

Microwave assisted sintering of metallic materials

Jürgen Schmidt, Thomas Schubert, Thomas Weißgärber, Bernd Kieback

*Fraunhofer Institut für Fertigungstechnik und Angewandte Materialforschung
Teilinstitut Dresden Pulvermetallurgie und Verbundwerkstoffe
Winterbergstr. 28, 01277 Dresden*

Abstract

Microwave assisted sintering is an emerging technology. The combination of conventional and microwave heating (so called hybrid heating) leads to rapid heating and enhanced densification. The technique is well known and established for the sintering of ceramics like silicon nitride and alumina. A lot of work is also done on tungsten carbide based hard metals. Typically, the microwave sintered parts show finer and more homogeneous microstructures, whereby the mechanical properties are improved. Examples of the microwave field densification of P/M steel Distaloy AE will be given. In the present work, the differences in the mechanical properties of conventional and microwave assisted sintered powders will be discussed.

1. Introduction

P/M-materials play an important role in production and development of various applications such as automobiles, portable equipment, aerospace equipment and business machines. These products span a variety of areas of applications, starting with self-lubricating bushings up to fully densified high strength structural parts.

Microwave sintering of materials is still a challenging area of applied research. The technique has been intensively studied for 20 years, especially in relation to the heating of ceramic and metallic materials. For metallic materials, the number of publications is quite small, but the feasibility of microwave sintering of metallic P/M parts is proven [1]. One of the materials used for such studies is the Distaloy AE P/M steel powder.

This powder produced by Höganäs AB is known as a microwave absorbing material without strong temperature influence on the absorption up to 900 °C [2]. It is also known that the powder has positive effect on microwave radiation during sintering. Microwave sintered samples achieve higher tensile strengths at lower temperatures than conventionally sintered parts [3]. Ductility is increased for the microwave sintered samples.

2. Experimental

2.1 Furnace

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 multimode microwave furnace consists of a 2.45 GHz microwave generator with a continuously adjustable power output (600 to 6000 W.) 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 (Figure 2). Above that, the furnace is equipped with conventional Mo-heaters (150 kW) outside the resonator. The atmosphere inside the furnace can be adjusted for inert gases like nitrogen, argon or for explosive gases and gas mixtures like hydrogen. Because of the hotwall-resonator with a water cooled mantle, there is no insulating ceramic inside the oven. With this, it is possible to work with metals or alloys which are sensitive to air and moisture.

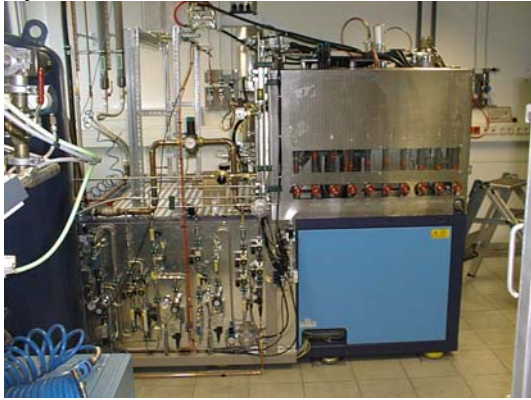


Figure 1:
MW sintering furnace with “hot wall”-cavity made of Mo.

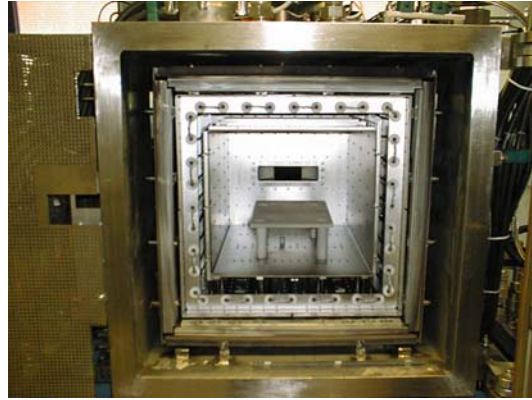


Figure 2:
Look inside the MW furnace with Mo cavity and charge carrier made of hBN.

