Aspects Concerning the Super-fast Sintering of Powder Metallic and Ceramic Materials

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Abstract

The application of the energy input principle used in spark plasma sintering technology (SPS/FAST) yields interesting results that are beneficial to the super-fast sintering of moulded parts made of ceramic and powder metallurgy materials, particularly if these materials include the finest-grain initial powders.
Thanks to this modified SPS/FAST technology it seems to be possible to exploit the full potential of submicron and nanoscale powders as well as the resulting microstructures even in connection with complex component geometries. Results have started to show in ceramic and powder metallurgical materials.

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sintering, FAST, SPS, SiC, cemented carbide

1 Introduction

The Spark-Plasma-Sintering technology [1, 4] has been considered as advanced technology for the solidification of submicron and nano materials for some years. At present, the extensive literature available and promising results focus on components with simple geometries such as disks, squares, rings, etc. Sintering devices for this technology are available from FCT under the acronym "FAST" (Field Assisted Sintering Technology).
In this regard, the most important aspect of "FAST" or SPS technology [2–3] is the extremely short time required for the heating process and the short soaking time at sintering temperature, which automatically leads to structures composed of very fine grains [5–7]. The properties of these structures are very promising, as they may make components stronger, tougher, and harder compared with conventional sintered materials.
Similar to conventional hot pressing the SPS/FAST technology is often applied at extremely high temperatures using pressing tools made of graphite, refractory metals or other special materials, which makes it hard to avoid the geometrical limitations mentioned above.

Now, exploiting the potential of nanoscaled or submicron powder for more complex shaped components, the use of mechanical force to support consolidation is hardly possible. This is why basic tests applying the SPS/FAST heating principle were conducted to find a way that enables such components to be manufactured in short sintering cycles while exploiting the potential of the initial powder. These development works focus, in particular, on finding ways to mould complex shaped components by means of dry pressing technology as well as MIM and PIM technology, respectively.

2 Experimental

In a spark plasma sintering plant FCT HP D 250/1 (Figs. 1–2) (maximum operating temperature of 2400 °C) a specially developed "tool" (high speed furnace cartridge, Fig. 3) was used to conduct fast sintering trials. To explore the basic potential of this procedure, the trials were conducted with materials that have been used under industrial conditions and proven to have a great potential. The use of these materials allowed us to compare the resulting structure, the material properties (strength, hardness, toughness), and, primarily, the achievable density with the conventionally sintered materials.
The experimental setup used (Fig. 3) makes it possible to sinter components up to 30 mm in diameter in the spark plasma sintering machine mentioned above for total heating times of a few minutes at maximum temperatures of above 2200 °C. A second experimental setup

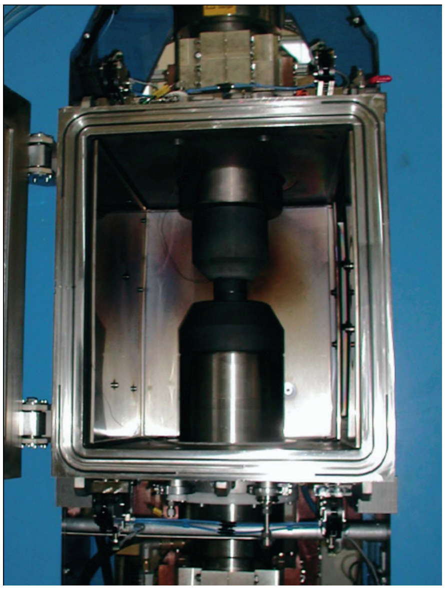

Fig. 1 • View into the SPS vacuum vessel

Fig. 2 • "High speed" kiln at an operating temperature of 1800 °C

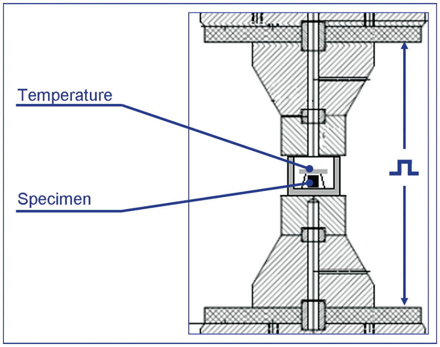

Fig. 3 • Experimental setup for SPS machine HP D 250 for an operating temperature of 2200 °C

allowing for the sintering of components up to 80 mm in diameter is under construction.

