"FAST" field assisted sintering technology- a new process for the production of metallic and ceramic sintering materials

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1 Introduction

Spark Plasma Sintering (SPS) – also called FAST (Field Assisted Sintering Technology) – is a new and innovative sintering technology, which is becoming increasingly important in the processing of numerous materials, such as nano-structured materials, composite materials and gradient materials. The process is based on a modified hot pressing process in which the electric current runs directly through the pressing mould and the component, instead of by an external heater. By means of the pulsed electric current and the so-called "spark plasma effect", very rapid heating times and short process cycles are achieved. This suppresses granular growth and the achieving of balanced states, which allows the creation of materials with compositions and properties not obtainable up to now, materials in the submicron or nano-scales, and composite materials with unique/unusual compositions.

This very promising process was developed by the Japanese company Sumitomo Coal Mining Co., Ltd., and according to the company, it is the most widely distributed innovative sintering process, with around 250 units (of which 4 are in Europe). As part of a project supported by the EU, a similar process (FAST = Field Assisted Sintering Technology) was developed in recent years to a marketable level by FCT Systeme GmbH (for principle, see **diagram 1**).

Based on decades of experience and the successful application of classical hot press technology, the development of this very promising sintering process was started at FCT Systeme GmbH approximately eight years ago. This was based on the thought of using very rapid sintering processes to create new ways for, on the one hand, the more economic production of sintering materials, and on the other the hand, the production of materials which could not be consolidated by means of the normal compression processes of the time.

There is relatively little information available on the industrial application of this new manufacturing technology, as the industrial companies using the process are naturally secretive about the practical implementation of the process.

The significant level of R&D work carried out in recent years in research and in industry is however a clear index of the high significance given to this production technology.

2 The FAST technology

The Spark Plasma Sintering process (SPS), also called Field Assisted Sintering Technology (FAST) is also known in the literature under the terms Field Activated Sintering Technology or Pulsed Electric Current Sintering (PECS).

SPS/FAST was developed with an eye to the generally established processes of hot press technology, though in the SPS process, the press mould and blank themselves are directly heated. This happens either through the supply of energy from the outside via the press mould and/or by direct current flow through the blank itself. Supply of energy occurs through the use of a specially designed source of DC pulses which provide the user with freely variable options for pulse control within a fixed range, and for adaptation to the prevailing conditions in the blank (mould) and especially in the material itself. The design of the DC pulse source is therefore of particular importance.

The basic design of such a FAST unit is shown in **diagram 1**.

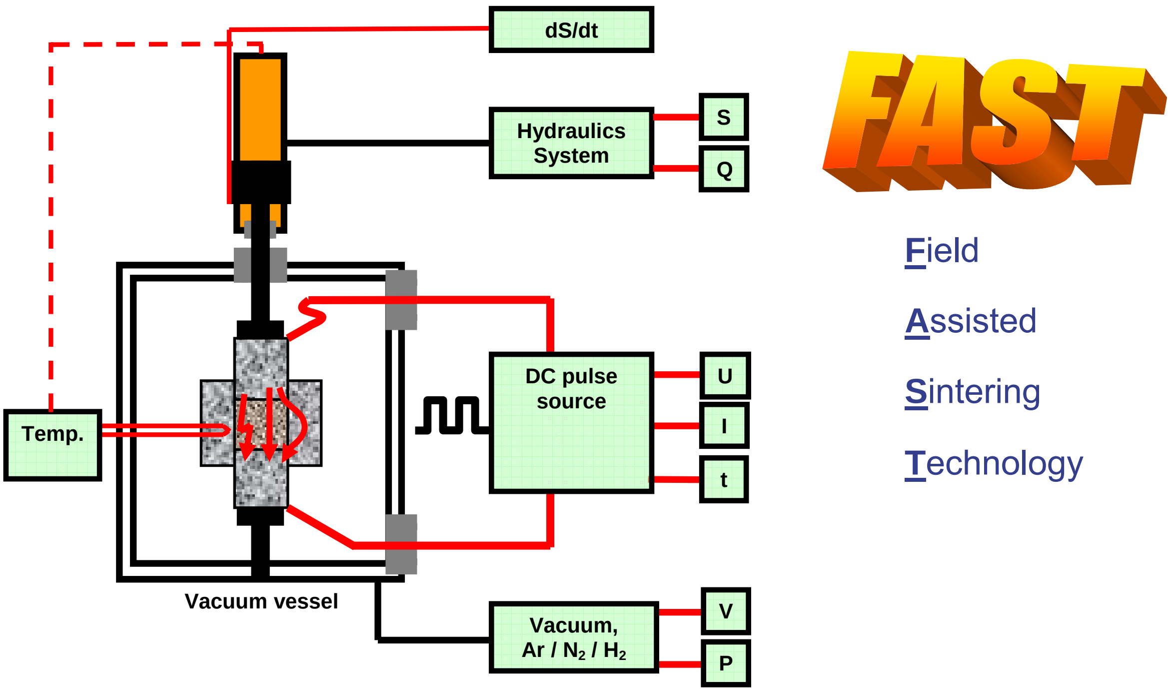


Diagram 1: *The SPS/FAST principle: Spark Plasma Sintering*

The basic theory of SPS/FAST heating depends on the fact that the pulse current passing through the mould causes the granular boundaries of the initial powder particles to partially heat up, and creates an electrical field with a plasma effect (for schematic presentation, see **diagram 2**). Here, the type and form of the electrical pulse, as well as its duration and strength, play a decisive role in achieving the desired SPS effect. The degree to which the present theoretical assumptions are 1:1 applicable to practice has not been explained in full detail and free of scientific doubt. However, observations during the consolidation process very strongly indicate the existence of the assumed cause and effect relationships. This applies particularly to materials which conduct or partially conduct electrical current, but also to ceramic materials which show electrically conductive properties only in the high temperature range. [**1**,**2**]

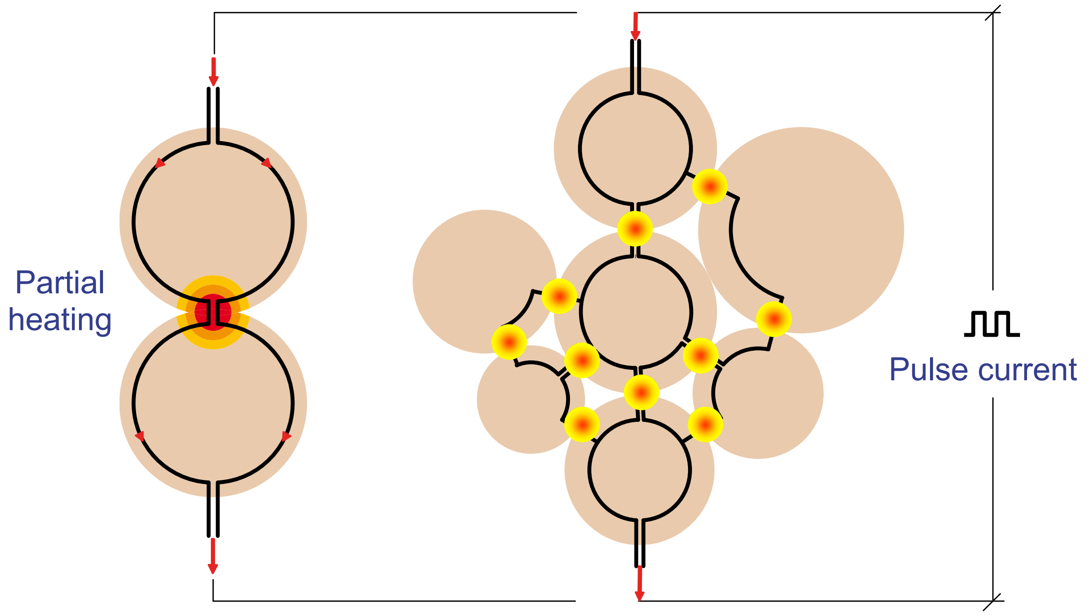


***Diagram** 2: The theoretical principle of FAST heating*

As mentioned above, the programmable DC pulse source represents a significant factor for the success of the FAST process. Accordingly, the greatest attention was paid to it even at the start of the extensive design work for the complete unit. In comparison to the relatively rigid functioning of the Sumitomo current supply, a declared goal of the R&D work at FCT Systeme GmbH was from the very beginning to develop a fundamentally more flexible pulse current source; this would enable the complete system to fulfil the relevant requirements for very varied applications, in order to achieve the desired SPS effect. The developers and designers at FCT Systeme benefited from, on the one hand, being able to access extensive company know-how on the construction of high temperature units, as well as rapid transistor power semi-conductors (IGBT), which had been available for only a few years.

The power unit which is now successfully implemented in FAST units enables symmetric loading of the supply network and prevents a phase shift on the network (cos φ); this is a great advantage especially at high operating ratings such as are becoming necessary for practical FAST applications.

The switching used by FCT Systeme GmbH allows the desired DC pulses with short pulse duration to be achieved without any problems. However, pulse deformations through the downstream high current transformer cannot be fully avoided (output current: 10 to 60 kA) (**Diagram 3**).

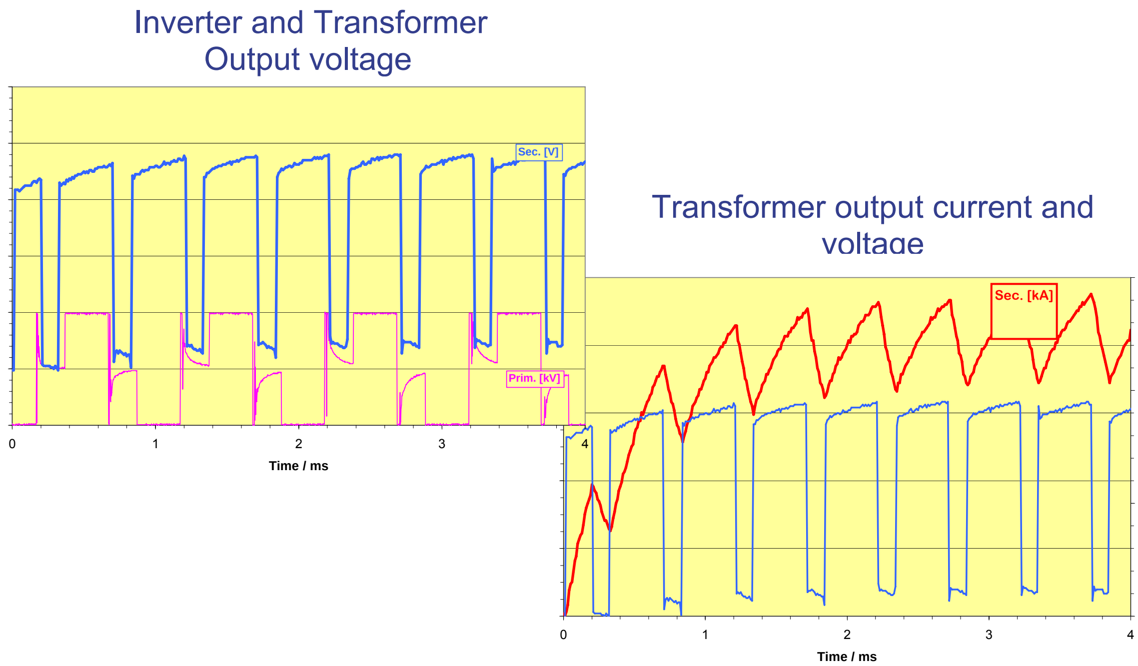


Diagram 3: *Creation of the DC pulses*

Diagram 4 very clearly shows the high flexibility of the DC pulse source used as per the principle presented, where pulse form, length, and pause as well as pulse group length and pause can be flexibly adjusted within very wide ranges, in order to be able to fully exploit the desired SPS effect. These properties of the power unit are necessitated by the high flexibility of the complete system and its suitability for the development and production of high performance materials.

Sophisticated software supports the user in the exploitation of the wide application range.

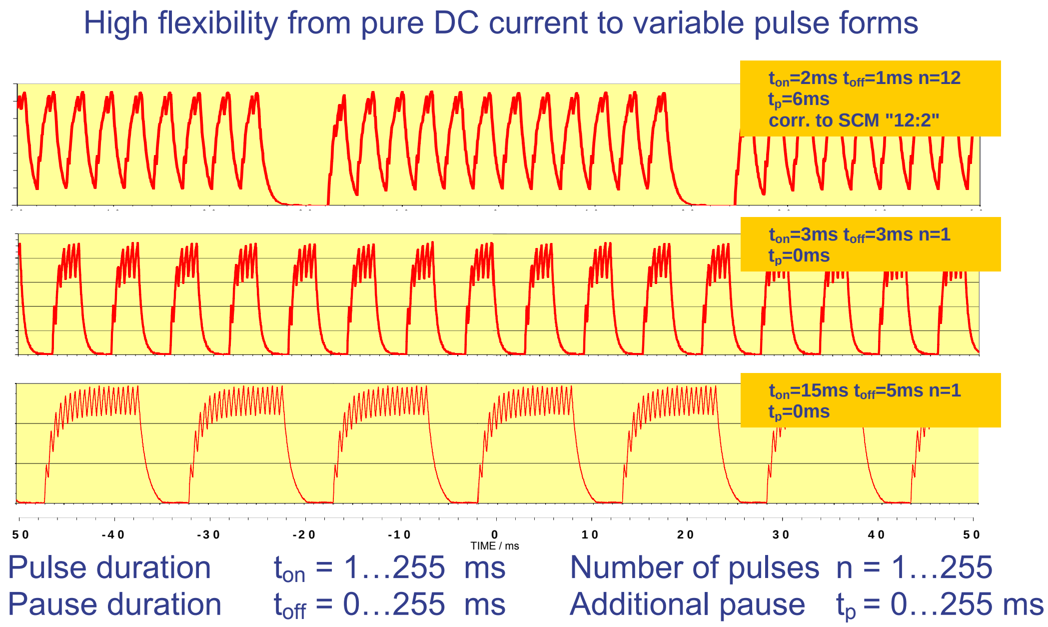


Diagram 4: *Pulse form modulation*

Even in the initial phase of the development work, it was quickly recognised that particular attention had to be paid to the predicted close relationship between mould layout, component material (blank) and the energy supply to the unit. Detailed knowledge of the critical parameters, both in terms of process technology and of material, is a prerequisite for the focussed and successful development of the FAST heating process. The highest priority is therefore assigned from the very start to simulation of the heating process, which will be described in more detail later. Even the initial work confirmed the expected very significant difference from classical sintering processes (especially the hot pressing technique related to the FAST technology), resulting from the direct heating of mould and blank.

