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Spark Plasma Sintering Of Diamond Impregnated Wire Saw Beads

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Abstract

Diamond MMCs are widely used for the cutting of stone and concrete. An effective way of cutting large sized parts is to use wire saws which have hundreds or thousands of small beads on a flexible wire. Typically, these diamond impregnated MMC beads were conventionally sintered by vacuum sintering, pressure sintering or sintering in continuous furnaces.
In the present study, the spark plasma sintering of Diamond/MMCs with Co-free Steel/Cu matrix composition was investigated. The results of the physical, chemical and mechanical properties of short time sintered beads are compared with conventional sintering. The focus of the study is on the interaction between matrix metals and diamond, as well as on the design of the multi-part pressing tools for net shape sintering.

Introduction

The digging of natural stones like marble, lime stone or granite as well as the processing of concrete or (reinforced) masonry demanded diamond tools, which match the demanding requirements of the tool/stone interaction. The established diamond tools are typically based on the diamond/cobalt-, diamond/copper- and diamond/iron-system. But for a lot of applications, the properties of the tool materials are not optimized for the problem.
Besides pressureless sintering methods, the hot pressing of diamond impregnated tools is widely used [1]. The use of modern short time sintering methods, e.g. Spark Plasma Sintering is economically interesting and allows an exact control of the diamond/matrix interface due to the short sintering times. Due to the direct heating by a pulsed electric current, high heating rates (up to several hundred deg. C per minute) and short sintering times can be archived. An advantage of this technique is the high degree of flexibility, thus it is easy to develop application adapted sinterbodies within short times.
In this study, we investigate the sintering behaviour of a diamond-impregnated steel/copper MMC along with an evaluation of the diamond/matrix interaction. Additionally, results from Spark Plasma Sintering with multi-cavity tools are presented.

Experimental

For the steel matrix, a gas atomized powder of a Cr-Ni-steel was used. The powder was mixed with dendritic copper powder in Cu-mass fractions between 20 and 60 %. The diamonds (40/50 mesh) were added with a volume fraction of 7.5 % in the composite (Fig. 1).

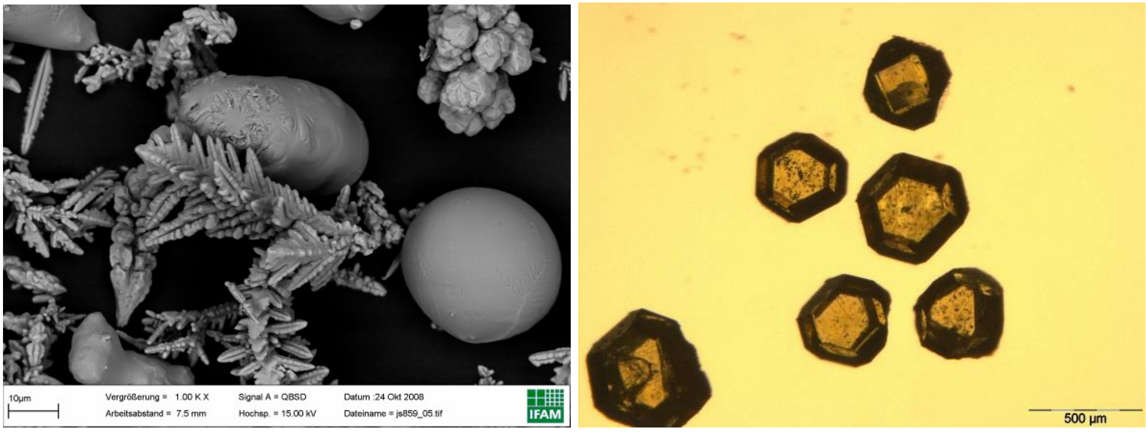

Figure 1:
SEM image of the powder particles used in the steel/copper mixture. The steel particles are round, spherical particles, the copper powder show a dendritic morphology (left). Stereo-microscopic images of the diamond crystals used in this study (right).

The Spark Plasma Sintering was performed using a HP D 250 C (FCT Systeme GmbH) high-throughput system. The samples were heated by a pulsed electric current which flows through the punch-die-sample-assembly using a high current and low voltage. In this study, the pressing tools were made of high-performance graphite and contain up to 17 cavities for simultaneous sintering. According to the SPS technique, the powder mixture is heated stepwise from room temperature to desired sintering temperature with a heating rate of 100 K/min in dynamic vacuum. Temperature measurement was done by a pyrometer. The measurement position was inside the pressing tool. The density of all consolidated bulk specimens was measured by the Archimedes technique. The microstructure was studied by optical microscopy and SEM of fracture surfaces or on sinterbodies of matrix powder without diamonds.

