

Hybrid Sintering – a New Trend for Innovative Material Solutions

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Hybrid Sintering is a new trend in sintering technology, combining different sintering methods, e.g. direct and indirect heating, uniaxial and gas pressure, high and low electrical fields, optionally completed by debinding, decoupled pre-heating, and cooling as well as rate-controlled sintering. Hybrid systems facilitate the development and production of all-new material solutions. Trendsetter FAST/Hybrid for example, complements the conventional hot pressing with an additional pulsed current direct heating of the powder compact (FAST/SPS). Compared to hot pressing, this combination allows a further improvement of heating rates and thermal homogeneity, superior to the characteristic advantages of the FAST/SPS technique. Several examples of the industrial application of FAST/Hybrid illustrate that hybrid systems provide new opportunities for the development and optimization of innovative materials, which are currently the subject of intensive research worldwide, in the fields of e.g. power engineering, electric mobility or aerospace as well as other future-oriented fields.

1 What is Hybrid Sintering?

The common definition of Hybrid can be found at Wikipedia, which states: "Hybrid in the technological context means a system, combining two different technologies. As a characteristic, each of these technologies presents a solution on its own, but the combination creates new, beneficial properties" (analogous translation of the German entry).

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FCT Sintering Technology	ACTIVE SINTERING PRINCIPLES			
	 HEAT	 GAS PRESSURE	 MECH. PRESSURE	 ELECTR. FIELD
PRESSURELESS				
GAS PRESSURE (SINTER) - HIP				
HOT PRESSING				
FAST/SPS				
Hybrid Sintering				
FAST/Hybrid (FAST/Flash)				

Fig. 1 Combination of active sintering principles forming different classical and hybrid sintering technologies

In the context of sintering technology, Hybrid Sintering can be defined by the active principle of sintering, as shown in Fig. 1. The most important active sintering principles are heat, gas pressure, mechanical pressure as well as electrical fields, forming different sintering technologies depending on their individual combinations.

The simplest sintering technology, pressureless sintering, simply uses heat to get the parts sintered. Gas Pressure Sintering and Sinter-HIP use gas pressure as a second

active principle additionally and simultaneously. Strictly speaking, this is yet Hybrid Sintering according to the common definition of Hybrid, because it combines two different technologies creating new beneficial properties.

The same is true actually for hot pressing (combining heat and mechanical pressure) and FAST/SPS [1], combining mechanical pressure and an electrical field (which in turn produces heat – to be precise – as a secondary effect).



Fig. 2 Hybrid sintering plant providing max. 125 kN uniaxial pressing force and 2200 °C working temperature at a gas pressure of up to 10 bar

But for manufacturers and users of furnaces, heat is a natural and obvious part of the system. For that reason, we are talking about Hybrid Sintering only if we have two active principles in addition to an actively heated furnace as is the case for the hybrid sintering systems presented in the next section.

2 Hybrid Sintering – a new trend

Hybrid Sintering plants – as defined above – are the latest trend emerging in sintering technology, initially in the area of laboratory applications as usual, before the sophisticated lab technology is exploited with the transfer to industrial, profitable applications.

One interesting example of a new hybrid system is the combination of hot pressing and flash sintering, providing a much higher electrical field compared to FAST/SPS. This new sintering technique allows the development of ultrafast sintering of materials with low electrical conductivity, e.g. oxides,



Fig. 3 Hybrid sintering plant providing max. 100 bar gas pressure and 2200 °C working temperature at a uniaxial pressing force of up to 125 kN

SiC, and others [2–5], opening the horizon for the production of technical parts as well as consumer goods made of oxide ceramics, e.g. tableware, floor and wall tiles and the like, with revolutionary low energy consumption.

Another example of Hybrid Sintering is the combination of Hot Pressing and Gas Pressurizing, which can be interesting for material development and prototype production of materials. This can be realized by combining a hot press with a gas pressure capability of e.g. max. 10 bar as shown in Fig. 2. Such systems are helpful for the consolidation of materials that tend to decompose at the sintering temperature, e.g. silicon nitride and special types of silicon carbide. Other applications are the sintering of high-purity materials or the synthesis of semiconducting compounds (e.g. for the production of high-efficiency LED lighting).

If there is still demand for higher gas pressure, a classical gas-pressure sintering furnace can be combined with a uniaxial hy-

draulic press, allowing pressures of 100 bar or more. Fig. 3 shows a picture of this type of hybrid sintering system, providing an additional 125 kN hydraulic pressing capability as well as a maximum working temperature of 2200 °C.