The basic tests currently examining tungsten carbide/cobalt, steel/titanium carbon nitrite, tungsten/copper, B_4C/TiB_2, and C-SiC show very encouraging results that should lead to further testing.

3 Test results/Examples

3.1 Cemented carbide

For initial tests, uniaxial dry pressed parts made of cemented carbide (tungsten carbide + 10 % Co) with a final size of 30 mm × 12 mm × 2 mm were sintered. As expected, the very first trials showed that temperature measurement and control is a special challenge for such extremely high heating rates (up to 2000 K/min). Nevertheless after some development work it became possible to sinter samples to the desired density and geometry within only 2.6 min (= heating time + soaking time). Figure 4 shows a typical sintering cycle and Table 1 some results of the trials using this material. Currently the application relevant material properties of the hard metal samples are analysed in comparison with conventional sintered parts. In the next step the special tailoring of the initial powders, e.g. reduction of cobalt content, should lead to a further improvement of the material properties (e.g. hardness, toughness) as well as a cost reduction.

3.2 Sintered silicon carbide

As an example for a technical ceramic material, sintering tests were conducted on ring-shaped standard parts (20 mm diameter) uniaxially pressed to approx. 1.8 g/cm³ of silicon carbide powder (BET 13 m²/g, with B/C additives). This material is usually sintered in standard furnaces with 12 h cycle time (soaking time = 3 h). Figure 5 shows a typical sintering cycle, in Argon atmosphere. The results are summarized in Table 2.

The sample density so far achieved, using a total heating time below 4 min, amounts to 3.03 g/cm³, which is close to the desired density of 3.12–3.15 g/cm³. The continuation of this work with specially tailored powder mixtures using raw materials with e.g. higher BET surface is currently scheduled in order to achieve further density enhancement without any change in microstructure or grain size, using similar or even reduced sintering

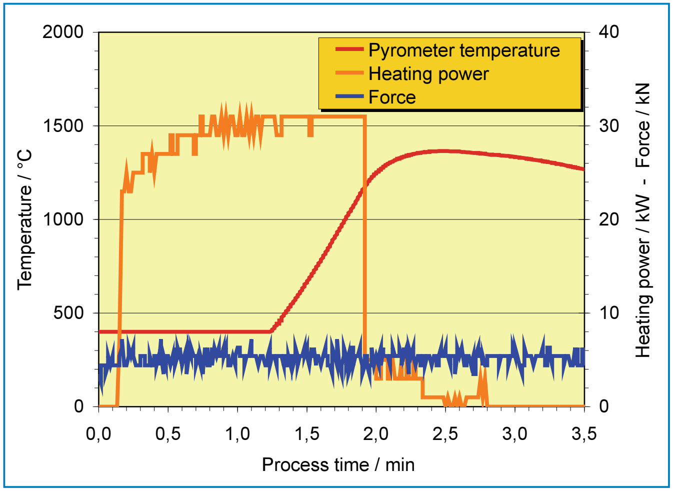

Fig. 4 • Typical sintering cycle for cemented carbide at 1365 °C

Table 1 • Results of the first sintering tests using cemented carbide parts (Density of conventionally sintered part = 14.4 g/cm³)

Test	Atmosphere (argon)	Temperature (max.) / °C	Time to soaking temperature (max.) / s	Soaking time / s	Density / g/cm³
1	1 bar	1435	140	300	14.4
2	0.1 mbar	1435	140	300	14.7
3	0.1 mbar	1400	100	160	14.4
4	0.1 mbar	1450	120	60	14.0
5	0.1 mbar	1365	100	60	14.4