The Raman spectra were recorded with LabRam System 010 (Jobin Yvon GmbH) in reflection geometry by the use of a microscope with several lenses (10- to 100-fold). An excitation wavelength of 488 nm (He/Ne-laser, 15 mW) was used. The resolution of the measured Raman shift was approximately 4 cm^{-1}. Additionally, the system contains two Notch-filters for the lower range of Raman-shifts. With these, modes starting from approximately 80 cm^{-1} can be detected.
Iron carbide Fe_3C, used as reference material was prepared by Spark Plasma Sintering of a mixture of carbonyl iron and graphite powder at 1050°C [2]

Reference samples of diamond-impregnated Steel/Cu MMCs were produced by vacuum sintering of cold compacted green bodies at sintering temperatures up to 1200°C.

Results and Discussion

The powder mixture was poured into the typical graphite die used for Spark Plasma Sintering, which contains horizontally and vertically 0.2 mm graphite foils to protect the die material. The sintering was performed above the melting temperature of the copper in the way of classic liquid phase sintering. Due to the typical temperature measurement during the SPS process, the temperatures of the die or the punch are lower than the real temperature inside the sinterbody [3]. At die temperatures higher than approximately 900°C the copper starts melting and infiltrates the steel powder network. An abrupt displacement of the punches can not be observed, but the microstructure changes drastically (Fig. 2 and 3).

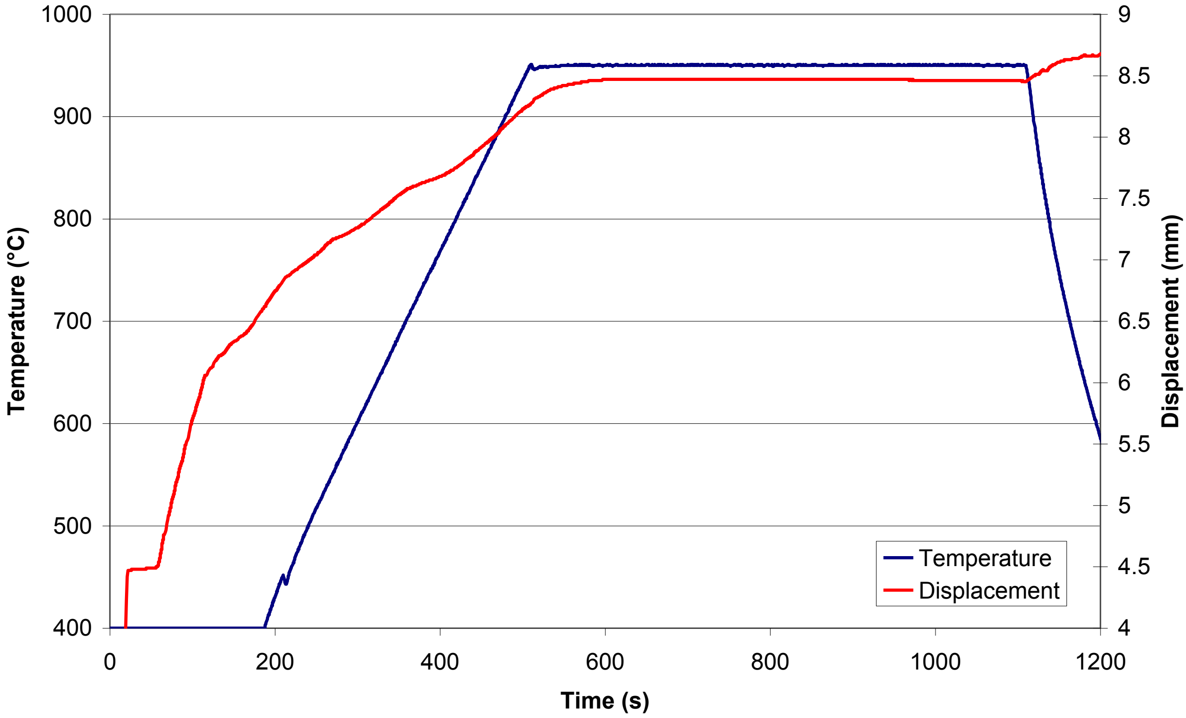

Figure 2:
Typical time depended temperature/displacement characteristics for the Spark Plasma Sintering of diamond-MMCs with Steel/Cu matrix.