Just like the conventional sintering technology from FCT for combined debinding/sintering processes and for reactive-bonding processes, these plants can be equipped with the corresponding retorts with defined gas flow as well as the required specific exhaust gas treatment (thermal post combustion, flaring apparatus, acid scrubber, etc). In addition, it is possible to visualize and record the sintering shrinkage of the product in real time. This is standard in plants with mechanical pressing force, and is achieved by precisely measuring the press plunger movement. In other plants, an optional dilatometer unit makes this possible. And optionally the systems can provide rate-controlled sintering based on force or heating rate, allowing the development of optimized sintering processes [6].

3 FAST/Hybrid – the trendsetter

A particularly important hybrid sintering technology is FAST/Hybrid, which has already passed the laboratory phase and arrived at first industrial applications [7] (see section 4). Hence, it is certainly not overstated to refer to FAST/Hybrid as a trendsetter for hybrid sintering.

Fig. 4 shows the principle and thermal gradients of FAST/Hybrid: with a combination of classical hot pressing using an external resistance or induction heater and the very homogeneous internal Joule heating of FAST/SPS it becomes possible to further enhance the heating rates with even better temperature homogeneity, superior to classical FAST/SPS with its immanent advantageous characteristics. This is realized by an independent control of both heaters, each with its own temperature sensor, making a compensation of radial thermal losses possible.

FAST/Hybrid systems have been very successful in the lab in the recent years. Consequently, the transfer to industrial, profitable applications is already underway, and nowadays a complete portfolio of FAST/Hybrid systems is available, from lab scale with a max. force of 25 kN, suitable for samples up to 30 mm size, to industrial scale systems

with up to 4000 kN pressing force, allowing part sizes of 400–450 mm.

4 Industrial application areas of FAST/Hybrid

Currently, several pilot productions using medium- and large-scale hybrid sintering are operating in industry, mainly FAST/Hybrid systems. The largest system so far is working in a company in northern Spain, focused on the manufacturing of high-end technical ceramic components based on innovative materials. This 4000 kN FAST/Hybrid system provides a heating power of 400 kW for internal Joule heating, completed by another 400 kW induction heating power.

Fig. 5 shows a graphic representation of this 10-m-high system on the left and a picture of the loading area on the right. With this system even pure tungsten carbide, without any additions like metallic binder, can be sintered in a short time to a high-quality, homogeneous and fully-dense part with 400 mm diameter, although temperatures of about 2100 °C are required for this material.

The industrial use of this system is focused in three different product/material types:

- Planar sputtering targets for thin film deposition;
- Ultra-High-Temperature Ceramic Matrix Composites (UHTCMCs) for extreme environment applications, mainly aerospace;
- Cermets for cutting tools and progressive stamping (punch and dies).

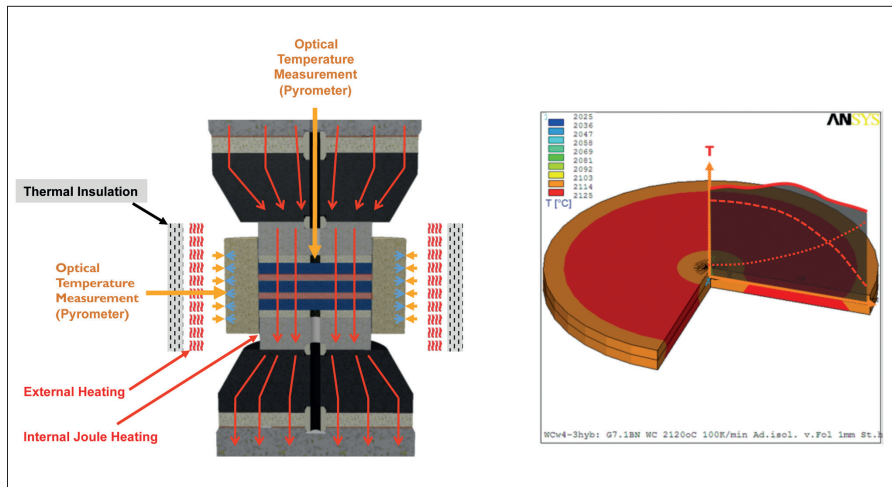


Fig. 4 Principle and thermal gradients of FAST/Hybrid, the combination of classical hot pressing using an external heater, and internal Joule heating of FAST/SPS

Sputtering targets are basically planar products (also rotatable) that are used for the deposition of thin films over substrates for different application purposes. When the end-user of the sputtering target (thin film coaters) is working on an industrial coating chamber, the stability of the target during the coating process is critical and the formation and growth of nodules in the target should be avoided.