By using more homogeneous starting materials, and with the aid of the FEM (Finite Element Method) for simulating heat distribution in the mould and test body, important understanding of the SPS/FAST theory was gained. This formed the basis by which focussed further work on the practical application (actual components) could be accelerated (**Diagram 5**). [**3**, **4**,**5**,**6**]

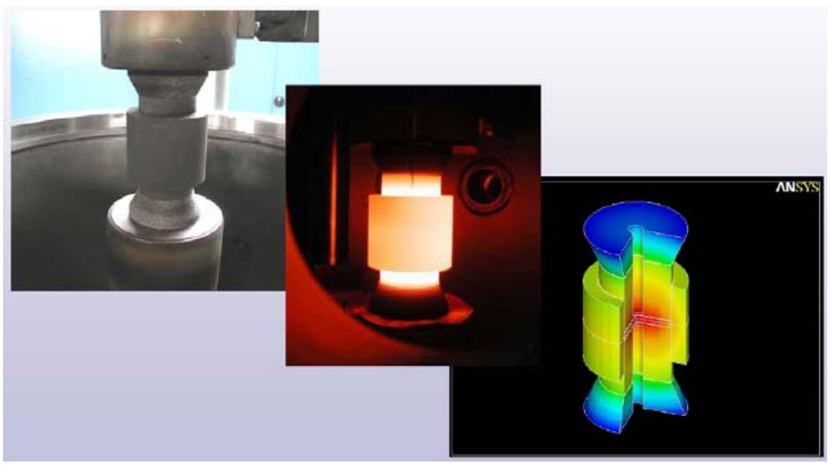


***Diagram 5**:* *Simulation of the FAST heating process*
(left to right: mould before and during the process, FEM simulation)

As part of the already mentioned European Research Programme for the development of FAST technology (2002 – 2006) particular focus – also because of the available key skills in the company – was placed on the consolidation of ceramic materials, compound materials, and composite materials for processing temperatures up to 2200°C.

As early as September 2003, after a relatively short development period, the first test unit was ready - this was the first European proprietary development (**Diagram 6**). It is equipped with a power unit with a current rating of 8,000 A, and it allows a maximum blank diameter of 80 mm.

Development goal:	Ceramics up to 2200°C
Project number:	GRD1-2001-40737
Unit type:	HP D 25/0
Main characteristics:	8 kA, 80 mm Ø, 2200°C, 250 kN
Ready for operation:	09/2003

Diagram 6: The first European SPS unit

In spite of wide-ranging power supply modifications which were required to overcome the unusually high currents combined at the same time with short DC pulses, this unit fulfilled all the expectations placed in it. It formed the starting point for the successful further development of the SPS/FAST concept for practical industrial applications.

3 FAST/SPS unit development

Building on the knowledge gained from the two year development phase, the focussed development of our own family of FAST units was begun. This was fundamentally oriented towards requirements from research and industrial applications (**Diagram 7**).

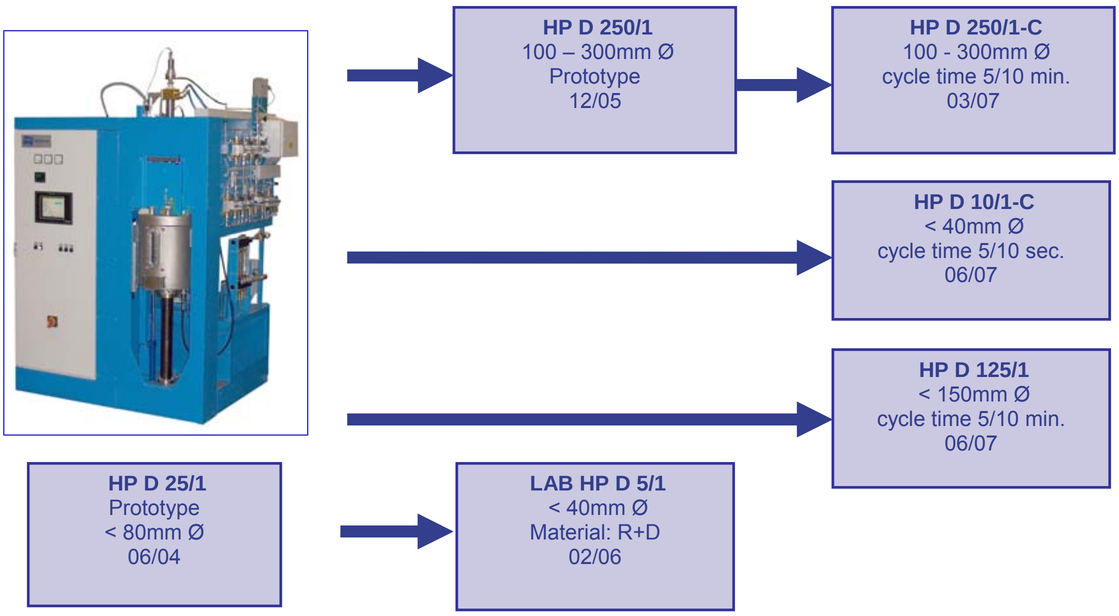


***Diagram** 7:* *The FAST unit HPD family*

The FAST/SPS units, until now sold worldwide by FCT Systeme GmbH, which are used very successfully both for research and development, and also in industry, are shown in diagram 8.

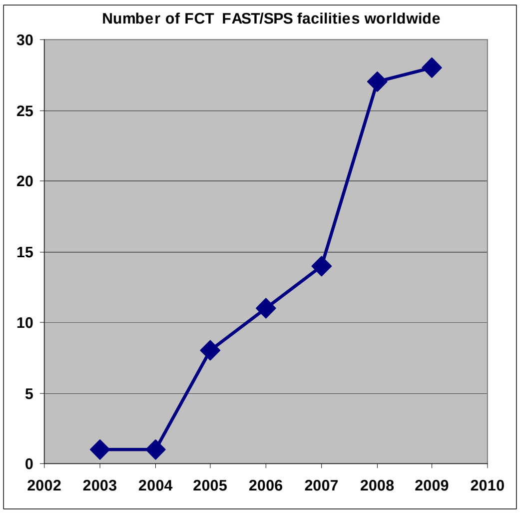


***Diagram 8**:* *The first European SPS unit*

The first prototype (HPD 250/1) has a maximum pressing force of 2500 kN. It is particularly suitable for product-oriented use in research and development, where, other than purely material-specific aspects, component properties play a significant role (e.g. bending strength, creep strength, tensile strength etc).

After only a short time, a desire arose in the industry for units of different types. On the one hand, there was a demand for smaller units which allow high output at short cycle times, and on the other, for large units for application tests with large format components. Industry-oriented institutes displayed a striking interest in large units with pressing forces of up to 250 t for component-related development work.

Concepts for two significant unit types were developed by FCT Systeme GmbH:

- a quasi-continuously operating FAST unit with diameters up to 300 mm and cycle times of 5 to 10 minutes. as well as
- various fast-running units with an eye to the TPA (dry-pressing automat) technology, with cycle times of 1 to 3 minutes or 5 to 10 seconds.

These types are designed for industrial production.

For the industrial implementation of material-specific development work, a FAST unit with 2500 kN pressing force, 60000 A max. pulse current, and 400 kW power is currently available (HPD 250, **Diagram 9**). Such a unit is currently in use at Fraunhofer IFAM, Dresden, for the execution of basic trials.

The suitability of these high output units for the production of real components from ceramic and metallurgic powder materials, as well as composites and combination materials has already been confirmed in many areas [**7**,**8**]. The "teething problems" of this type of unit - mainly a result of difficulties in connection with the extremely high currents and application temperatures (2200°C) - have been overcome. While the development of industry-relevant concepts is continually being worked on, the next step is the series manufacture of components with very specific material properties.

Type: HPD – 250 / 1 :

Pressing force	100…2500	kN
Piston stroke	0…200	mm
Piston velocity	0…4	mm/s
Temperature	RT…2,400	°C
Gas pressure	$5 \cdot 10^{-2}$…1,100	mbar
Pulse voltage	0...10	V
Pulse current	0...60.000	A
Pulse time	1...1000	ms
Pause time	0...1000	ms

***Diagram** 9:* *The FAST production system*

Diagram 10 shows a semi-continuous production unit of type HPD250C. This is today already in multiple use in industry; its uses include the industrial manufacture of sputter targets.

***Diagram 10**:* *FAST (SPS) production unit: HP D 250/C, semi-continuous operation*

4 Influence of temperature measurement

As in all thermal consolidation processes, the temperature measurement is of great importance. Because of the exceptionally rapid FAST process flows, old and trusted temperature measurement processes can often no longer be used. Therefore it is even necessary sometimes to turn back to comparative measurements (energy supplied) - at very high process speeds the only practical solution, after all. The positioning of the measurement points is decisive for the quality of temperature measurement, in order to gather meaningful temperature values in a "physically clean" and correlatable manner.

In the classical hot press process, a significant temperature gradient (spatial temperature difference) occurs between the surface parts and the centre of the blank during processing, when Joule's heat energy is provided from the outside (from induction or resistance heating), especially for voluminous components. This has the effect that the production of voluminous components using hot press technology is limited in terms of component homogeneity and material properties. Additionally, a hot press cycle requires a relatively large amount of time, because the actual application of pressure can really take place only after a suitable period of time for temperature balancing.

Both these weaknesses of hot press technology, lack of component homogeneity and long cycle times, can be overcome by means of the SPS/FAST process. The application of force can take place in a single step (full press force from the very start) or also in several steps. However, with optimum process and mould design, the former is to be preferred (**Diagram 11**).

For the unavoidably high heating rates needed to achieve the intended material properties at the same time as shortening cycle times, optimum temperature measurement is, as already stated, extremely important. Thus, derived from our own successful hot pressing concepts, a measurement concept was developed which enables the temperature to be measured directly on the die which is to be consolidated. For the rapid SPS technology, this is the one and only way.

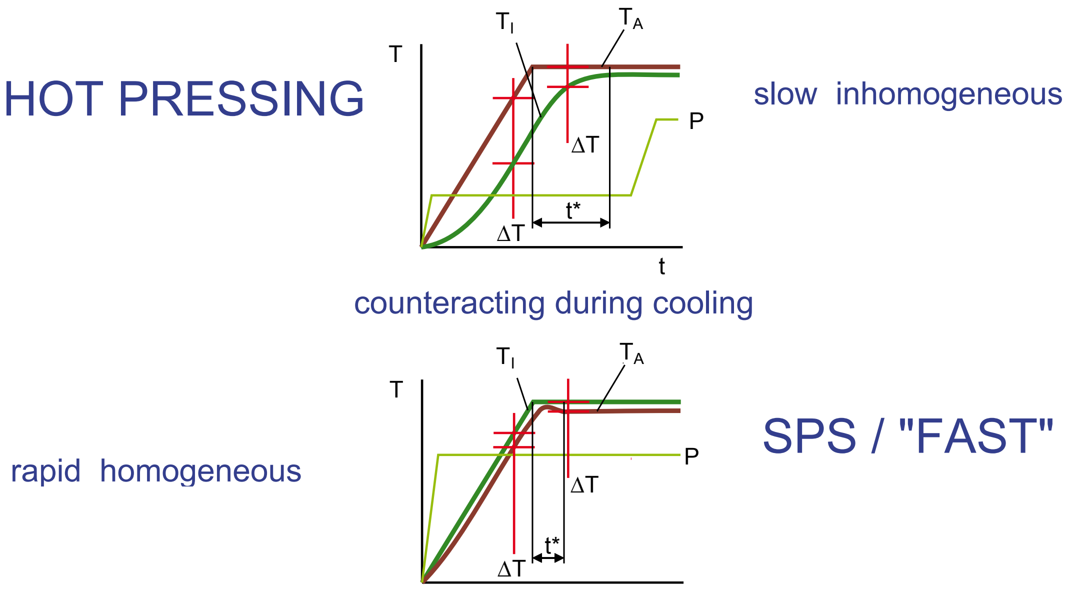


Diagram 11: *Temperature homogeneity: Comparison of hot pressing and FAST*

Diagram 12 shows the effect of the measuring point on the "quality" of the temperature measurement. Significant differences (more than 200 °C) in measured temperature in the outer area of the mould - as is usual in hot pressing technology - result when a relatively small press mould is used at a working temperature of 1500 °C. This is in comparison with measurement directly on the die - as used in the FAST technology. This would naturally affect temperature control during the process, and the interpretation of the process. As can also be seen in **diagram 12**, temperature settings with very small deviations on the mould and the die are possible today, thanks to modern temperature control systems (thermo-element and/or pyrometer).

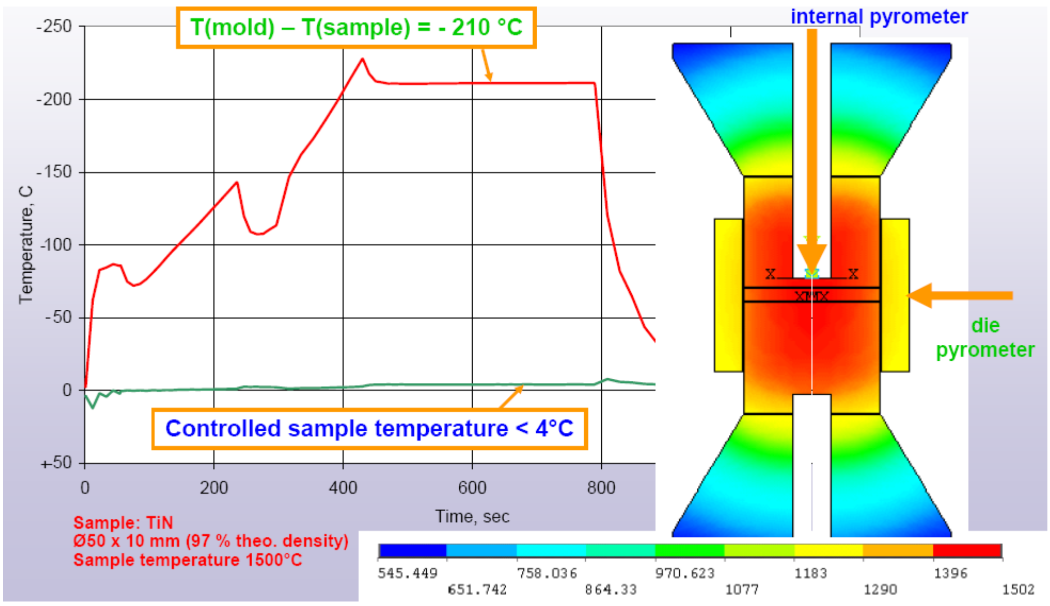


Diagram 12: *Mould and die temperature:* ΔT

Significant potential for the rapid SPS technology exists in the possibility of being able to create very fine, tight joints, and to almost completely maintain the structure of the initial powder in the finished blank. The reason for this is the short cycle times. It is superfluous to mention that for this, exact temperature measurement and therefore sophisticated control technology is essential. This can be demonstrated through the example of a submicron silicon nitride powder (YAG) (**diagram 13**).

