Microwave-Assisted Sintering of Co-based Composites

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Abstract

Cobalt-based-composites are primarily used as cutting or drilling tools for hard materials. Beside the WC/Co-Hard metals, the Co/diamond-composites are important. The preparation of these materials is quite demanding, because the high temperatures and the long heat treatment may cause the damage of the diamond surface (e.g. due to graphitisation). Therefore, the pressureless sintering has less importance for the production of these kinds of materials up to now.

The processing of WC/Co-composites with the help of microwave-assisted sintering is known for years. The success of this technique assumed, that the production of pressureless sintered Co/diamond-materials is possible too. This work presents and discusses a study of the microwave-assisted sintered Co-based composites in order to compare them with conventional sintering. The results were evaluated among other methods through mechanical testing and microstructure analysis.

1. Introduction

Cobalt-based MMCs are common materials for cutting tools and wear resistant applications. The metal is added to create a binder which cements the refractory particles together to form a solid body. The predominant materials used are carbides like the hexagonal tungsten carbide, WC. These hardmetal composites are the most successful combination of hardness, toughness and strength which are universally used for drills, pins for dot-printers, wood machining and cutting tools.

The carbide grain size has a major influence on the properties of the final product and is of vital importance. The control of the grain size throughout the whole process is thus crucial. Microwave sintering was successfully used for the sintering of WC/Co-composites. One major goal of the microwave application for sintering purposes is the benefit from volumetric heating. This leads to lower amounts on porosity in the sintered part and the shorter sintering time, necessary in case of the microwave sintering results in a finer grain size compared with conventional sintering [1].

Typically, WC/Co composites contain less than 25 vol.% cobalt and predominantly carbides, which act as absorbers for microwave radiation (so called suszeptors). Because of this, microwave sintering of WC/Co is known longer then the microwave sintering of PM parts in general [2, 3]. Cobalt-based MMCs with high Co-contents are used as tools for the cutting or sawing of natural stone. As hard materials diamond and cubic boron nitride are widely used. The production technology of diamond/Co-composites mainly consists of pressure assisted sintering like hot-pressing. Because of the degradation of the diamond surfaces at temperatures above 900°C, the sintering should be very quick or at low temperatures. The successful use of the microwave radiation for the sintering of WC/Co and other metals (e.g. steel [4]) suggests investigations on MMCs with high cobalt contents. For this study, Co/Al_2O_3-, Co/SiC- and Co/diamond-composites are sinterd with microwave support in comparison with results from conventional sintering. Al_2O_3 and diamond are microwave transparent materials. The absorption of MW radiation by alumina at higher temperatures is negligible for this work. SiC is a well known MW absorber and widely used as a susceptor in MW heating technology. Additionally, SiC reacts with Co at higher temperatures, so it is possible to compare the reaction with and without MW heating.

2. Experimental

Commercially available Co-powder (99.8 %, Alfa Aesar, 1.6 µm) was mixed with different amounts of alumina (ALCOA, T60 0.2-0.6 µm) or silicon carbide (ESK-SiC GmbH, F46 300-425 µm) (up to 20 vol.%) in a tubular shaker. Co/diamond cutting tool green bodies with 5.5 % diamond (300-420 µm) were used for the experiments. The mixed powders were pressed to bending test bars with a pressure of 200 MPa. The green density is about 50-60 % of the theoretical value for all samples. The densities of the sintered samples were measured by the Archimedes' method.

The microwave furnace used in this study was designed and fabricated by Gero Hochtemperaturöfen GmbH, Neuhausen, Germany and InVerTec e.V., Bayreuth, Germany. (Figure 1) The explosion proof multimode microwave furnace consists of a 2.45 GHz microwave generator with a continuously adjustable power output (0.6 to 6 kW.) Input and reflected microwave power are measured, from which the absorbed power in the resonator was calculated. The resonator is completely made of molybdenum sheet. Above that, the furnace is equipped with conventional Mo-heaters (150 kW) outside the resonator (hot wall-resonator).