These nodules tend to form on the surface of the sputtering target during the process, near the racetrack region, and can change the sputtering rate leading to the formation of defects in the sputtered film. The target grain structure has been identified as one parameter determining their formation. Hence, a sputtering target should ideally

have a very fine and homogeneous microstructure, a high density and a very-high chemical purity.

FAST/SPS as well as FAST/Hybrid are promising technologies for the efficient production of sputtering targets with high electrical conductivity as well as homogeneous and fine grain size, due to fast heating rate, low dwell temperature and thermal gradients immanent to this special sintering technology. This made the development of highest-quality sputtering targets for TCO (Transparent Conductive Oxides) made of AZO (Aluminium-Doped Zinc Oxide) and ITO (Indium-Doped Tin Oxide) possible. TCO's thin films are used in photovoltaic cells, transparent electrodes in LED's and in numerous displays and touch screens.

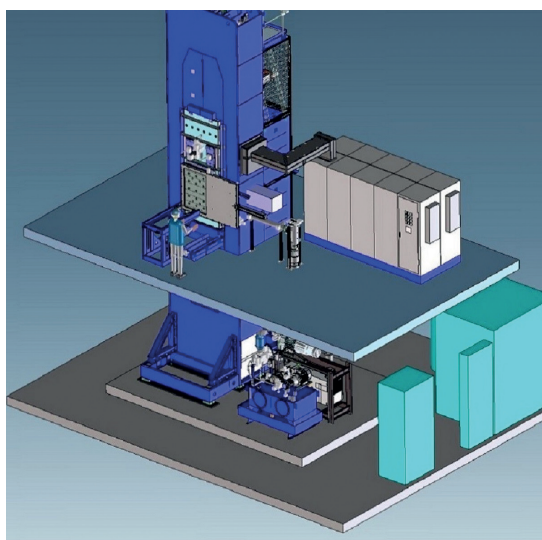


Fig. 5 Large FAST/Hybrid system: graphic representation (l.), and picture of the loading area (r.)



Fig. 6 3-inch sputtering targets made of AZO and ITO, respectively

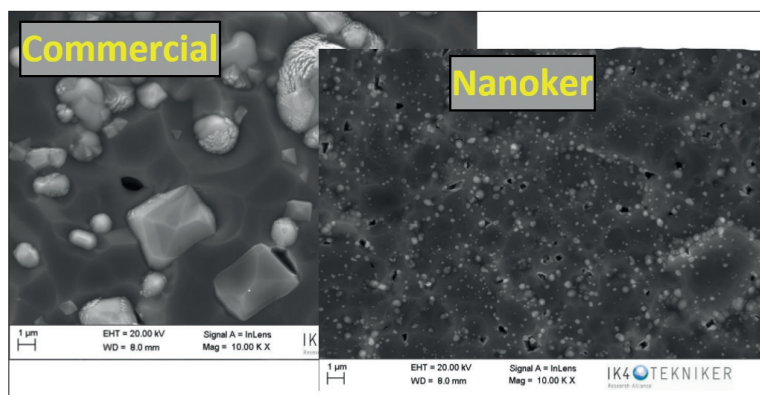


Fig. 7 Microstructure of the high-quality AZO target compared to commercial grade

Fig. 6 shows such 3-inch targets developed for lab-scale sputtering equipment's, and Fig. 7 shows the microstructure of the high-quality AZO target compared to a commercial grade.

For the efficient production of larger diameter TCO sputtering targets maintaining this high quality simultaneously, it has been shown that the hybrid principle is essential. Only with FAST/Hybrid was it possible to get similar microstructural homogeneity, high density and electrical conductivity, even in large diameters (see example in Fig. 8 with an AZO disc of 230 mm).

Ultra-High-Temperature Ceramics (UHTC) is a family of materials that can withstand extreme environments at temperatures higher than 2000 °C with low degradation. These materials are mainly based on borides, carbides and nitrides, with zirconium diboride (ZrB_2) as one of the most promising. One of the main drawbacks of these materials are its brittleness and low thermal shock resistance, which limit its application for

structural purposes. This is the reason why second phases, like silicon carbide, or reinforcements, such as fibres, are added to increase their capability to bear loads and impacts.

A UHTCMC's is therefore a material composed of an ultra-high-temperature ceramic matrix (such as ZrB_2) and a reinforcement made of fibres (such as carbon fibres), like the microstructure shown in the Fig. 9. The densification of this kind of materials is complex as it requires high sintering temperatures (ranging 1850–2050 °C), inert atmospheres and the simultaneous application of pressure during sintering to achieve a good fibre-matrix interface. The low resistivity of the powder compact makes this material an ideal candidate for FAST/SPS or FAST/Hybrid consolidation.