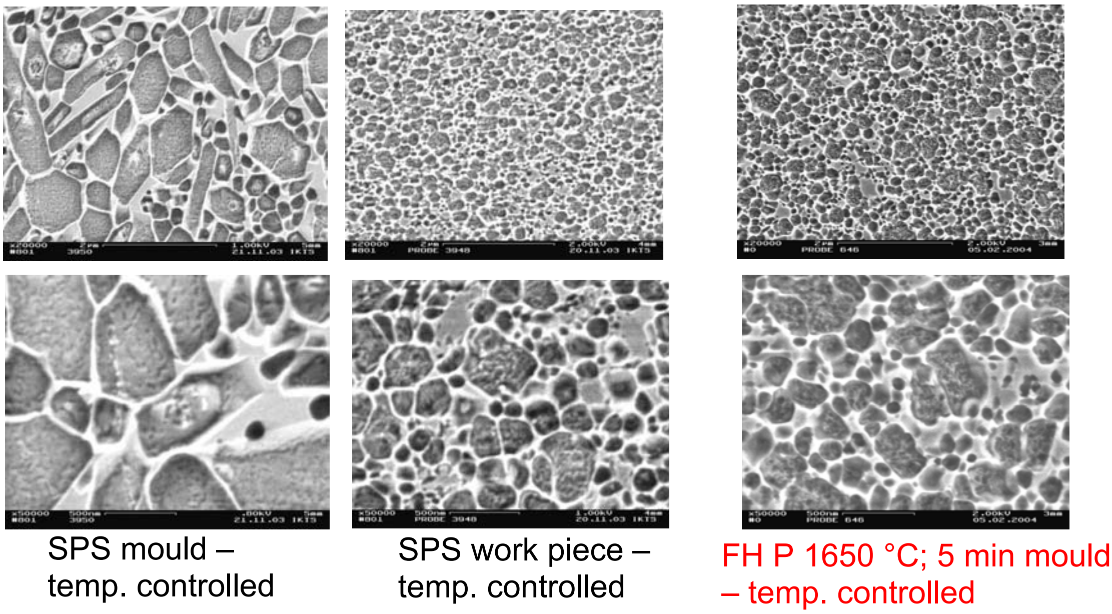


***Diagram 13**:* *Submicron Si_3N_4: SPS sintered (temperature control on the exterior of the mould and directly on the die) and hot pressed (temperature control on the mould)*

To verify the results above, a further trial was carried out in a very rapid hot pressing unit with relatively small format test parts of silicon nitride. As well as the visual examination of the joint, the measurement of the α-/β-phase state of the resulting pressed blank is very conclusive (**diagram 14**). The ratio of α-Si_3N_4 to β-Si_3N_4 allows important conclusions to be drawn about the actual working temperature of the workpiece: Temperature differences of more than 200°C are probable.

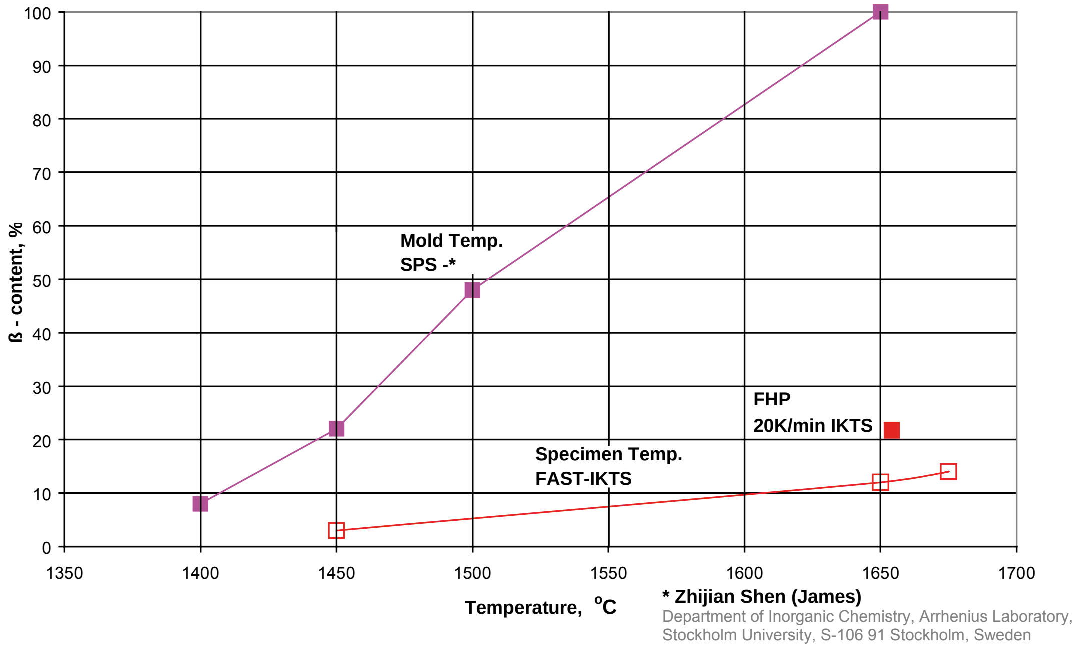


***Diagram** 14:* *Standard silicon nitride (E10): SPS sintered and hot pressed*

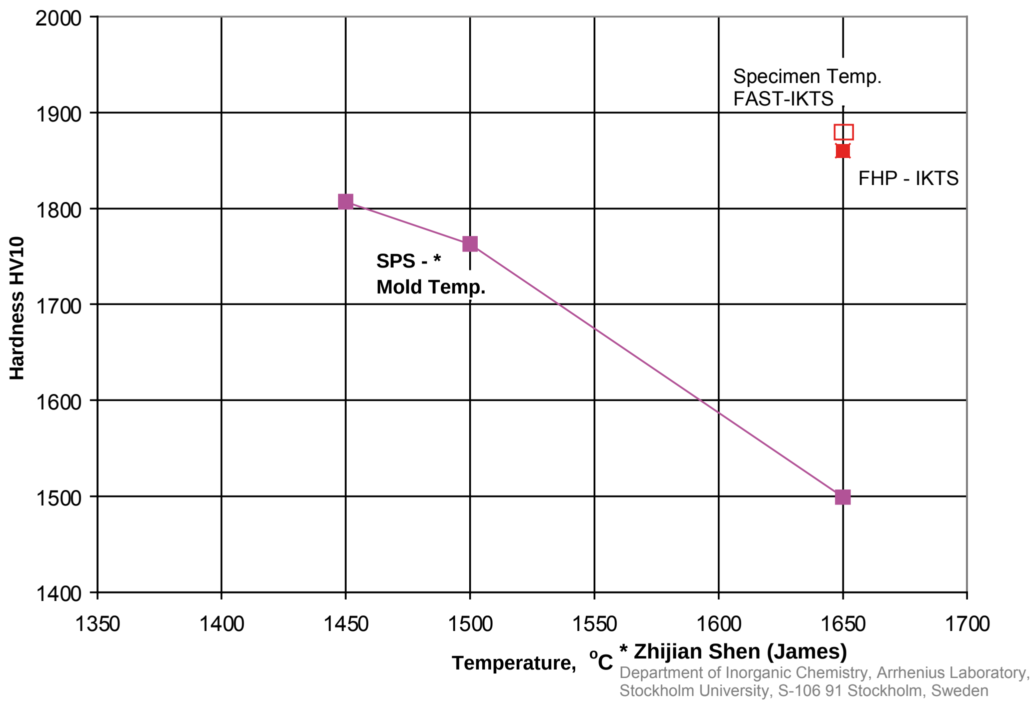


***Diagram 15**:* *Vickers hardness compared with the temperature measurement point*

The hardness measurement of the resulting components supports this theory (**diagram 15**). With the SPS technology, rapid processing using the actual working temperature of the mould also results in significantly better hardness values and this at significantly lower sinter temperatures. Again, the very similar appearances of the SPS and rapid hot press technology are also of importance. For the further development of the FAST concept, the knowledge of the actual temperature of the die is an important prerequisite for the correct interpretation of the trial results, and therefore of their transfer into practice.

Similar trials which confirm the above were carried out at the Fraunhofer IKTS in Dresden. Here, the effect of the temperature difference between die and mould temperature on the material joint and hardness were measured via the frictional wear behaviour (**diagram 16**).

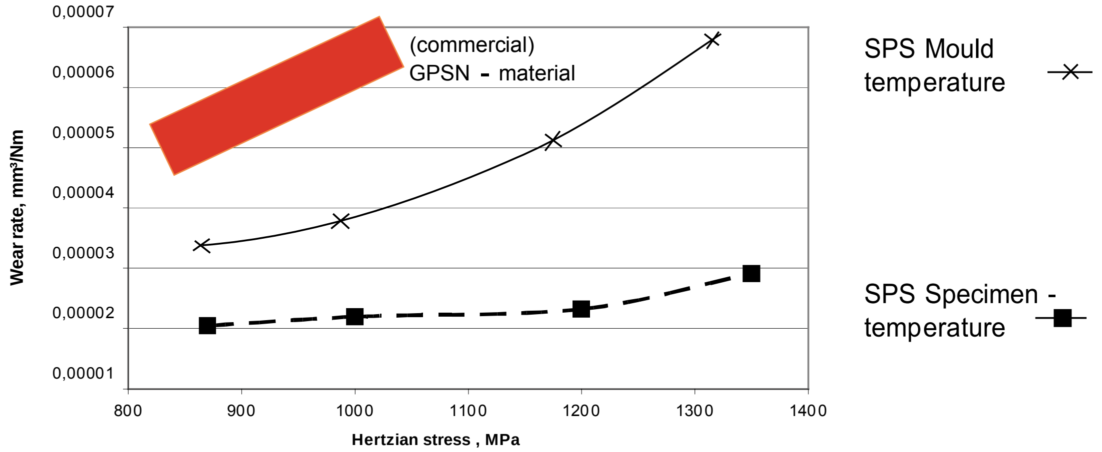


Diagram 16: Frictional wear behaviour of silicon nitride, unlubricated =f($T_{\text{measurement point}}$)

Already here the advantage of the SPS technology is clear with regards to joint creation in comparison with commercial gas pressure sintered materials, and also the resulting potential. All trials were carried out in relatively small moulds with diameters around 50 mm (**diagram 16**). The temperature homogeneity in the blank is discussed below.

5 Mould/die interactions

The temperature distribution which is relatively uncritical for small components - in the die or the mould - becomes a decisive criterion for larger components. Already, early trials with approximately calculated moulds based on graphite uncovered the problem of insufficient temperature homogeneity in the blank. The effect of the temperature on the thermal and electrical properties of the material being consolidated is particularly evident. Great attention was therefore paid to this effect, particularly to the changes in the material during the consolidation process. This was very time-consuming, and required the development of new measurement methods. Starting from the packing density in the pre-consolidated state, these properties changed by several base 10 powers during the consolidation process, caused both by the penetrating temperature and the pressure, and by the resulting density and the state of the joint. Therefore, corresponding basic tests are necessary for "new materials" in order to determine the relevant properties at least roughly, and thus to allow theoretical calculations on the course of the SPS process.

The theoretical work which has become available in the meantime in connection with the new measurement methods available, and the use of Finite Element-Simulation (**diagram 17**) represents an important basis for interpreting the component material/mould material system. The specification of mould geometry and material, especially of the press ram and the base ram are here of decisive importance. Work to date has been based on situations in which the filled mould is placed in the unit before the process and removed from it afterwards. Work currently ongoing is intended to optimise fix-mounted FAST moulds in terms of thermal and electrical properties in such a way that in a recurring process a stable operating state is achieved which results in homogeneous blanks, and at the same time does not overload the FAST unit during continuous operation.

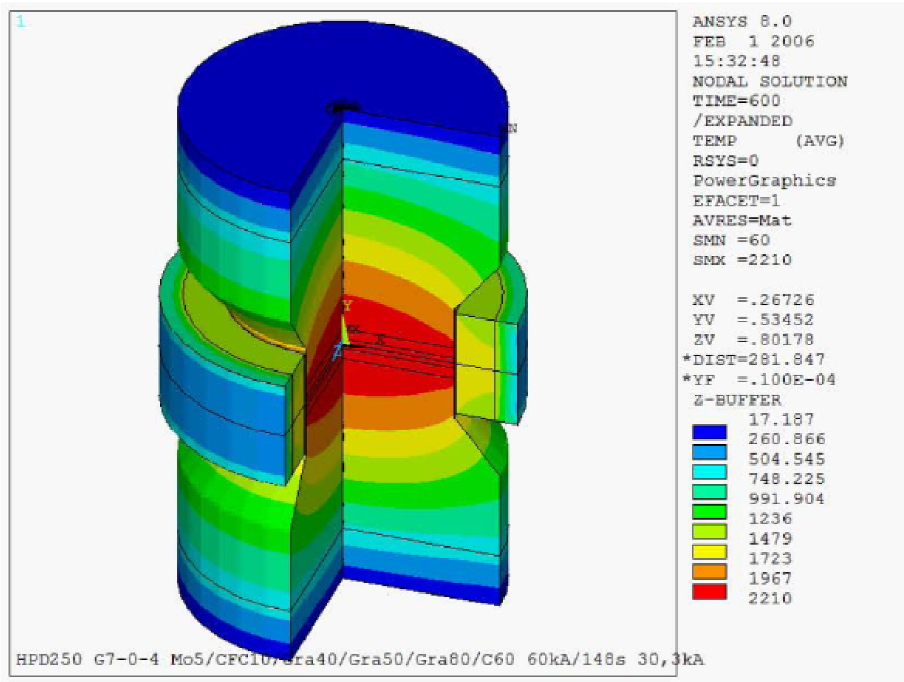


Diagram 17: *FEM simulation of the heating-up phase*

From the wide range of materials which can be consolidated using the FAST process an extraordinarily broad spectrum results in connection with the various potential mould materials. The range of electrical properties of these materials is correspondingly large. This fact is made clearer in **diagram 18** through the display of the electrical conductivity values of a small selection of important materials (at room temperature).

