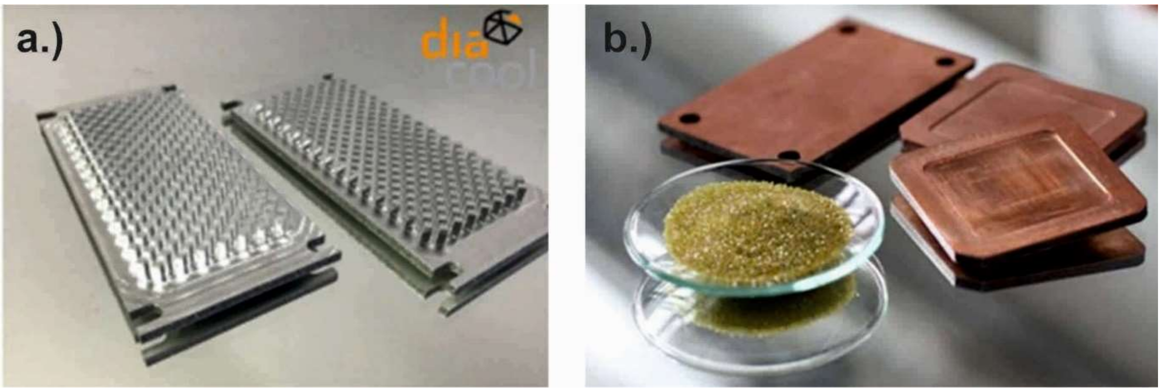

Figure 36: ***a.)*** *Aluminium-diamond sandwich structure with pin fin structure on one side* ***b.)*** *Base plates and head spreaders for high performance computing applications (Courtesy of RHP Technology GmbH).*

7. Trends and outlook

Despite its establishment in industry for more than 50 years, FAST/SPS and related technologies are still niche technologies which are limited to material systems difficult to process by other technologies. The following challenges limit broader application:

- Maximum heating rate and temperature limited by tool design and sample size
- Large dimension parts with diameters beyond 100 mm require careful adjustment of temperature profiles to avoid failure (41)
- Occurrence of temperature gradients might cause density gradients and residual stresses
- Exact temperature measurement and temperature control
- Limitations with respect to high cooling rates, standard cooling rates below 150°C/min
- Realization of complex shapes
- Chemical reactions between sample and tool material
- Reducing conditions that may cause oxygen release from oxide ceramics
- Mismatch of thermal expansion between sample and tool
- Change from graphite tools (moderate conductivity) to metal tools (high conductivity) might require adaption of the Proportional-Integral-Derivative (PID) controller for accurate temperature control
- Productivity issues, especially due to limited cooling rates and elaborated filling and mounting of the tools

Special kinds of electric field assisted sintering

In recent years, great efforts have been undertaken to make ECAS processes more effective and to expand the range of applications. Progress is based on, amongst other factors, significant increase of heating rates, reducing thermal gradients, triggering new modes of densification as well as decreasing maximum sintering temperature and dwell time. It has been shown that especially high heating rates are a promising approach to significantly accelerate sintering kinetics, resulting in full densification of ceramic and metal powder compacts in seconds. Nevertheless, most of these new technologies are still in lab- or prototype scale. **Figure 37** gives an overview of the different kinds of novel sintering technologies. For more details, we refer to literature.

Hybrid FAST/SPS

Hybrid FAST/SPS devices are on the market, which, in addition to standard FAST/SPS operation mode, enable the superposition of induction or resistance heating by placing an external heating element around the FAST/SPS tool (17) (27). This concept can help to further increase heating rates and thermal homogeneity – however, italso allows for the creation of temperature gradients on purpose. For high-throughput production, hybrid FAST/SPS plants with additional pre-heating and cooling zones are available (see **Section 2.4**). Another technical option is the combination of a FAST/SPS device with an additional external AC or DC power source with maximum voltage of up to 1000 V. With such device, it becomes possible to initiate flash sintering of ion- or semi-conducting materials, which requires high electric fields to force current flow through the sample initiating direct Joule heating of the sample.

Flash sintering

In 2010, flash sintering was introduced as novel sintering mode (42) (43). A current flow is forced through a powder compact made of a semi-conducting material by applying a suitable combination of electric field and temperature. Usually, electric fields up to several 100 $V\cdot cm^{-1}$ are applied, while current density during the flash remains in the range of a few $A\cdot cm^{-2}$. For initiating the current flow, there must be electrodes placed on the sample surface. No load or a fairly moderate load is applied on the electrodes, mainly to improve the contact. Heating of the sample during flash is primarily based on resistance heating (Joule heating). There are different possibilities to control flash sintering. In voltage-to-current control mode, an electric field is applied to the electrodes, and the sample is heated up by an external heating element. When a material- and sample-specific onset temperature is exceeded, current starts to flow through the sample, accompanied by luminescence. After the onset of flash, avalanche-like increase of current leads to heating rates beyond 1000 K/min, followed by the densification of the sample in seconds. To avoid melting of the sample, current density must carefully controlled after the onset of flash by switching the power source accordingly. As an alternative mode of operation, in current-rate or power-rate controlled flash sintering, the heating current/power is increased either linearly or stepwise to a distinct value while the sample is placed in a heated furnace (44). This mode enables better control of the flash sintering, but it may take several minutes to achieve full densification. Due to the almost complete dissipation of the heating power by the sample, flash sintering is discussed to be very efficient and economic, but scaling up of technology is still limited by risk of localized current path formation, especially when enlarging the sample volume. Flash sintering can be conducted in a hybrid FAST/SPS equipped with external power source and electrodes, which can be placed between punch and sample.