In this case, the sintering of medium to large components (from 70 mm up to 230 mm in diameter) by using conventional FAST/SPS sintering without the aid of an external heater, did result in materi-

als with good homogeneity throughout the thickness and diameter (Fig. 10), although it can be expected that – similar to TCO sputtering targets – the additional external heating of FAST/Hybrid will be essential, when it comes to larger part sizes, higher heating rates and/or higher quality requirements respectively. These materials will be used for the manufacturing of nozzles for the combustion chambers of rockets and for Thermal Protection Systems (TPS) of space shuttles (Fig. 11).

Materials for cutting tools and stamping must fulfil a range of high technical specifications, specifically in the mechanical properties, such as toughness, strength and hardness, but even grain size and its distribution is essential, especially at the cutting edge. The development of advanced Technical Ceramics for these requests is continuously improving with increasing product development/design capabilities, high-added-value products and sustainable processing.

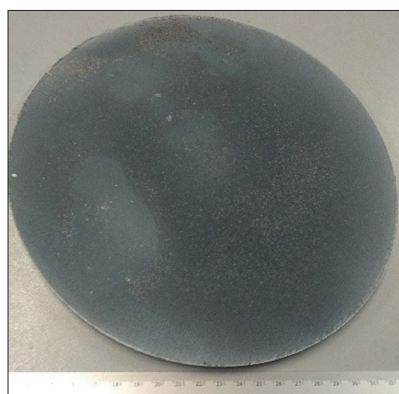


Fig. 8 230-mm diameter AZO sputtering target

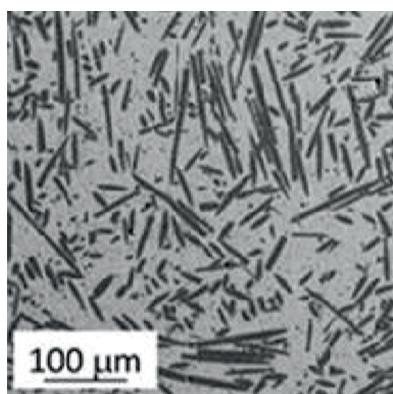


Fig. 9 Microstructure of a ZrB_2 /fiber UHTCMC (Source: CNR-ISTEC)

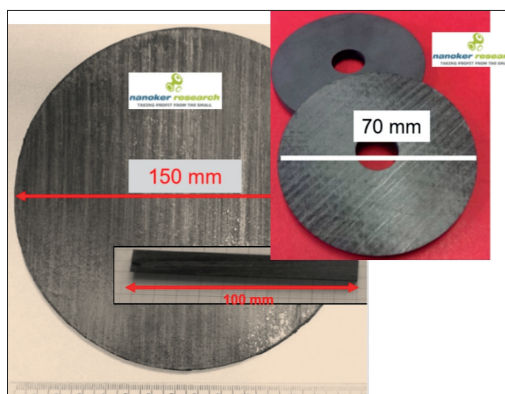


Fig. 10 UHTCMC parts sintered by FAST/SPS

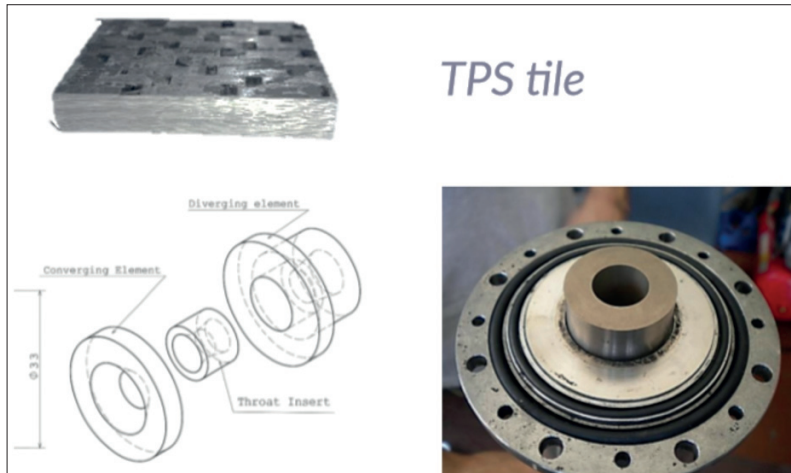


Fig. 11 TPS tiles and nozzle prototypes made of UHTCMC (Source: Charme Project)