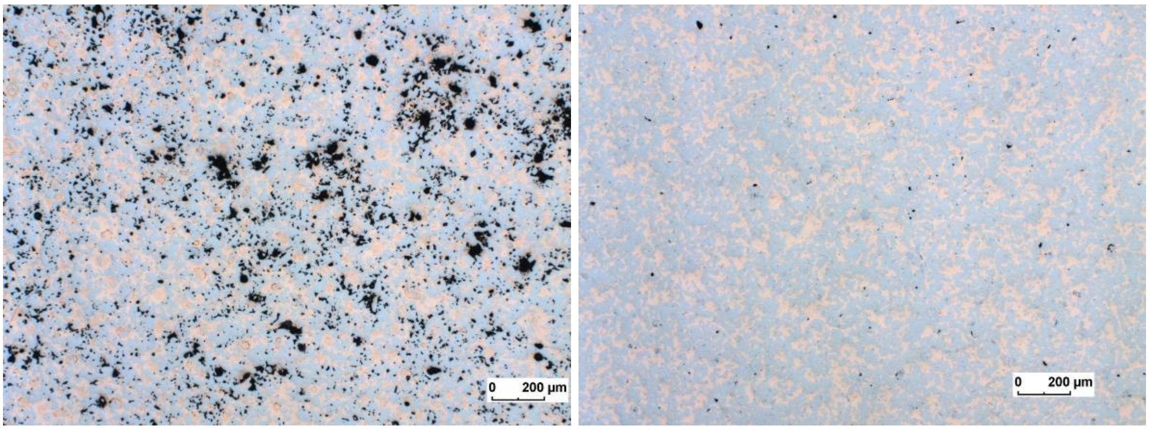

Figure 3:
Microstructure of the Steel/Cu sinterbodies, Spark Plasma sintered at 800 °C (left) and 1000 °C (right) without dwell time.

Due to the reaction between the iron and the carbon of the diamond, the surface of the diamond starts to degenerate. Depending on sintering temperature and sintering time, the surface of the diamonds show reaction zones with different characteristics. For low temperatures and short times small surface layers can be observed, higher temperatures and long sintering times led to craters or to complete destruction of the diamond. Figure 4 show representative examples for diamonds sintered in Steel/Cu matrix for different sintering temperatures and dwell times. The metal matrix was removed by etching with concentrated nitric acid at room temperature.
Precise control of the reaction between the diamond and the matrix is important for the bonding between the phases and consequently for the cutting performance of the tool material.