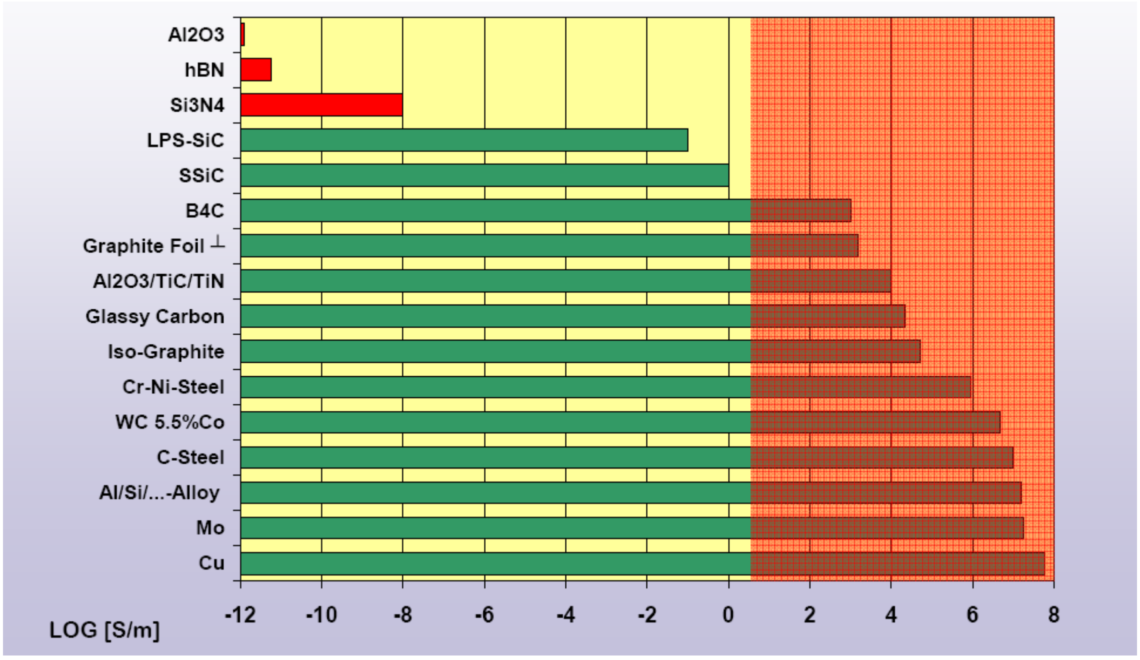


***Diagram 18**:* *Electrical conductivity of various materials (at RT)*

For practical, exact calculations it is naturally not the values at room temperature, but rather those at the temperatures during the consolidation process. **Diagram 19** illustrates the dependence on temperature of the electrical resistance values through the example of a copper-chrome material which is currently being developed for use as a contact material. The values were specially determined during a FAST process in an HPD unit.

It may incidentally be mentioned that the density of the sample material also has an effect on its electrical conductivity.

The use of graphite materials when designing moulds is a logical and in principle correct consequence. However, graphite can be viewed only as the starting point for further developments, because some of its properties, such as its creep behaviour, contact resistance, and above all its wear behaviour, make it advisable to look for more suitable materials which ensure reproducible continuous operation of the FAST process.

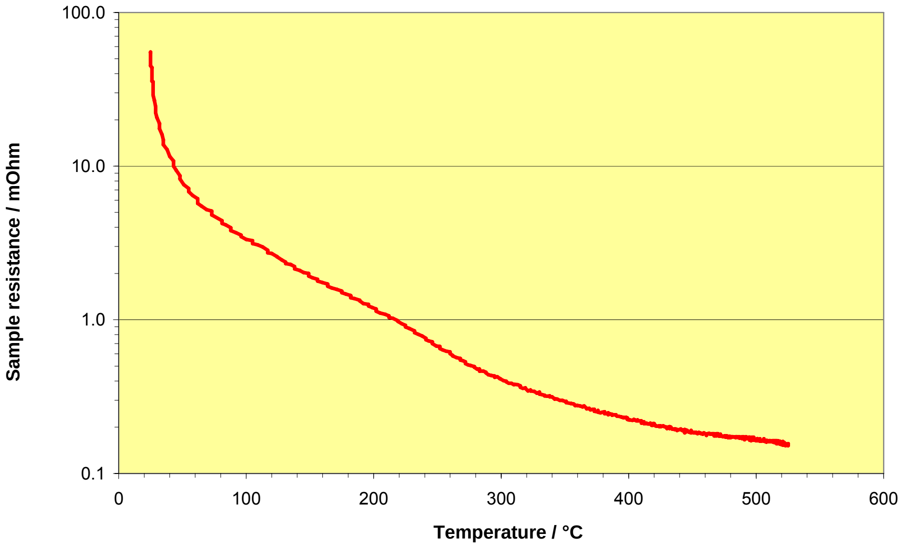


***Diagram 19**: Sample resistance Cu25Cr = f(T)*

The current density within the press ram and the press mould are to a limited extent also a measure of the gradual heating of the mould and of the material to be consolidated (depiction of current flow: **Diagram 20**). Permanently stable operating conditions can be achieved through the use of electrically insulating external press covers. Reproducible process results are achieved in this way, but inevitably there is also a strong effect on the current density distribution within the press ram, and this must be taken into account in the mould calculations. Both variants are used with large scale components i.e. electrically conductive and non-conductive mould covers. At the current state of development, greater chances of success for continuous operation are assigned to the non-conductive mould mantle. It is also necessary to take into account the very high current densities which can cause defective or very non-homogeneous results if the mould is not precisely adapted, e.g. through incorrect or imprecise tolerance values. This can happen both in the low temperature range up to 500 °C (e.g. aluminium), and also in the high temperature range up to 2200 °C (e.g. tungsten carbide).

The heavy reliance of the process results on the electrical properties of the press mould and of the blank makes very precise setting of the process parameters essential. In general, the working range of FAST units in terms of maximum voltage and maximum current is limited by the achievable power values and finally of those values transferable into the die. As a result, the electrical properties of mould and blank have an important effect on the duration of the whole process.

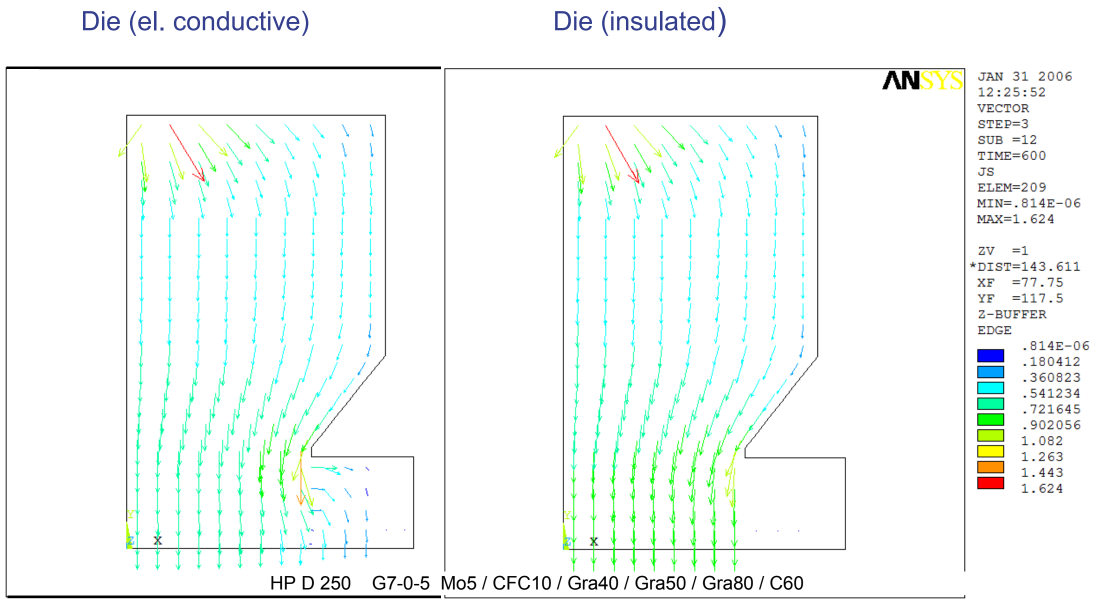


***Diagram 20**: Current flow in the press mould*

The electrical resistance of the press ram and of the material to be consolidated again affect the geometric design and the possibilities of ensuring optimum unit operation. In **diagram 21**, the die resistance is presented in relation to the power of the unit. The area hatched in red represents the limited working area in which ideal process results are achieved. Going above the limit leads to extremely high energy losses through the press ram; falling below the limit, the energy uptake is too low, which leads to unacceptably long process times.

The "fine tuning" of this optimum working area is done through the mould and material properties. In principle, appropriate calculations are carried out before the design of the mould, and if necessary, additional measurements are made to narrow down the known experimental values.

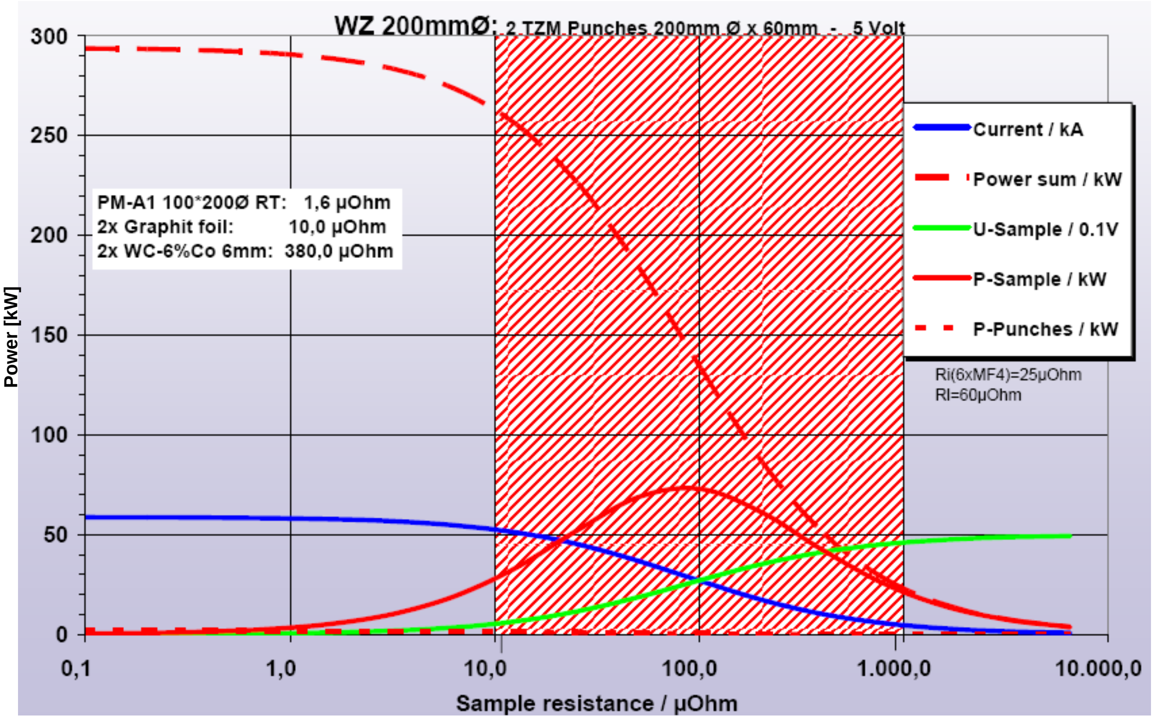


***Diagram 21**:* *Effect of electrical conductivity (example)*

Manufacturing industry increasingly requires components with particular properties, e.g. nanostructured joints, extremely fine granularity or the most complete component homogeneity, at the same time as high density. Extreme component properties naturally present extreme requirements with regard to the operating parameters, the mould design, and the material preparation.

In spite of achieving high absolute density values (>99% of theoretical density), initial trials with blanks (Ø 200 mm) of very fine tungsten carbide (cobalt-free) resulted in pronounced – and therefore not tolerable for the intended use – variations in the density distribution. It was therefore found necessary to carry out further optimisation work in terms of mould design and process technology, with the clear aim of achieving the best possible component homogeneity. This was achieved largely through the layout of the mould. Today, with large moulds (e.g. Ø 300 mm) it is possible to achieve good temperature homogeneity (deviations less than 20 °C) at the same time as low deviations in density (< 0.5%) and hardness (< 10 %) (**diagrams 22, 23**).

It should again be clearly pointed out that every component material requires the creation of its own mould concept. Significant modifications to the mould concept for the SPS/FAST process may be required for even slight deviations in the raw material properties, although these used to be completely acceptable when other manufacturing technologies were used.