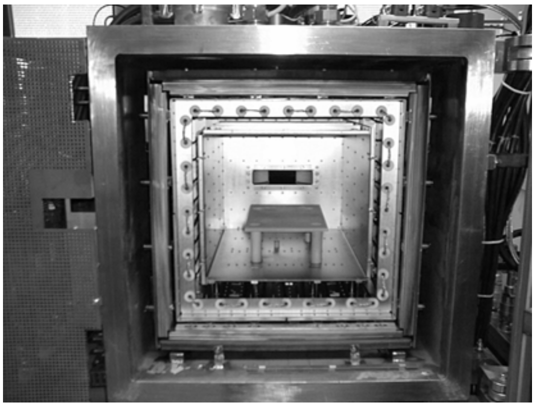

Fig. 1: Look inside the MW furnace with Mo cavity and charge carrier made of hBN.

Temperature measurements are done by infrared pyrometry (Keller HCW PZ 20). The value for the emissivity was calculated by the comparison of the temperatures measured by thermocouple and pyrometer after conventional heating.

Conventional and microwave sintering of the samples was carried out in the same furnace to ensure comparability. In general, the bars were placed on a charge carrier made of hBN. For some microwave sintering experiments, they were also placed inside a thermal insulation made of alumina (KVS1800, Rath GmbH). The sintering is done in N_2 / 6% H_2 atmosphere at different sintering temperatures measured by the pyrometer. The heating rate was 300 K/h; the holding time was 20 min.

At the beginning microwave sintered samples were conventionally heated to a sample temperature of about 800°C. For experiments with higher MW field intensities, the samples were preheated only to 700°C. In all cases, after thermal relaxation, the microwave radiation was switched on and the samples were heated to the target sintering temperature. The heating time and the holding time were in the same order for conventional and microwave sintering.

3. Results and Discussion

The change of the emissivity with the temperature for Co-based MMCs is shown in figure 2. Firstly, the emissivity decreases with increasing temperature. This can be understood because the reflectivity of the sample increases with increasing temperature. The origin of this behaviour is the lower surface roughness at higher temperatures.

The relative densities of the Co/10 vol.% SiC MMCs, prepared by conventional and microwave sintering are shown in Fig. 3. Higher densities were archieved with increasing temperature up to 950°C. Higher temperatures lead to the reaction between SiC and the Co-matrix metal. Obviously, the reaction products are responsible for the reduced densities at temperatures above. The bending strength of the Co/10 vol.% SiC corresponds well with the sintered density (Fig. 4).

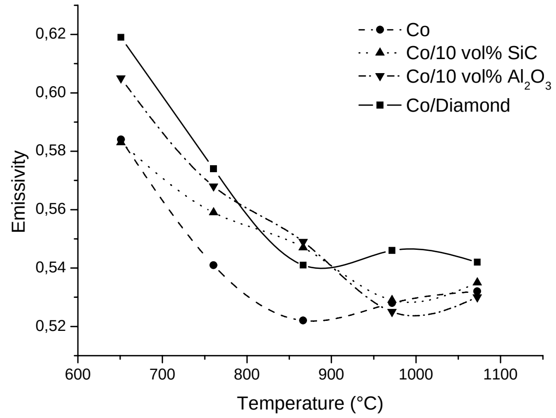

Fig. 2: Emissivity for Co-, Co/10 vol.% Al_2O_3, Co/10 vol.% SiC, and Co/diamond powder compacts obtained by conventional heating of bending test bars compacted with 500 MPa.

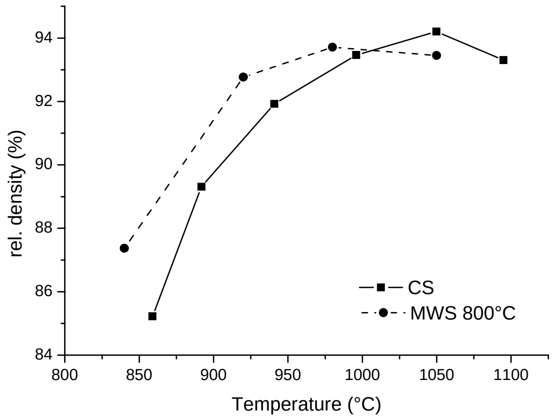

Fig. 3: Relative density of Co/10 vol.% SiC composites for conventional and microwave sintering.