Flash Spark Plasma Sintering (Flash SPS)

A process similar to flash sintering can be conducted even in a conventional FAST/SPS setup without external power source. Literature introduces this process as flash spark plasma sintering (Flash SPS) (45) (46). Potential of Flash SPS has been demonstrated on lab-scale for ceramic and metal powder compacts (47). If applied to semi-conducting ceramics, an external heater or a specific tool design is required to achieve temperatures for initiating Flash SPS. For conductive powders, the possibility of direct Joule heating of the sample eases the preheating. During Flash SPS, densification is supported by applying a well-defined mechanical load via the punches of the FAST/SPS device. Often, minimum load required for FAST/SPS operation is sufficient for achieving high sample deformation. Characteristic parameters of Flash SPS are voltage below 10 V and very high heating rates in the range of $10^4 – 10^6$ K·min^{-1}. Rapid densification relies on a DC current pulse of several 10 kW with a defined time length. This enables the sudden supply of extremely high heating power. To avoid overheating and sample melting, maximum power of the current pulse must be limited. Estimation of temperature distribution during Flash SPS by numerical simulation indicated the occurrence of large temperature gradients up to several 100°C, which is critical for achieving homogeneous microstructures.

FAST-forging

Another new method for processing net-shaped parts from metal powders is a process called FAST-forge, a two-step solid-state hybrid-manufacturing route (48). Here, FAST/SPS is used to produce a shaped preform billet followed by a one-step precision hot forge in an external forging device resulting in near net shape consolidation. If forging is done in combination with direct Joule heating of the sample, electroplasticity is discussed as additional mechanism supporting material deformation (49).

Electro sinter forging (ESF) and Electro discharge sintering (EDS)

Other promising alternatives for ultra-fast densification of conductive powders in milliseconds are electro-sinter-forging (ESF) (50) and electro-discharge-sintering (EDS) (51). Both technologies are quite similar and enable high heating rates up to 10^6 K/min by the sudden release of energy stored in a capacitor via the sample. Highly conductive punches made of copper or copper alloys are required to conduct ESF and EDS cycles, which are sensitive to wear, especially at their faces. Spontaneous capacitor discharge is difficult to control making reproducibility of the process challenging. The main difference between ESF and EDS is the kind of applying the load. In the case of ESF, the load continuously increases during the capacitor discharge. In EDS, maximum load is already applied before discharge. Usual loads are in the range of 50 MPa to several 100 MPa. ESF and EDS are limited in sample size. When exceeding a diameter of 20 mm, risk of forming localized current paths increases.

Ultra-fast high temperature sintering (UHS)

Another novel technology for realizing heating rates up to 10^4 K·min^{-1} and temperatures up to 3000°C is ultra-fast high temperature sintering (UHS) (52) (53). Here, a powder compact is placed between two flexible, conductive felts (e.g. made of graphite). Rapid heating of the felts by Joule heating and heat transfer to the sample by thermal conduction enables heating rates up to 10^4 K·min^{-1} followed almost complete densification of ceramic compacts in a few seconds. Recently, such setup was operated successfully even in a FAST/SPS device (53) (54). Demonstration of this technology is still limited to lab-scale.

Cold sintering

Recently, cold sintering has gained a lot of interest in the sintering community (55) (56) (57). The application of water or other liquid based sintering aids in combination with high pressure of several 100 MPa enables almost full densification of oxide ceramics at temperatures below 500°C. Solution/precipitation, often accompanied by decomposition of the main phase are mechanisms involved in low temperature densification. Therefore, post thermal treatments are often required to regain the main phase for the aspired functional properties. Conducting cold sintering cycles in a FAST/SPS device is an attractive alternative to cold sintering in less-instrumented heated uniaxial press due to its easier process control with respect to heating rate, temperature, load, atmosphere, and sample displacement.