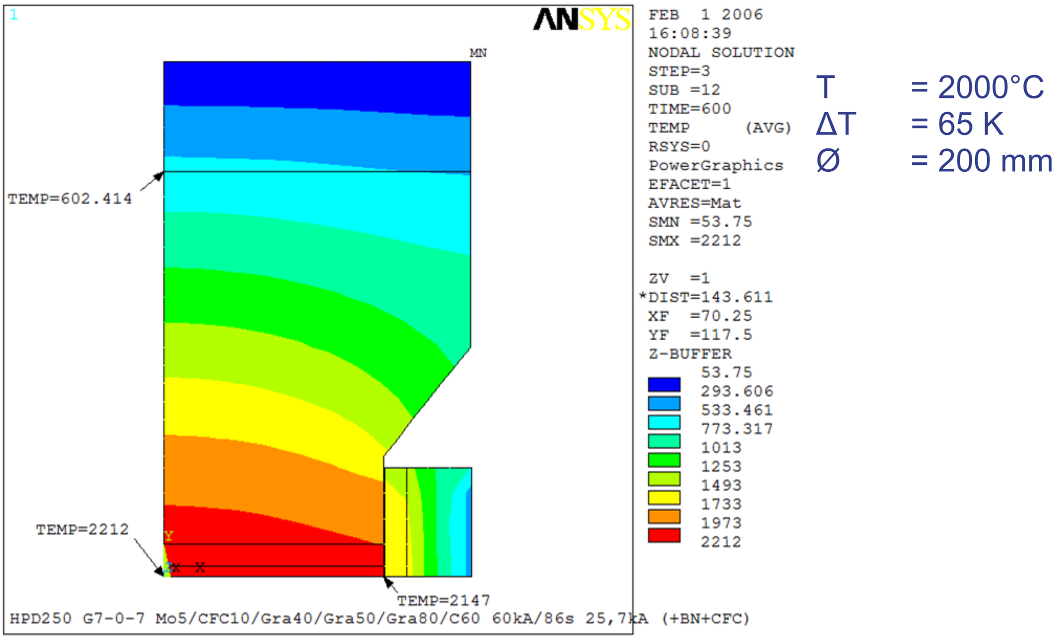


***Diagram** 22:* *Finite Element Simulation (ΔT-minimisation)*

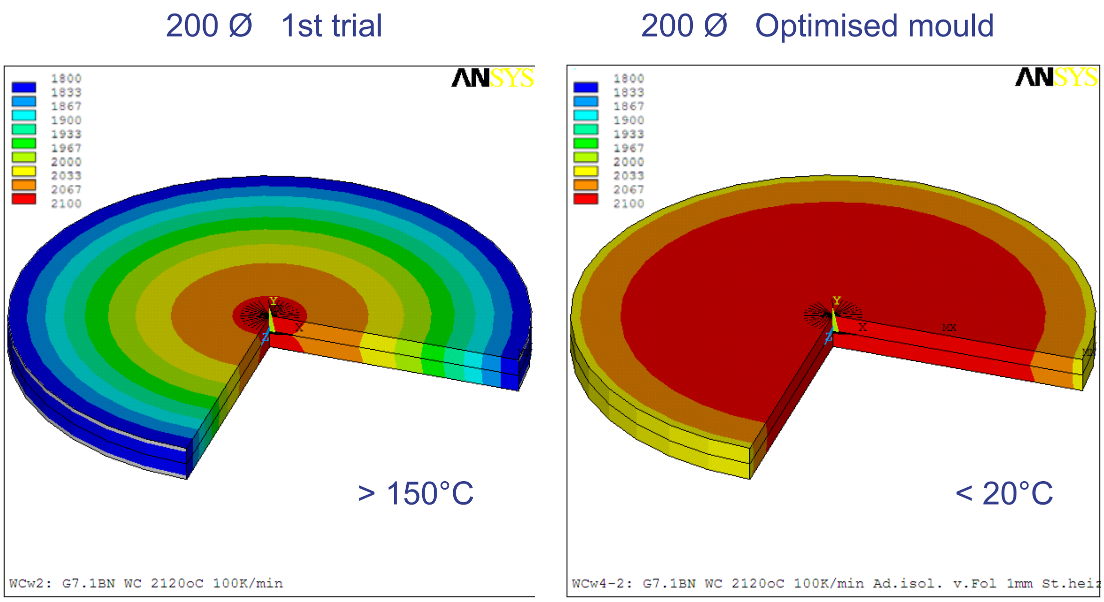


***Diagram** 23:* *Temperature distribution in a mould Ø 200 mm, 2100 °C, 100 K/min*

A large proportion of ceramic materials have a relatively low electrical conductivity, which is further reduced by the porosity in the initial phase of consolidation. The result of this is that the overwhelming proportion of the electrical current does not flow through the material, but through the conductive press mould. **Diagram 24** shows the calculated proportion of the current flowing through the material in comparison with the total current. This calculation has not taken into account the effect of porosity.

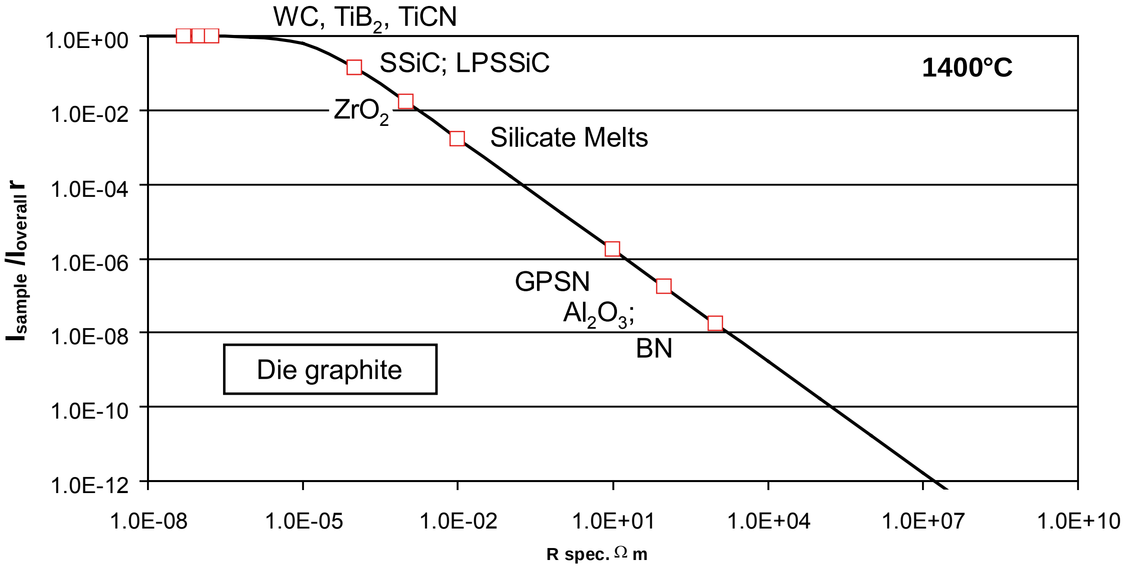
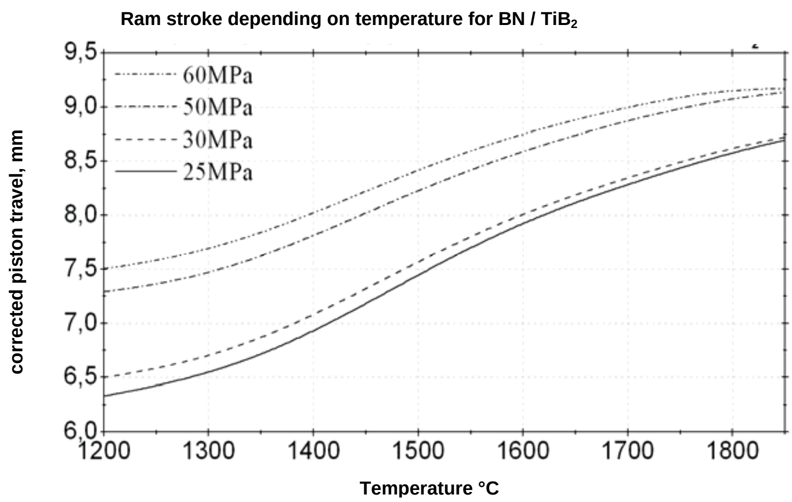


***Diagram 24**:* *Calculated proportions of current through the material in comparison to the total current for various ceramic materials (moulds made from graphite) [**9**]*

With the exception of the hard metals, transition metal carbides, and many borides, direct current flow through the material is small. This has two consequences: for these materials, thermal gradients in the material will occur especially at rapid heating rates and large sample dimensions, and secondly, actual SPS effects can hardly be expected. Our own measurements on Si_3N_4 materials have shown that at least dimensions up to 60 mm diameter and heating rates up to 100 K/min result in homogeneous materials [**10**].

The effects of drastic reductions in the sintering temperature reported in the literature should be critically examined. They are to some extent conditional on the type of temperature measurement. However, the effect of pressure on consolidation (**diagram 25**) (similar to hot pressing) and prevention of the reduction of sintering activity through surface diffusion and granular growth in the heating phase may be mentioned as real effects.



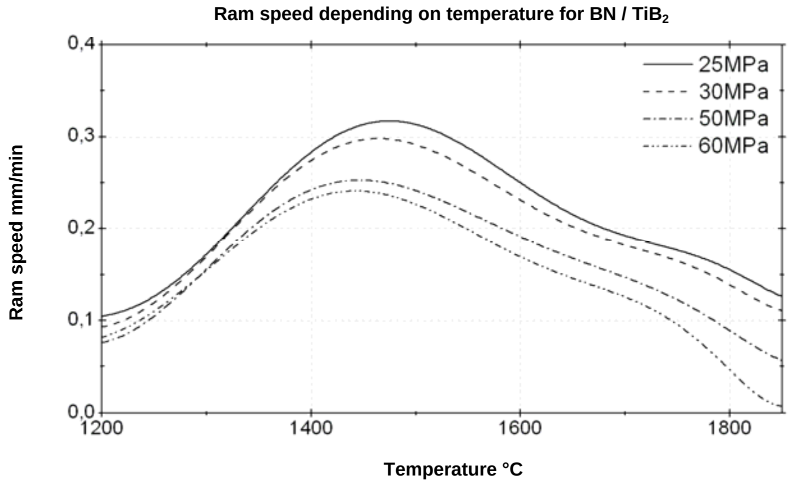


***Diagram 25**:* *Consolidation of BN/TiB_2 composites as a function of the pressure. The maximum consolidation temperature is significantly reduced by pressure*

Regardless of this, we see great potential in the method for multiple applications also for ceramic materials. In particular, these are:

Materials which are currently not hot pressed (e.g. BN, TiB_2/BN composites, SiC whisker reinforced Al_2O_3, B_4C). Thanks to the short cycle times achievable, there are possibilities here for producing "near net shapes", and savings in the finishing work. Initial investigations in IKTS have shown that the homogeneous consolidation of BN/TiB_2 composites is possible at least up to sample diameters of 80mm, and also that non-rotation symmetric components can be consolidated homogeneously.

The short cycle times and the pressure, which can reach 100 MPa or more, allow the consolidation of nanomaterials with minimum granular growth. This enables the relatively economical production of nanomaterials and components. As well as the production of nanomaterials, the method can be used for the manufacture of ceramic materials with a defined structure using sinter forging (superplastic moulding) [**11,12**,**13**].

The short cycle times (a few minutes) permit new material concepts for very varied applications in many areas (e.g. cutting and attrition materials, bio-materials, multi-function materials). Thus, the first work has started in the consolidation of diamonds and cBN/WC/Co composites [**14**,**15**,**16**], which cannot be manufactured using conventional methods. In the area of bio-materials, there have been reports of ZrO_2-HAP composite materials of high strength, for example [**17**]. These components cannot be sintered using the conventional process.

High density functional materials [**18**] and also transparent ceramics can be manufactured effectively using the method.

The method allows the sinterability of components built up in a structured way, e.g. multi-layer systems or even spark plugs for diesel engines.

The rapid heating permitted by the FAST system can moreover result in special microstructures in liquid phase sinter materials. This is conditional upon high over or under saturation of the melt. Further investigations will be required to show whether that is the cause for the rapid phase change and granular growth sometimes mentioned in the literature as occurring when consolidating Si_3N_4 materials above a critical temperature.

The possibilities mentioned will be illustrated here with an example. Nanocrystalline ß- Si_3N_4 materials have wear behaviour reduced by a factor of between 3 and 5 compared with normal materials [**19**]. The production of these materials is based on powders which are about three times more expensive than conventional Si_3N_4 powders. At low temperatures, FAST-consolidated materials have a stably adjustable content of α-Si_3N_4. These materials thus manufactured with process times of a few minutes have similar wear values as nanomaterials, under oscillating fretting (**diagram 26**).

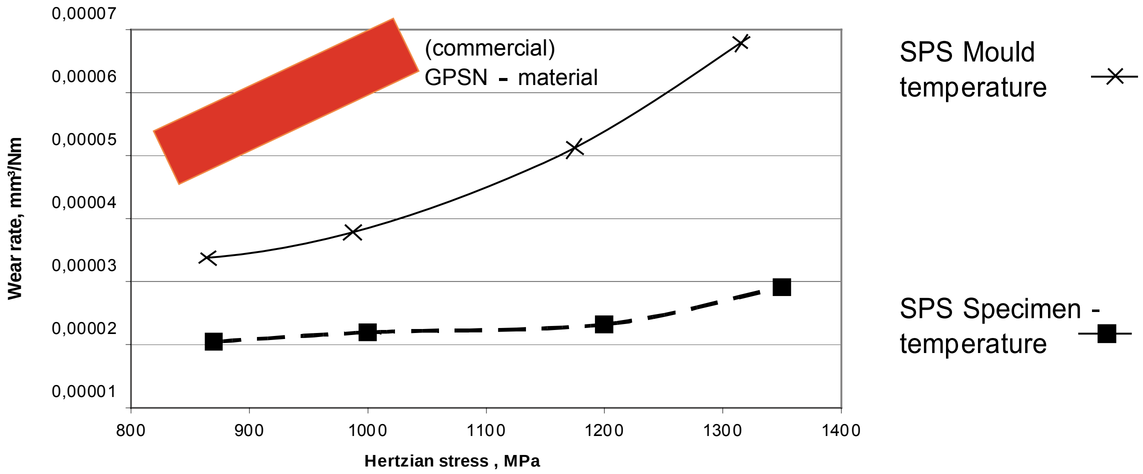


***Diagram 26**:* *Fretting of conventional (a) and nanocrystalline (b) ß-Si_3N_4 materials under unlubricated conditions, and of Si_3N_4 ceramics (high proportion of metastable α-Si_3N_4), which were consolidated using SPS (c-d) [**10**]*

6 Examples of FAST activities

Two important core goals were at the focus of the further development of the FAST unit concept: One was to conceive a reliable technical mould, with the aid of which the desired maximum temperatures could be achieved without system overload. The other was to massively shorten the sintering cycle times, partially down to a few seconds.

The first consolidation trials were therefore carried out in an early phase of development. The consolidation behaviour of composite materials which show a significant sintering behaviour only at temperatures greater than 2000 °C was investigated. Because of their particular properties, composite materials of this type have a wide range of potential applications, e.g. as wear protection, for ballistics use, and for high temperature applications, etc.

We give here the example of a titanium diboride-silicon carbide material, which it was possible to bring almost to its full density in a relatively short cycle of 15 minutes – without a significant second phase (**diagram 27**). In assessing the process mentioned, it was noticed that the ranges with the highest consolidation activity - in other words, consolidation rate - were well below the maximum working temperatures applied; nevertheless, it was necessary to reach these temperatures in order to achieve the desired high density values. Further SPS/FAST tests with different borides and carbides show very similar behaviour. The next step will be to transfer the knowledge gained from the production of small components to larger scale components, using nano-scale powders.