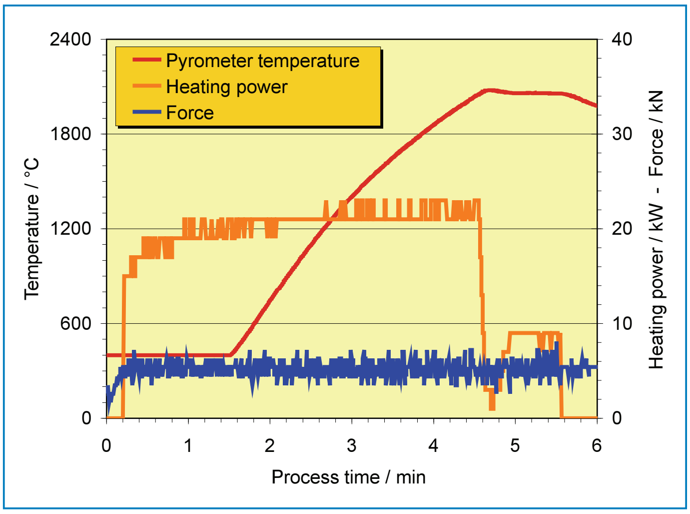

Fig. 5 • Typical sintering cycle for SSiC at 2060 °C

Table 2 • Results of the first sintering tests using SSiC parts (density of conventionally sintered part = 3.12 g/cm³)

Test	Temperature (max.) / °C	Time to soaking temperature (max.) / s	Soaking time / s	Density / g/cm³
1	2075	265	60	2.99
2	2060	210	60	3.00
3	2060	175	60	3.03

time and temperature. These tests are intended to show if the same or similarly beneficial material properties can be achieved for more complex geometries as were achieved by applying standard SPS technology.

3.2 C/SiC-composite ceramics

Another example for a possible application of the high speed sintering system is the siliconizing of initial bodies made of carbon fibres. Siliconizing is performed for component wall thicknesses of approx. 20 mm at a maximum temperature of 1550 °C and during short total intervals of no more than 5 minutes. The microstructure analyses conducted so far have not shown any significant differences to standard components siliconized over a longer period of time. As they have also been very promising, these tests will be continued with a larger "high speed system".

4 Outlook

Since the results gathered from previous development work using fast SPS sintering technology are very promising, work is underway to create a "high speed cell" integrated into a dry pressing automat (Fig. 6) and operating under vacuum or pressure of up to 60 bar, which is intended to fully exploit the conceivable potential of this technology.

It appears to be possible to predict the benefits previously limited to simple geometries also for complex shaped components that could be made by SPS/FAST technology using submicron or nanoscaled initial powders. This is of particular interest for the machinery and plant construction sector and, in particular, for the automotive industry. We are justified in our high expectations for the results of further studies.

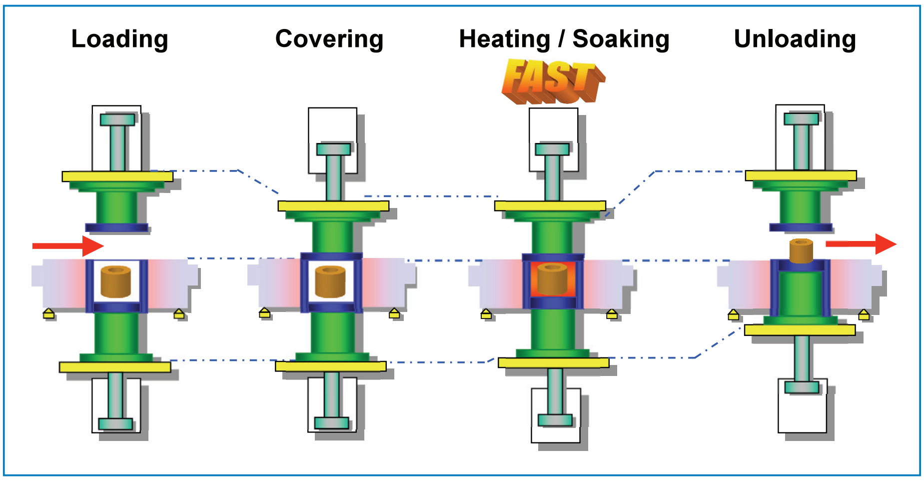

Fig. 6 • Integration of SPS "high speed" technology in powder processing technology

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