Fig. 12 Al_2O_3 -TiC-SiC disc with 150-mm diameter and 11 mm thickness

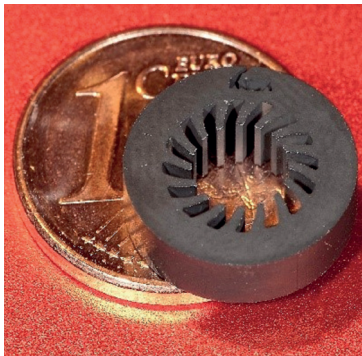


Fig. 13 Al_2O_3 -TiC-SiC sample: micro-machining by means of EDM



Fig. 14 Ceramic cutting tools made of Al_2O_3 -TiC-SiC

Nowadays, the current processing technologies have a limitation to manufacture and convert these promising materials into complex custom-made components. At this point, a development of ceramic based materials was possible by FAST/SPS which allows economic production of large ceramic

blanks that can be shaped by cost effective and – in the field of ceramics – non-conventional machining processes, such as Electrical Discharge Machining (EDM). The ceramic materials can be machined into complex 3D-shapes, overcoming several limitations of conventional processes, like

diamond tool grinding and sawing, and allowing more accurate, flexible and cost-effective shaping. For this purpose, the newly-developed materials are electrically conductive ZrO_2 - and Al_2O_3 -based composites, focused on ZrO_2 -WC/TiN and Al_2O_3 -TiC-SiC systems respectively. The mechanical properties of these innovative ceramics fulfil the high demanding requirements for engineering applications, such as industrial machinery components, cutting tools, wear parts and so on.

Fig. 12 shows an Al_2O_3 -TiC-SiC 150-mm-diameter disk sintered by FAST/SPS. The sintering conditions for this material are 1780 °C and 40 MPa under vacuum atmosphere. This material, specifically designed for cutting tools, has a very homogeneous and dense microstructure and can be machined by means of EDM with very precise features (Fig. 13–14).

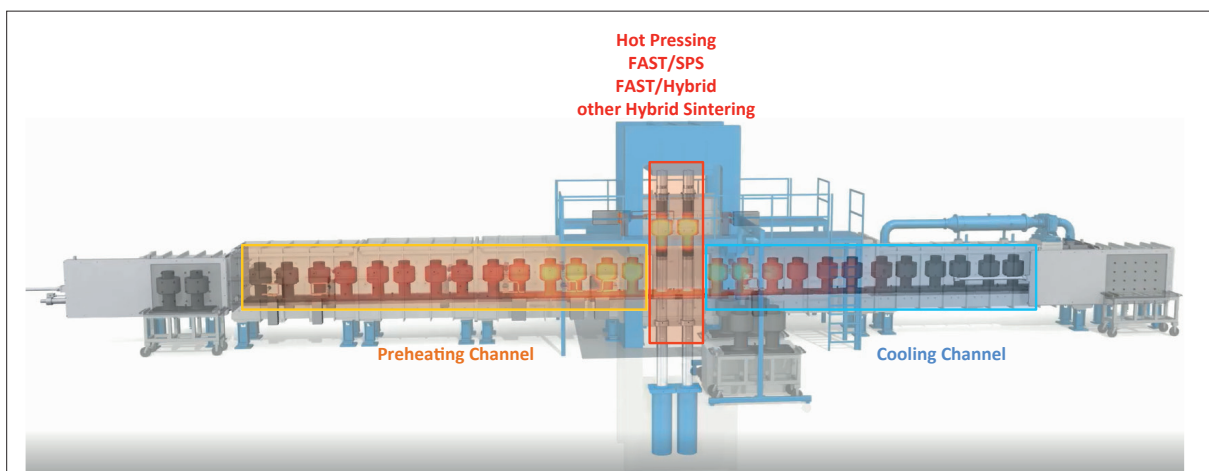


Fig. 15 Industrial hybrid sintering plant for highest throughput

5 High-throughput Hybrid Sintering

If highest efficiency is required, especially in case of large parts, the thermal mass of the part together with the surrounding pressing tool, leading to a long cooling period, can be a major problem. A very good solution for this challenge is the decoupling of the time-consuming cooling from the densification period with a double-chamber system, using a separate cooling chamber.

If this is not sufficiently effective, the chamber can be extended to a cooling channel, even if the thermal shock sensitivity of the sintered parts prevents fast cooling. If the inherently short cycle time of FAST/SPS or Hybrid/FAST still does not deliver sufficient productivity, the addition of a pre-heating channel at the input side of the unit can reduce the cycle time further [8]. Such a high throughput systems is currently under construction at FCT, as shown in Fig. 15, with a

pre-heating channel on the left and a cooling channel on the right. The centre main chamber can be realized as conventional Hot Pressing, but also in the form of FAST/SPS, FAST/Hybrid or other Hybrid Sintering technologies.

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