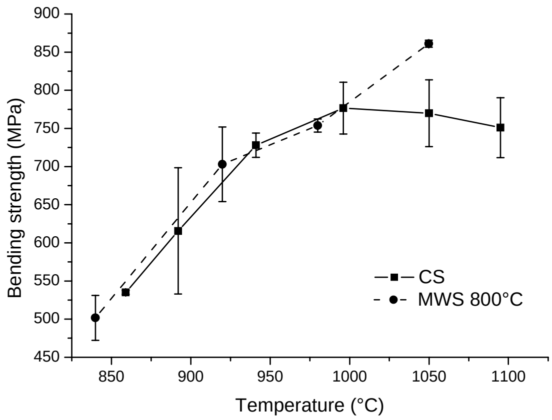

Fig. 4: Bending strength of Co/10 vol. SiC composites for conventional and microwave sintering.

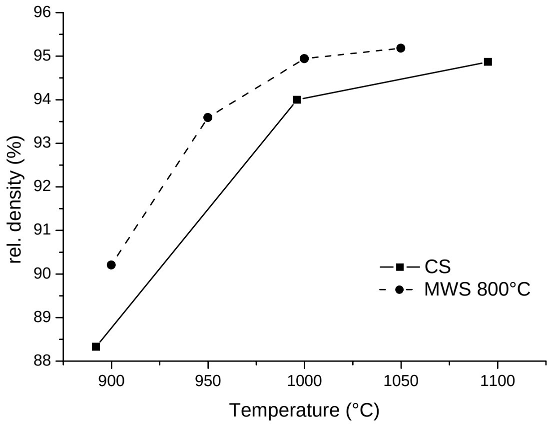

Fig. 5: Relative density of Co/10 vol.% Al_2O_3 composites for conventional and microwave sintering.

For Co/Al_2O_3 composites, the behaviour is similar to the Co/SiC MMCs (Fig. 5). In case of Co/Al_2O_3 no reaction occurred in the investigated temperature range.

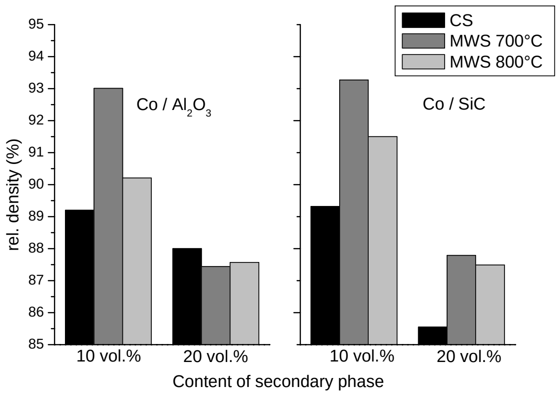

Fig. 6: Relative density of different Co/SiC and Co/Al_2O_3 composites for conventional and microwave sintering (sintering temperature 900°C).

Fig. 6 compares the relative density of Co/SiC- and Co/Al_2O_3-composites with different amounts on ceramic phases for conventionally and microwave sintering. With increasing content on alumina in the Co/Al_2O_3-MMCs, the influence of the microwave radiation decreases. For Co/20 vol.% Al_2O_3, the relative densities of samples prepared by both methods are the same within the range of the measuring error. For Co/SiC composites, the relative density of the microwave sintered samples is always higher than for the conventionally sintered ones. But also for samples with higher SiC contents, the influence of the microwave radiation tends to decrease. The effect of the microwave radiation is in all cases dependent on the strength of the electromagnetic field. If the field intensity increases, the density of all samples increases also. This shows that the activating effect on the sintering process is originated by the microwave radiation.
At temperatures above 900°C, SiC reacts with the Co matrix to form intermetallic phases (Co_2Si, CoSi) with carbon precipitations (Fig. 7). This reaction leads to higher porosity and lower densities of the samples. The relative densities given for temperatures above 900°C are calculated by the theoretical density of Co/10vol.% SiC and Co/20vol.%. SiC respectively. The exact phase composition (Co, SiC, Co_2Si, CoSi, C) is unknown and because of this, the theoretical density not exact.