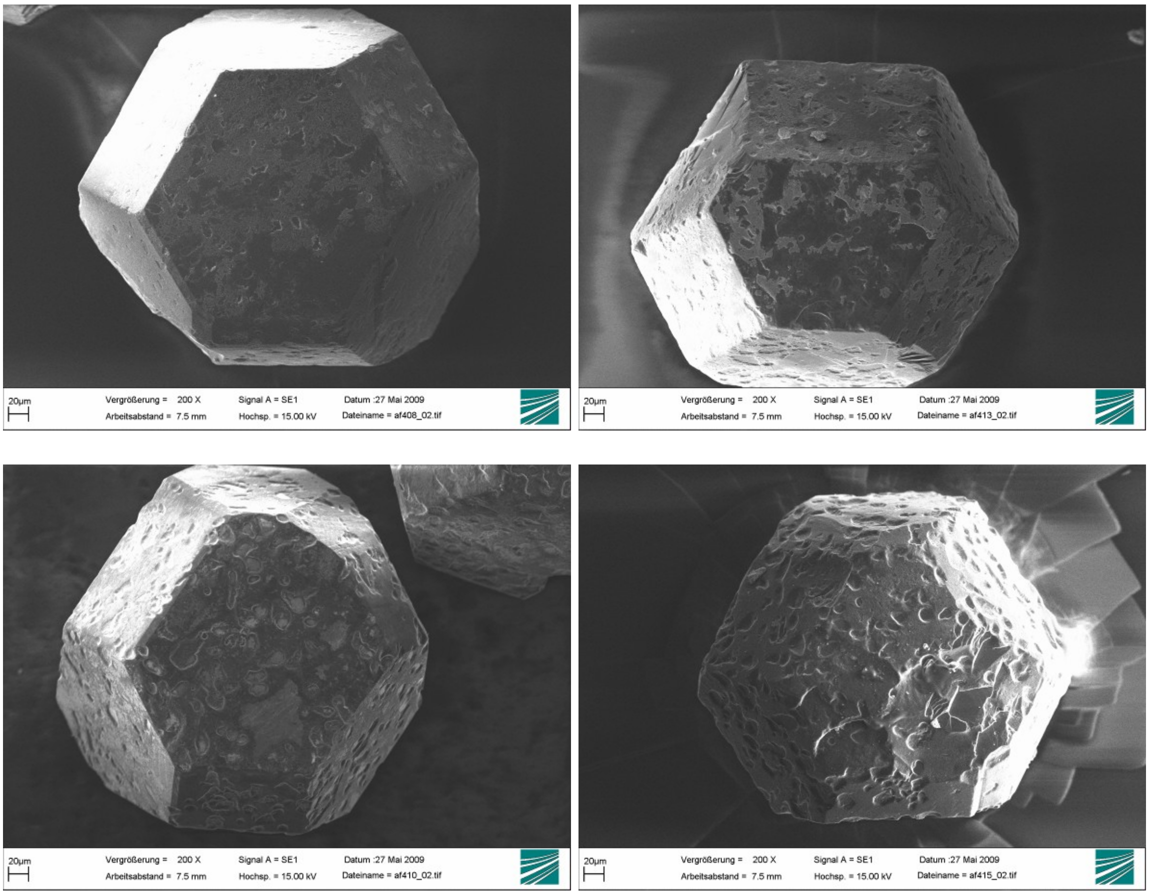

Figure 4:
SEM-Images of diamonds after Spark Plasma sintering in a Steel/Cu matrix for different temperatures and sintering times. Upper row: left: 900°C, 0 min; right: 950°C, 0 min; Lower row, left: 900°C, 5 min; right: 950°C, 5 min.

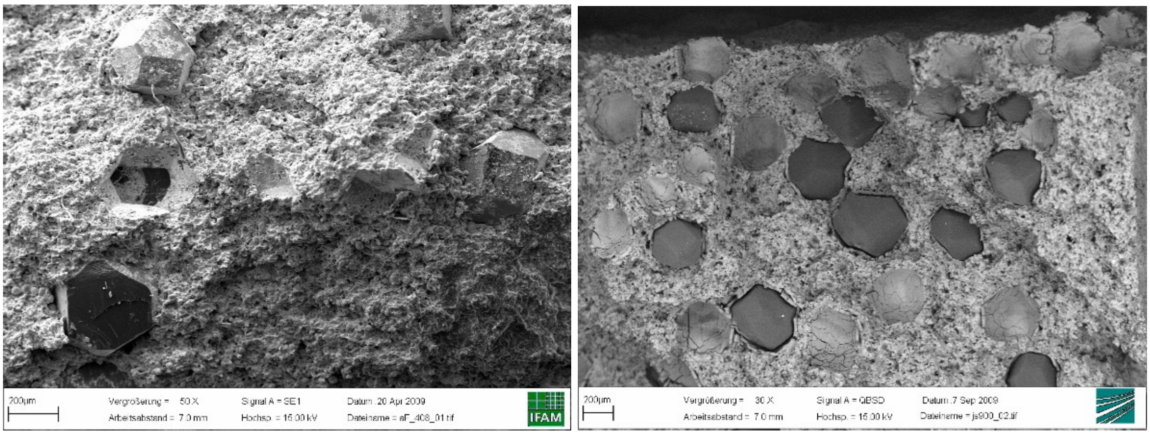

Figure 5:
Left: Fracture surface of a Spark Plasma sintered Diamond/Steel/Cu composite with diamond pullout pits, containing residual traces of the reaction layer.
Right: Fracture surface of a conventionally vacuum sintered Diamond/Steel/Cu composite, showing extensive cracking within the diamond pullout sides.

Fracture surfaces of sintered Steel/Cu matrices show the extent of bonding between the diamond and the matrix - Fig. 5. There are some diamonds with transgranular cracking in the diamond. In contrast to the conventionally vacuum sintered samples, there is no evidence of transgranular cracking within diamond pullout sites. Reaction layers can be observed in both cases, but the occurrence is less for the short time sintered samples.

Local analysis of the diamonds and the diamond pullout sites with micro-focus Raman-spectroscopy were used for qualitative chemical analysis. Figure 6 show the spectra of several analyses compared with reference materials. The results show that the reaction between the iron matrix and the diamond forms iron carbide Fe_3C. Differences between the curve of the Raman-shift between pure Fe_3C and the residue in the metal matrix (inside diamond pullout pit) can be explained by the composition of steel powder, which is used for the Steel/Cu matrix. The analysis of diamonds shows the same results for sinter regimes with observable deterioration of the diamond surface. After the generation of a thin reaction layer, the first peaks at 1585 cm^{-1} and 2660 cm^{-1}, indicating the Fe_3C formation, can be observed.