As well as the classical metal and ceramic materials, functional ceramics are becoming increasingly important – for SPS/FAST, still a largely unresearched area in terms of the use of this process technology to achieve specific material properties. Current examples of this are electro-optical, piezoelectric and magnetic materials. Extensive research work in recent years has resulted in a truly vast flood of publications in the area of spark plasma sintering, and this gives some idea of the potential of this technology. In reference to this, mention may be made of the current studies at the Fraunhofer IKTS and at IFAM.

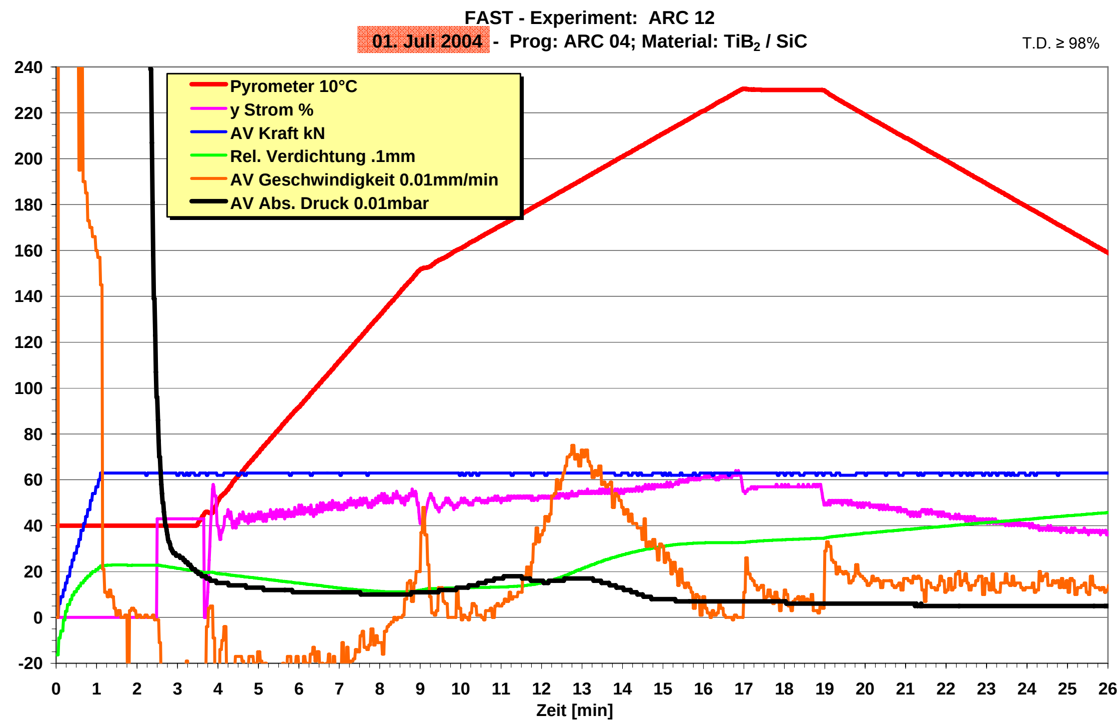


***Diagram** 27: Early FAST experiment at 2300 °C*

Progress in recent years in the area of thermo-electrical functional materials has raised the hope that, with the aid of FAST technology using nano-scale starting materials, a significant step forward has been made in the practical application of this technology.

Nanotechnological approaches have realistic opportunities precisely with **functional materials**, as this is where significantly improved properties can be achieved. For thermo-electric materials which convert DC and AC current into temperature differences, figures of merit were obtained which appeared hardly achievable during the last five decades (**diagram 28**). ($Z = S^2 \cdot \sigma / \lambda$ with S = Seebeck coefficient [μV/K], σ = electrical conductivity [Ωcm], λ = thermal conductivity [W/mK] and T = absolute temperature [K]) can be used as a measure of the quality and efficiency of a thermoelectric material. Materials where ZT > 1 are mostly designated as High-ZT materials. These highly efficient materials are structured as multi-phase composite materials with precipitations on a sub-micrometre or nanometre scale (nano-composites). For the economical production of large components, the often used thin layer technologies are however unsuitable because of the low deposition rates; other methods mostly allow only the production of "academic" test quantities. This limitation is evident when one remembers that standard dimensions for Peltier elements (for heating and cooling) and for thermo-generators (for creation of current e.g. from waste heat) have material thicknesses of 0.5 to 2 mm (**diagram 29**). Although with the aid of molten metallurgy it is possible to produce large quantities of high quality thermo-electric materials, they can scarcely be further processed because of their mechanical properties (very brittle, cracks from high cooling rates, etc). Apart from that, the currently common thermo-electrical materials on the basis of Bi_2Te_3 (mixed crystals) with large "single crystal" domains and stronger texturising should be mentioned. Because of the high anisotropy of the physical properties in the structure, the effectiveness can thus be increased, but on the other hand, the mechanical strength is reduced through the ease of splitting the individual layers of the crystal lattice. Through a protective milling technology and subsequent compaction of the nanostructured powders by means of the SPS process, large semi-finished components can be manufactured which are suitable for further processing into modules (**diagram 30**). Because of the rapid sintering, there is no or at most negligible granular growth, and so the nanostructure established during crystallisation is retained. Additionally, reductions in quality from the loss of texturising are compensated by good granule-to-granule contact (good electrical conductivity because of the surface cleaning of the activating SPS process) and many granule boundaries (poorer heat conductivity).

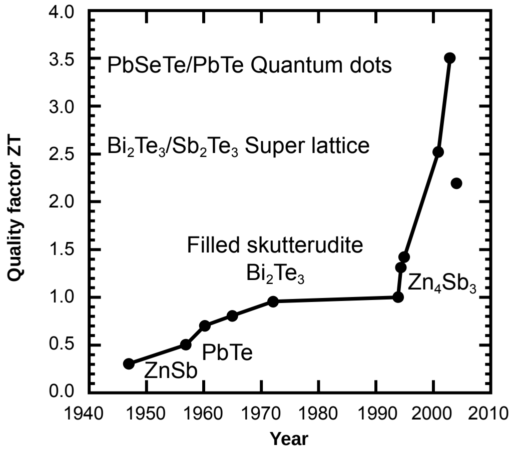


***Diagram 28**:* *From 1995 - effect of new nano-scale materials [20]*

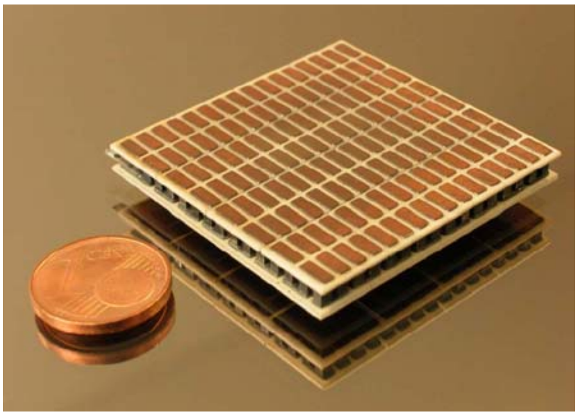


***Diagram 29**:* *Peltier element with 254 thermo-pairs from n- and p-conductive thermo-electrical material on the basis of Bi_2Te_3. [**20**]*

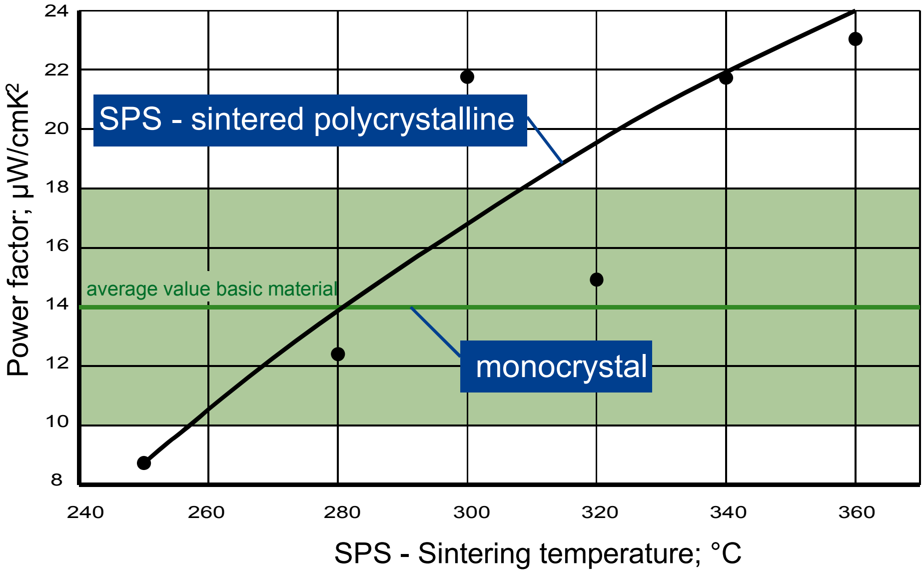


***Diagram 30**:* *Thermo-electric power factor ($S^2 \cdot \sigma$ of SPS sintered, polycrystalline Bi_2Te_3 in comparison with the single crystal starting material [**20**]*

Composite materials on the basis of titanium and titanium alloys are attractive construction materials for lightweight applications. As the titanium alloys have outstanding specific strengths, high ductilities, toughnesses, corrosion and creep resistances, they can be used as the matrix for composite materials with application temperatures of more than 500°C. The best known today are long fibre reinforced materials based on titanium. Particle reinforcements offer the advantages of relatively cheap powder metallurgy production and of isotropic properties. Additionally, after the sintering process, these materials can be more easily further worked, e.g. by forging. In recent years, many results were published in the literature on the use of various reinforcement particles. Examples include the use of TiB, TiC, rare earth metal oxides, or silicon carbide. The incorporation of SiC in the titanium matrix is of great interest, as the low density of the material can be further reduced. However, at high temperatures, SiC reacts with the matrix, and this leads to the formation of brittle intermetallic phases (silicides) and to a significant reduction in the properties of the composite materials. Coatings were successfully developed for the incorporation of SiC fibres, and these impede contact between SiC and Ti during production. Diffusion barriers of this type can barely be reproduced or produced efficiently for particles < 50 µm. Therefore consolidation processes are required which allow pressure together with low temperatures or short sintering times for compacting.

For the production of particle reinforced titanium-based materials, commercially available TiAl6V4- (< 100 µm) and SiC powder (F400) were used. The powder mixtures were prepared in the ratio TiAl6V4/15 vol% SiC in a tubular mixer. These mixtures were charged without further pre-treatment into the graphite press mould ($Ø_{internal}$ = 100 mm and 200 mm, 30 MPa) of the SPS system, and continuously heated in a vacuum at 100°C/min to sintering temperatures between 700°C and 1100°C. After reaching the maximum temperature, the press mould and sintered material were cooled freely in an argon atmosphere.

Detailed investigations by other authors who worked intensively with the formation of reaction layers in the Ti/SiC system for conventional hot pressing indicate that holding times of 30 min and sintering temperatures of more than 850°C are necessary in order to obtain sintered bodies with a residual porosity of < 10 % (**diagram 31**). Only at sintering temperatures > 900°C can reaction zones between SiC particles and the titanium matrix be demonstrated. For short time sintering using the SPS process, at the same sintering temperature (no holding time), the sintered densities are greater than those which can be achieved using conventional hot pressing (holding time 30 min). This may be caused by the sinter-activating influence of the SPS effect on the one hand, and on the other hand by the known differences between the measured and actual temperature of the sintered material.

The maximum sintered density of approx. 95% of the theoretical density is achieved at around 850°C; a further increase is hardly possible. Because of the good electrical conductivity of the metallic titanium powder in the powder mixture, the ohmic resistance is lower than that of the press mould. A large proportion of the pulsed current, which causes the direct heating through Joule's heat, flows through the sintered material. In comparison with the press matrix or press rams, the temperature of the sintered body is therefore higher. This results in a temperature gradient from the middle of the cylindrical sintered body outwards. Its consequence can also be shown, especially in systems which are very temperature sensitive such as Ti/SiC, as a higher density in the middle then in the areas near the surface. This becomes clear when the progression of the density across the radius of the sintered body is determined (**diagram 32**). Based on the investigations which clarify the start of the reaction of the SiC particles with the titanium matrix in relation to sintering temperature and time, it can be shown that during short time sintering under adverse process conditions, temperature differences of 150 °C can arise between the interior and the surface of the sintered body. Through the use of efficient insulating materials (graphite felt), which protect the press mould (especially the matrix) at high temperatures against large losses due to heat radiation, the inhomogeneities can be largely avoided. It was thus possible for the first time to manufacture large Ti6Al4V/15 SiC sintered bodies without residual porosity, even though there is at present a need for further optimisation in the near the surface. [**21**]

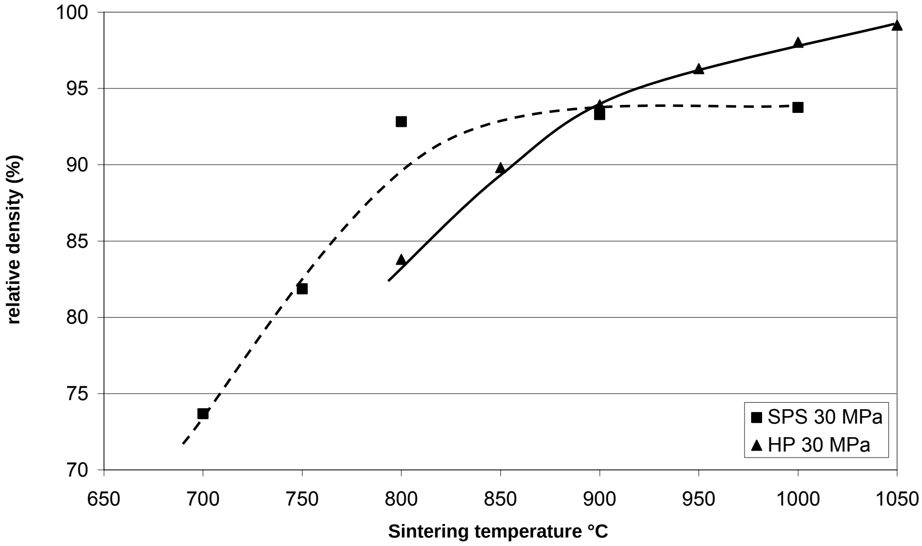


Diagram 31: *Progression of the sintered density (∅ 100 mm) of Ti6Al4V/15 vol.% SiC powder mixtures against temperature. Above 850°C the first reactions can be seen. [**21**]*