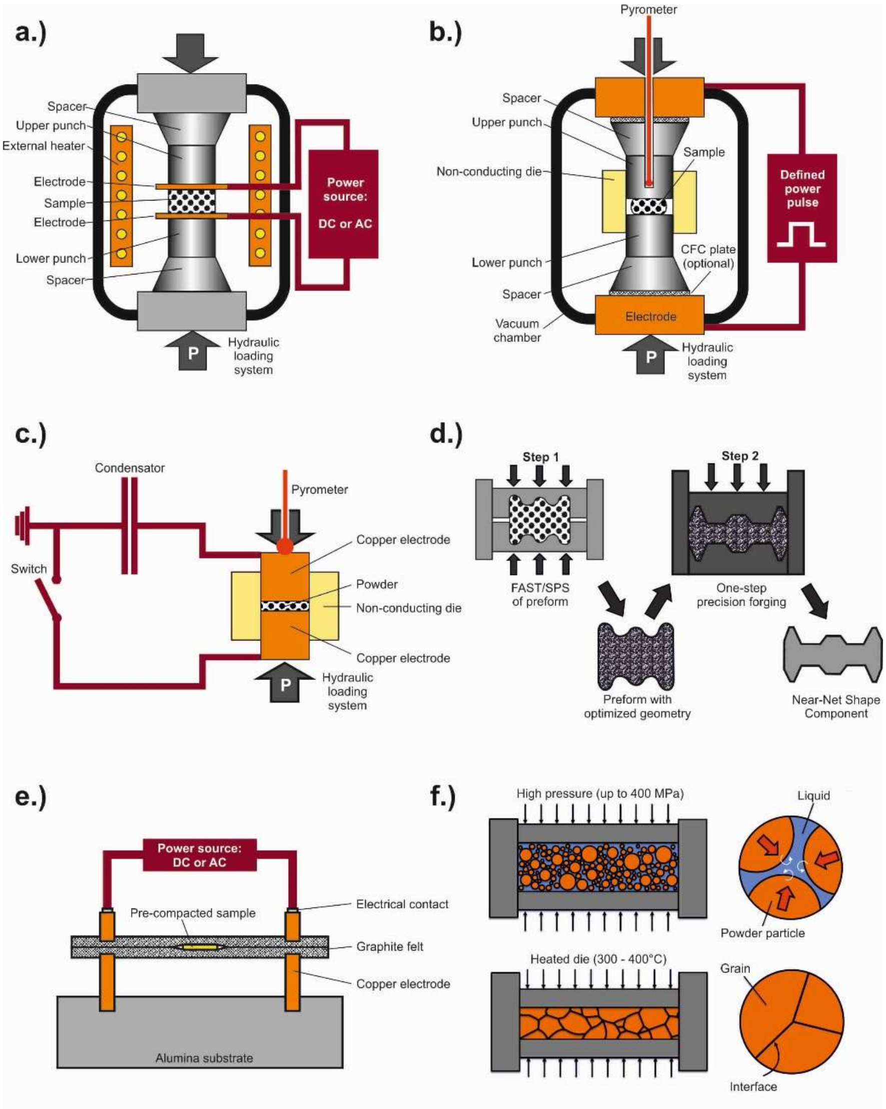

Figure 37: *Overview of novel sintering technologies* ***a.)*** *Flash sintering* ***b.)*** *Flash spark plasma sintering* ***c.)*** *Electro discharge sintering* ***d.)*** *FAST forging* ***e.)*** *Ultra-fast high temperature sintering* ***f.)*** *Cold sintering.*

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9. Contributors

Prof. Dr. Martin Bram
Forschungszentrum Jülich GmbH
Institut für Energie- und
Klimaforschung
IEK-1: Werkstoffsynthese und
Herstellungsverfahren
D-52425 Jülich
Germany
Phone +49 2461 61 68 58
m.bram@fz-juelich.de
(Concept of booklet)

Prof. Dr. Olivier Guillon
Forschungszentrum Jülich GmbH
Institut für Energie- und
Klimaforschung
IEK-1: Werkstoffsynthese und
Herstellungsverfahren
(Institute director)
D-52425 Jülich
Germany
Phone +49 2461 61 51 81
o.guillon@fz-juelich.de

M.Sc. Monica Keszler
Forschungszentrum Jülich GmbH
Institut für Energie- und
Klimaforschung
IEK-1: Werkstoffsynthese und
Herstellungsverfahren
D-52425 Jülich
Germany
Phone +49 2461 61 38 17
m.keszler@fz-juelich.de

Yannick Beynet
Dr. Antoine Van Der Laan
Norimat
51 rue de l'innovation
31670 Labège
France
Phone +33 6 26 36 40 58
beynet@norimat.com
vanderlaan@norimat.com

Jens Huber
Dr. Fritsch Sondermaschinen GmbH
Dieselstr. 8
D-70736 Fellbach
Germany
Phone +49 711-51832-221
jens.huber@dr-fritsch.de

Benjamin Luthardt
Dr. Jürgen Hennicke
FCT Syteme GmbH
Gewerbepark 16
96528 Frankenblick
Germany
Phone +49 36766 824 227
b.luthardt@fct-systeme.de
j.hennicke@fct-systeme.de

Dr. Erich Neubauer
RHP-Technology GmbH
Forschungs- und Technologiezentrum
A-2444 Seibersdorf
Austria
Phone +43 2255 20600 110
e.ne@rhp.at

Dr. Johannes Trapp
Fraunhofer Institute for Manufacturing
Technology and Advanced Materials
IFAM
Winterbergstr. 28
D-01277 Dresden
Phone +49 351 2537 392
johannes.trapp@ifam-dd.fraunhofer.de