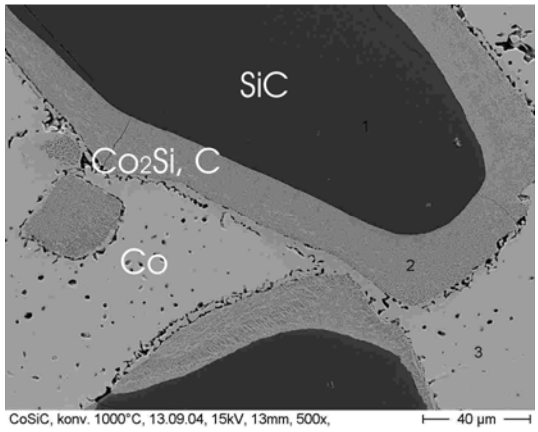

Fig. 7: Reaction zone between Co and SiC in the Co/SiC MMCs sintered at 1000°C. (SEM, BSE)

Quantitative image analysis of metallographic preparations shows different amounts on reaction products for both sintering methods (Fig. 8).

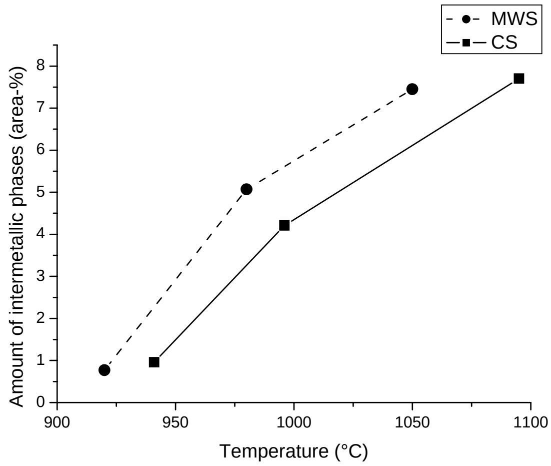

Fig. 8: Amount on products of the reaction between Co and SiC in the microstructure, measured by quantitative image analysis.

The reaction between SiC and Co seems to be accelerated in the presence of the electromagnetic field.

It is conspicuous, that the difference between types of MMCs is very small. SiC, as a suszeptor absorbs microwave radiation much more than alumina and acts as an internal heating source [5]. In these investigation, an additional effect on the sintering, based on the different behaviour in the electromagnetic field is not significant.

The effect of the microwave radiation on the sintered density for conventional and microwave sintering of Co/diamond composites is show in fig. 9.

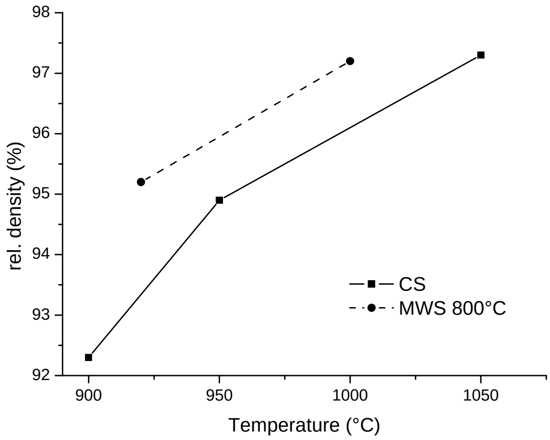

Fig. 9: Relative density of Co/diamond composites for conventional and microwave sintering.

At the maximum value of the density, closed porosity is reached. With longer sintering times, the density can' further increased. Microwave sintering leads to higher densities for all samples. With this technique, the sintering temperature for Co/diamond-composites can be decreased up to 100°C or allow shortening the process time.

4. Conclusion

Microwave sintering is shown to be an applicable method for the sintering of Co-based composites with high Co-content, too. The electromagnetic field accelerates the sintering process and allows to shorten the sintering time or to reduce the sintering temperature. Especially for the sintering of sensitive materials, microwave sintering has the potential as an alternative production technique.

5. References

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