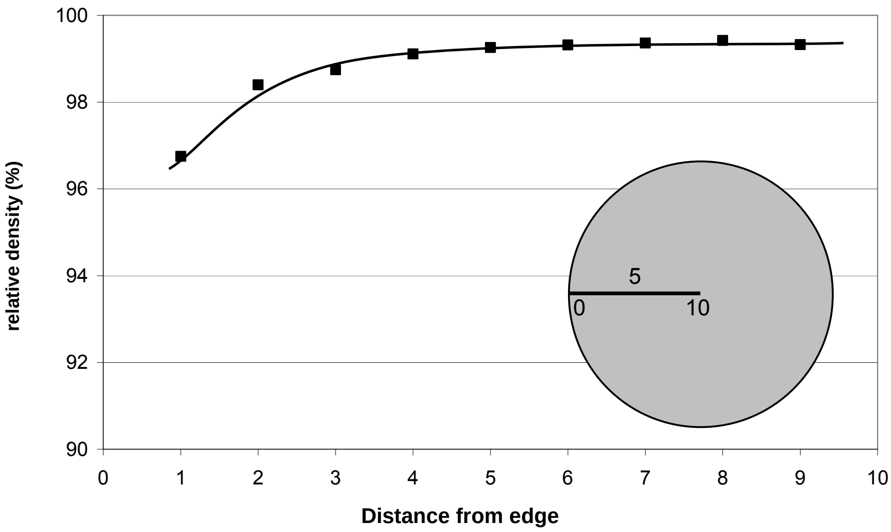
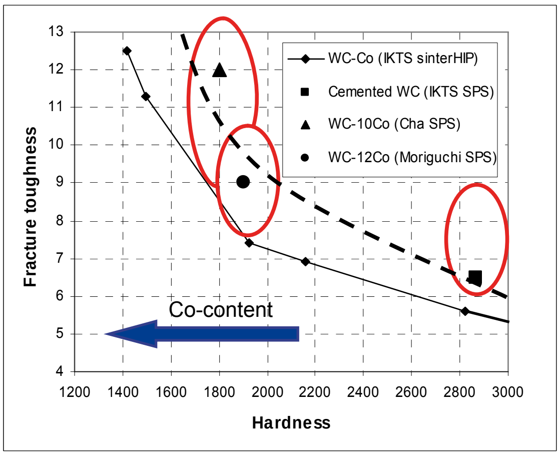


Diagram 32: *Density distribution in a short time sintered Ti6Al4V/15 vol% SiC sample (∅ 200 mm, T_{Sinter}= 900°C) after optimised processing. [**21**]*

A high potential is seen for the FAST technology to affect the hardness/toughness relationship of tungsten carbide-cobalt materials. Various researches have shown that by means of the SPS/FAST technology, higher fracture toughness can be achieved for the same cobalt content (**diagram 33**). [**21**]



EuroPM2004
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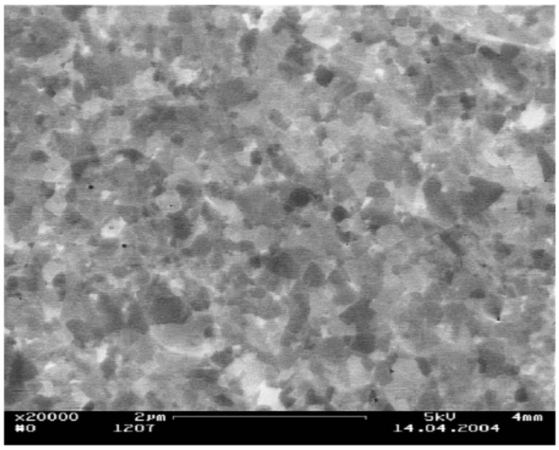

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J. Schmidt, Y.Grin

***Diagram** 33:* *Hardness and fracture toughness of ultra-fine WC-Co [**21**]*

***Diagram** 34:* *Manufacture of binder-free ultra-fine WC (SPS) [**21**]*

Here its is above all interesting that with a decrease in Co-content, the effect of the SPS technology in terms of improved fracture toughness also decreases. At present however, the effects of mould properties (amount of wear!) and cycle time on the process result place limits on this area of application.

Another very promising area of application for SPS technology is the production of sputter targets which are needed as the starting material for the most varied coating processes, such as PVD, laser, or ion beam technology. Targets of metals/metal carbides etc., of ceramic materials, or of compositions which cannot be made by melting technology are usually manufactured by powder metallurgy, through the pressing of powders or powder mixtures and subsequent sintering, or by hot pressing. The advantage of the FAST process is shown in the outstanding homogeneity of the targets created with very much shorter process times.

Diagram 35 below shows the process flow for a FAST consolidation of a Co-free tungsten carbide material for sputter targets with a diameter of 200 mm. Under an optimised process flow, a final density >99% of the theoretical final density was achieved with an active sintering time of 40 minutes.

There is potential for further process optimisation. It is thus possible to sinter two blanks in one cycle if the mould is optimally designed. By further development of equipment technology (higher power), further shortening of the cycle time is possible.

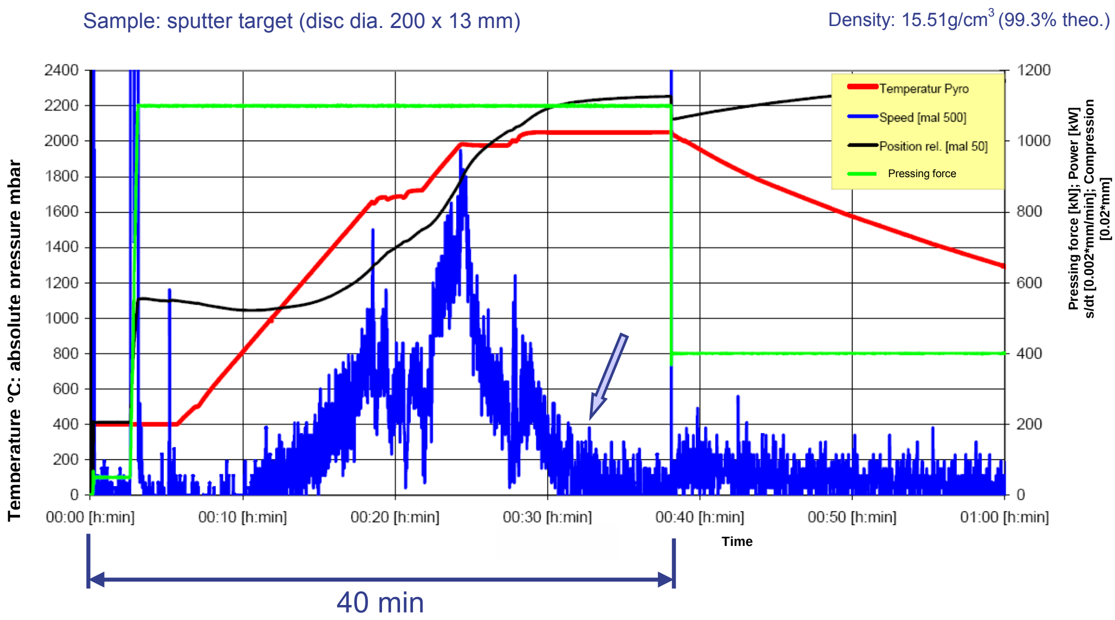

***Diagram 35**:* *Material: WC (Co-free), dimensions: Ø 200 x 13 mm, SPS sintered in the HPD 250*

The efficiency of the FAST technology is currently demonstrated in the successful transfer of the knowledge gained to date (component diameter 80 m) to high volume components, for example made from aluminium alloys which can be manufactured only via powder metallurgical processes - as starting blanks for extrusion or forge form technology.

It was thus possible to achieve almost complete consolidation of the blanks with an exceptionally short cycle time (**diagram 36**). [**22, 23**, **24**]

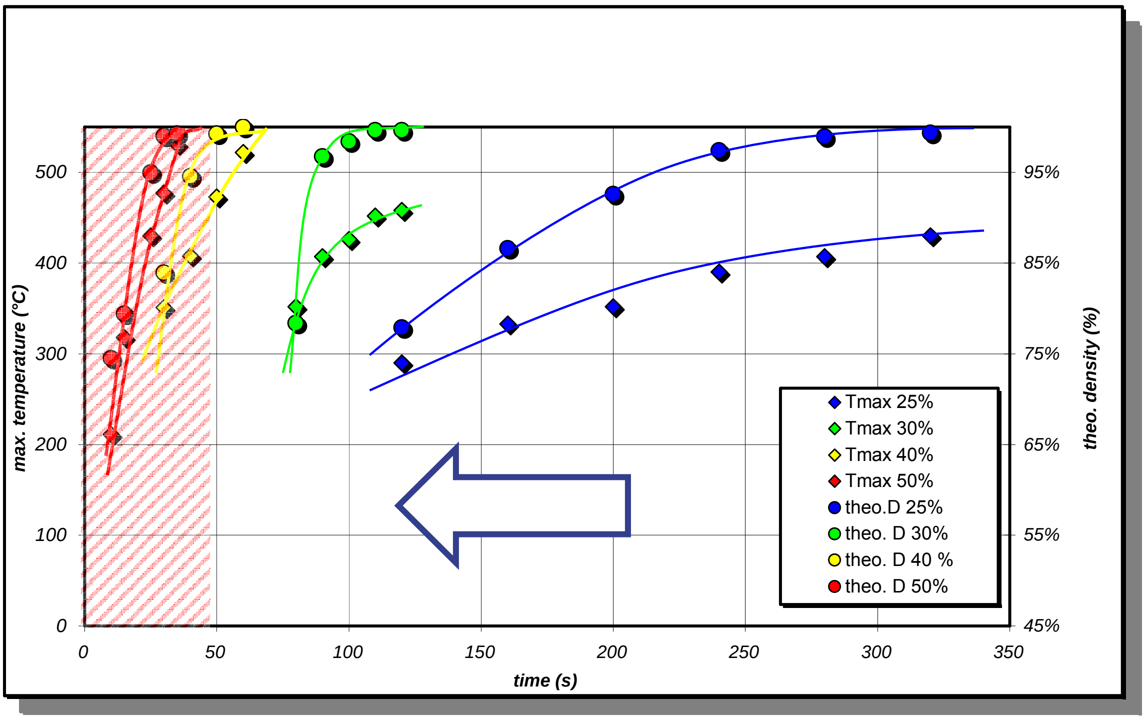

***Diagram** 36:* *Temperature and theoretical density as a function of the time and the heating current [**25**]*

The images of joints in **diagram 37** illustrate that materials of this type can also be manufactured almost without pores, using very short cycles – without exceeding the material's melting point.

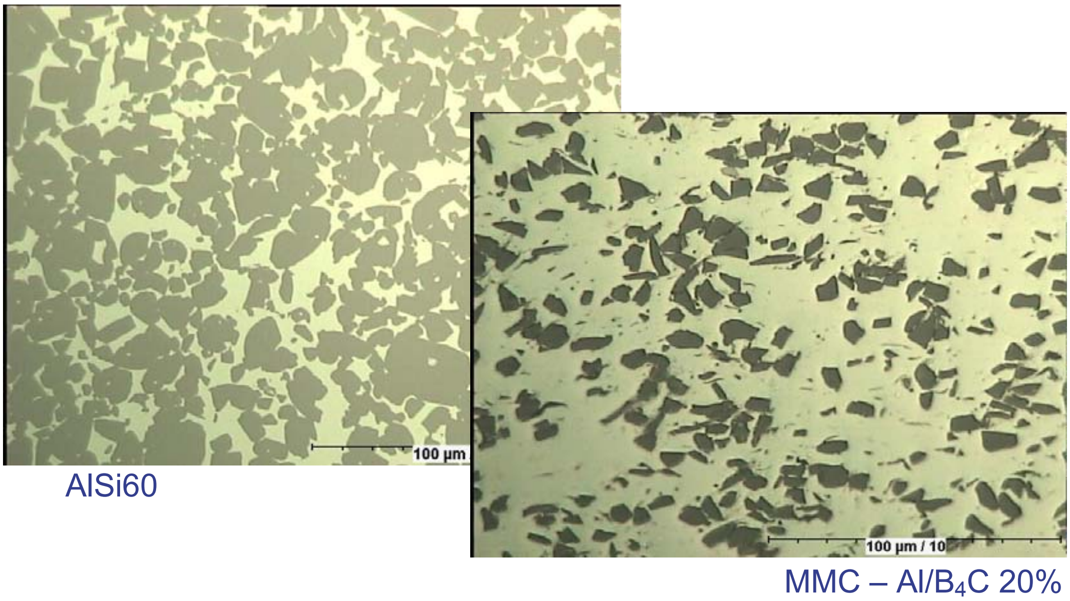

***Diagram** 37:* *FAST-consolidated aluminium materials [**25**]*

As already mentioned many times, the most important argument in favour of SPS/FAST is the short cycle time while still achieving the optimum material properties. The main effective criterion increasingly shows itself to be the energy flow into the blank. For equipment technology, this means that above all the energy supply must become even more flexible and powerful.

The current status of the production of forged blanks made of aluminium-silicon alloys (density > 99%) is a cycle time below 90 seconds, using a special technology for "energy distribution" (energy flow into the blank) while precisely measuring the energy supplied (**diagram 38**).