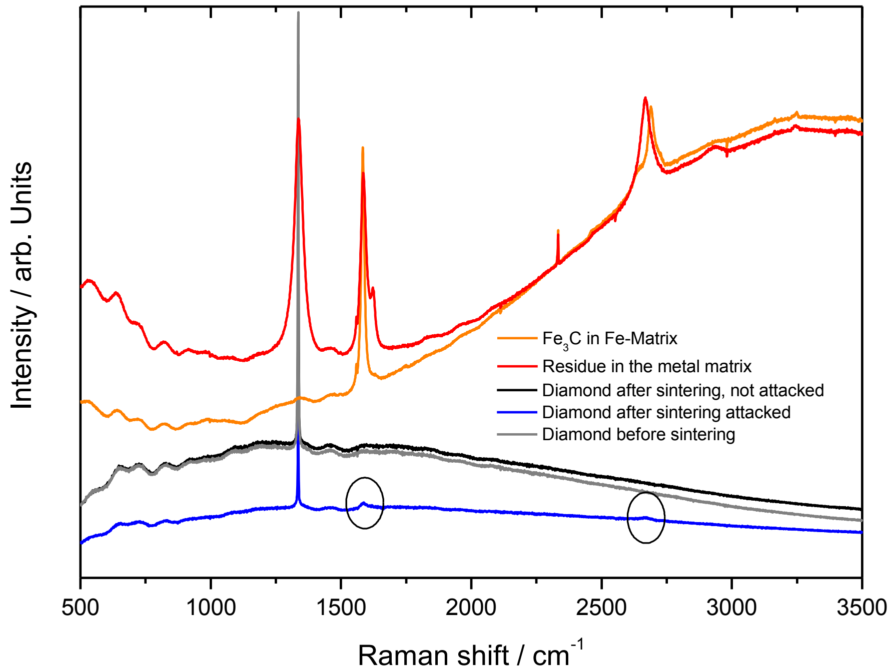

Figure 6:
Intensity of the Raman shift for diamonds sintered in Steel/Cu matrix and residual traces in diamond pullout pits.

To increase the productivity of the process, multi-cavity dies for the simultaneous sintering of several diamond impregnated wire saw beads, composed of a steel core with internal screw thread and a sintered diamond-MMC on the outside were developed (Fig. 7). Sintering and bonding to the steel core can be done in one single step by Spark Plasma Sintering.

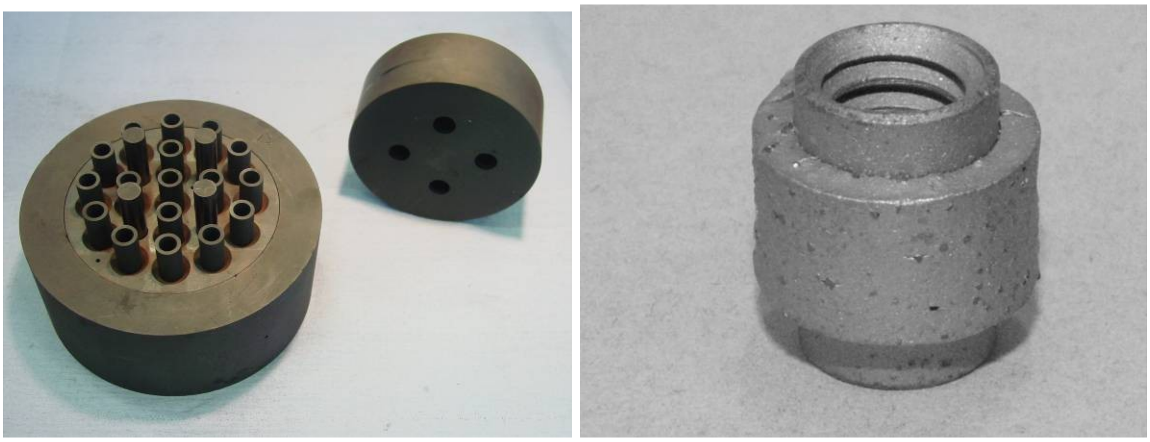

Figure 7:

17-fold multi-cavity die insert for the Spark Plasma Sintering of diamond-impregnated wire saw beads (left). Spark Plasma Sintered wire saw bead (diamond in Steel/Cu matrix) (right).

Summary
The results of this investigation demonstrate that diamond impregnated wire saw beads can be successfully produced by Spark Plasma Sintering. The use of multi-cavity dies allows the economic production of large quantities.
Due to the short sintering time, the interface between the diamond and the Steel/Cu matrix can be easily adopted to different applications. By the control of the temperature and the sintering time, the extent of the reaction between the diamond and the steel can be varied. The analysis of the reaction layer on the surface of the diamond and on the inside of diamond pullout sides indicates the formation of Fe_3C.

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