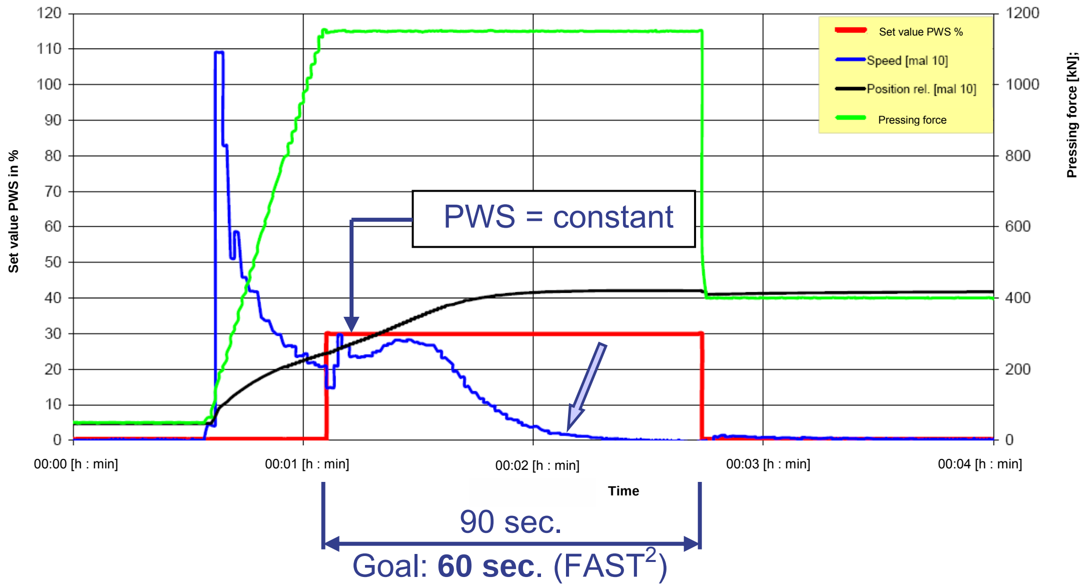

***Diagram** 38: Material: Al/Si alloy, dimensions: Ø 82 x 25 mm, SPS sintered in the HPD 250*

The most recent research project at the FCT Systeme company in the area of spark plasma sintering is called "FAST²", with the goal of even achieving shorter cycle times (< 10 s), with an eye to dry press technology (TPA-FAST). Here too, the material properties achieved, which are greatly superior to cast components, are an incentive to transferring the laboratory results to practice.

A titanium carbonitride/aluminium oxide may be taken as an example for the production of combination or mixed crystal materials; this is today established with cut ceramics for working hardened steel, for example. Component densities > 99% were successfully achieved by means of suitable material and process optimisation, with an active cycle time less than 3 minutes. Continuing investigations into these very promising achievements are running, especially with regard to the use of even finer initial powders (**diagram 39**).

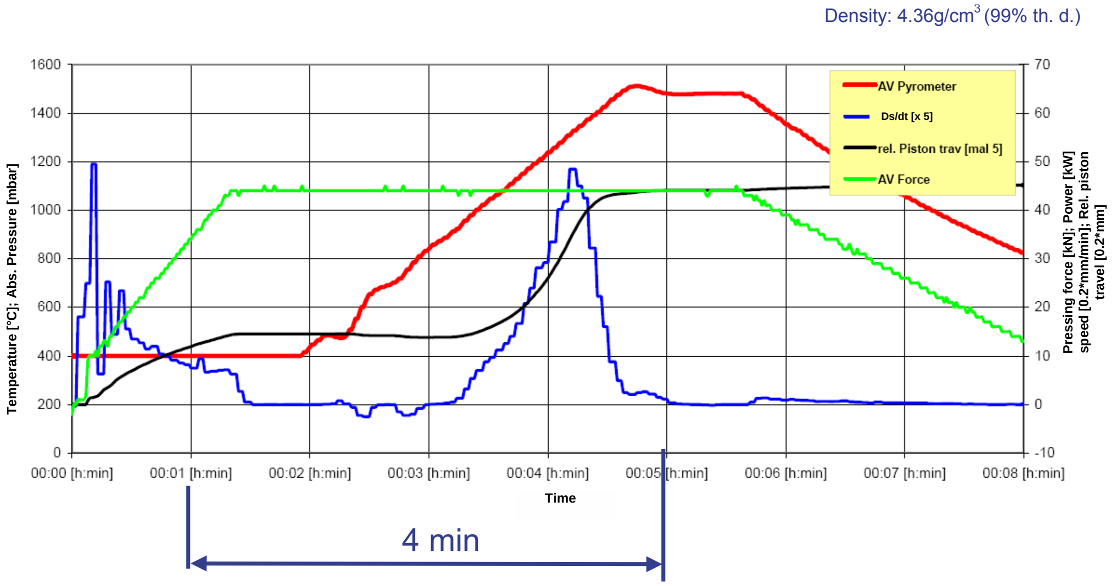

***Diagram** 39:* *Material: Al_2O_3 (submicron) / TiC/N, dimensions: Ø 40 x 8 mm, SPS sintered in the HPD 250*

There is a wide range of opportunities for further trials on the consolidation of metallic and non-metallic materials by means of FAST technology. [**26**,**27**,**28**,**29**,**30**,**31**] However, detailed descriptions of the individual possibilities would exceed acceptable limits.

7 Prospects

The results achieved so far in connection with the FAST technology have created great interest in continuing this work, both in terms of basic research relating to new materials and material properties, and also concerning the practical application of the research results.

***Diagram** 40: The "European Integrated Project on Nanotechnologies and Nanosciences"*

Applications of the FAST/SPS technology occur mainly in the areas of electro-technology, machinery construction, the automotive industry, and medical technology.

At the international level, an extensive long-term R&D project with the title "Nanoker" (**diagram 40**) is currently running, in which FAST technology plays an important role, especially in terms of the consolidation of nano-scale materials.

German R&D projects which are based on SPS technology for the production of nano-materials with special properties are listed in **diagram 41**. The points of emphasis are aluminium materials, composites and combination materials, including graduated materials.

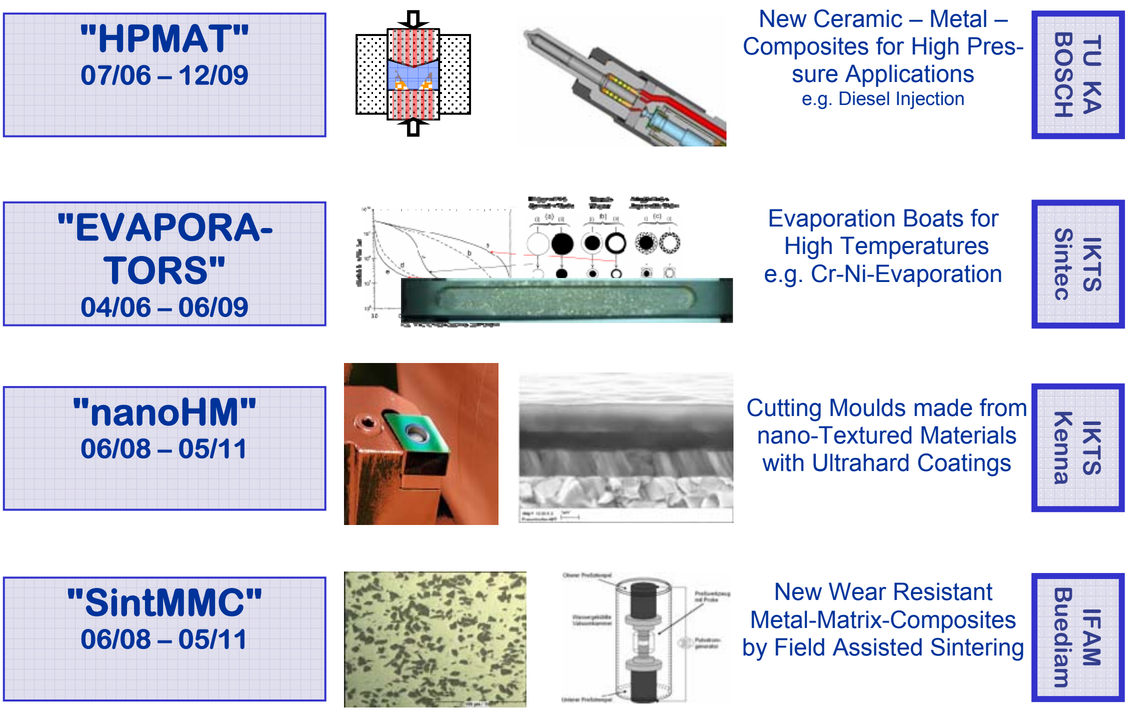

***Diagram 41**:* *German R&D projects with FCT participation*

Increasingly, industrially supported research projects are being run with clear goals as to the intended materials and components.

In addition to the ongoingly important foundation work on material development, the realisation of more complex component geometries is in the foreground of future considerations. Up to now, only simple geometries (discs, rings, cylinders, etc) have been realised (**diagram 42**). The first work on the production of objects with corners (right-angled, square) is under way. The major task of implementing more complex components ("near net shape") still lies ahead.

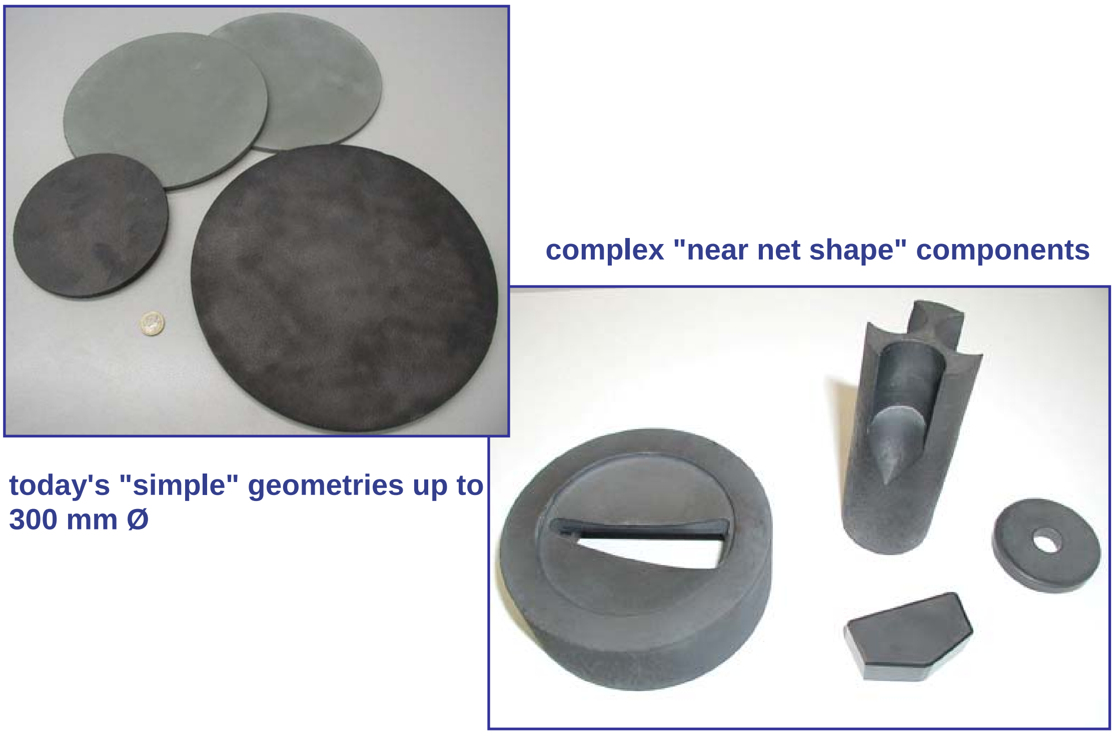

***Diagram** 42:* *Prospects under geometric aspects*

As industrial users of FAST units must above all be cost-oriented in their work, the shorter cycle times are deemed to be of great importance, in addition to the improved material properties. Because suitable performance values per time unit are required for the mass production and use of the corresponding components, the FAST technology is currently being transferred to the TPA base technology. On the one hand, this is the most difficult part of the development work up to now, but it is also the most forward-looking (**diagram 43**). The first successes have already been recorded, but further intensive development work will be required in the coming years. Such a system, through which a more rapid transfer from low temperature levels (500 °C) to temperatures over 2200 °C should be achieved, is under construction. The main characteristics are the reproducibility of the processes, mould wear (lifetime) and machining allowances.

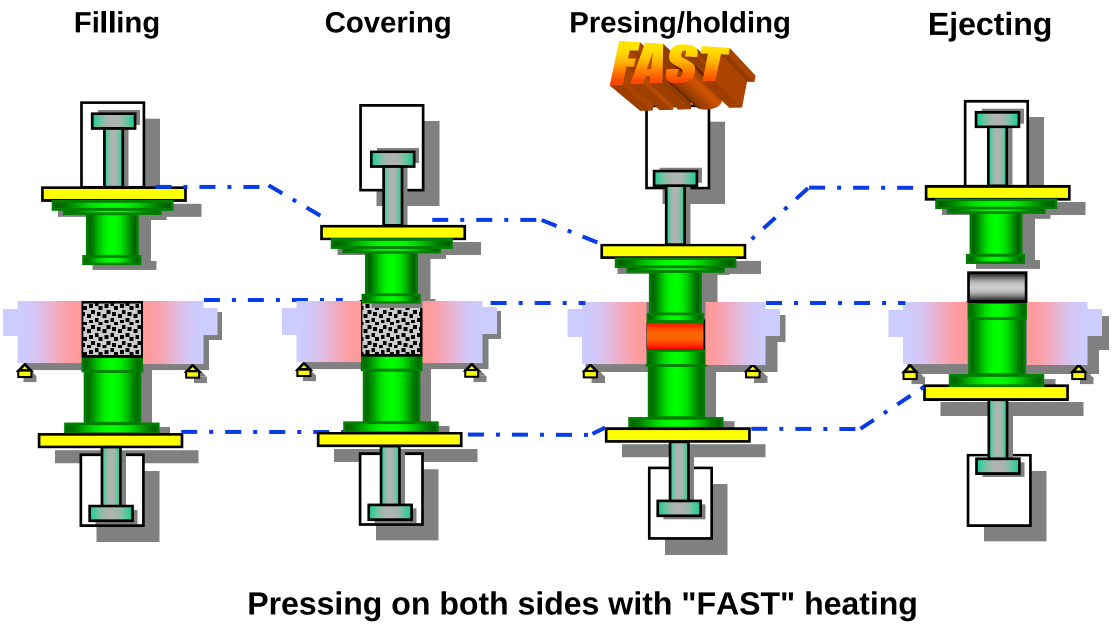

***Diagram** 43:* *Implementation of FAST in the TPA technology*

Here as well, mould optimisation is the focus of interest because of the required mechanical and electrical properties of the press mould, especially with regard to the intended high temperatures.

In the end, all considerations of this process must take into account that regardless of the possibilities already recognisable, development successes can be expected only if the production costs can be reduced and/or the material properties can be significantly improved.

There is great anticipation of the future practical application possibilities of this very promising process.

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