Temperature measurements are done by infrared pyrometry (Keller HCW PZ 20). In order to obtain correct temperature values, it is necessary to know the emissivity of the sample. This value was determined with a thermocouple placed in the sample. The sample was heated stepwise to temperatures below and above the sintering temperature with conventional heating. The value for the emissivity was calculated by the comparison of the temperatures measured by thermocouple and pyrometer. The onset temperature of the pyrometer is 350°C.

2.2 Materials and Characterization

Commercially available atomized P/M-steel powders (Distaloy AE + 0.2 or 0.5 % C + 0.6 % Kenolube P11, Höganäs AB) are pressed to tensile test bars with a pressure of 500 MPa. The green density is about 7.0 g/cm³. The Distaloy powder is diffusion-prealloyed with 3.75 % Ni, 1.43% Cu and 1.5 % Mo.

The densities of the sintered samples were measured by the Archimedes' method.

2.3 Sintering

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 N₂ / 6% H₂ atmosphere at different sintering temperatures measured by the pyrometer. The heating rate was 300 K/h; the holding time was 20 min.

The microwave sintered samples at first were conventionally heated to a sample temperature of about 850 °C. For higher sintering temperatures (above 1200°C), it was necessary to heat the samples up to 950°C. Then, after thermal relaxation, the microwave radiation was switched on and the samples were heated to the sintering temperature. The heating time and the holding time were in the same order for conventional and microwave sintering.

2.4 Mechanical testing

The mechanical properties of conventional and microwave sintered samples were determined at room temperature. Tensile strength was measured using a Zwick 1476 testing machine. Tensile strength and ultimate strain were appointed by an average of four samples.

3. Results and Discussion

3.1 Emissivity of the samples

The change of the emissivity ϵ with the temperature for Distaloy AE pressed with 400 MPa and 700 MPa is shown in figure 3. First, the emissivity decrease with increasing temperature. This can be understood because the reflectivity of the sample increases with increasing temperature. A similar behavior is known for the electrical conductivity of Distaloy AE powder [2]. For higher temperatures, ϵ is slightly increasing.

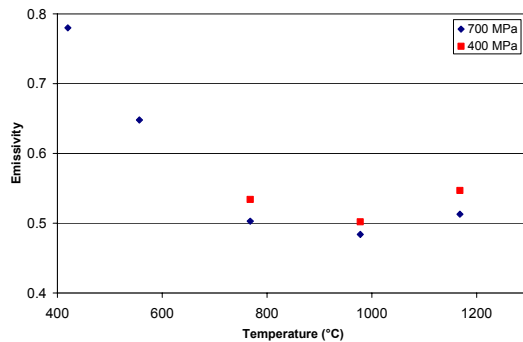


Figure 3: Emissivity for Distaloy AE-steel powder compacts obtained by conventional heating of tensile test bars compacted with 400 MPa or 700 MPa.

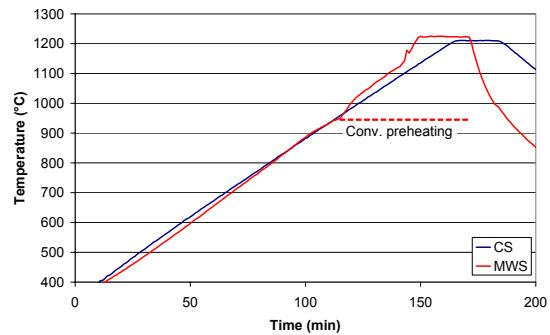


Figure 4: Comparison of the time-temperature profiles for Distaloy AE sintered in conventional and microwave processes. The representative samples were sintered at 1211 °C (CS) and at 1225 °C (MWS), respectively.

For Distaloy AE-steel powder compacts pressed with 700 MPa, ϵ is about 0.5 for temperatures above 750 °C. This is in good agreement with the published data for other alloyed steel with non-oxidized surfaces [4, 5].

The difference between the emissivity of the green body and the sintered compact is also dependent on the green body density. For a sample compacted with 400 MPa, the value of ϵ is slightly higher than for a sample pressed with 700 MPa. This is understandable because the surface porosity of the partial compacted samples is much higher than for the samples pressed with 700 MPa. The reflectivity is lower and the emissivity increases.

Figure 4 compares the time-temperature curves for conventional and microwave sintering. The samples in the microwave sintering experiment are insulated by a casket made of alumina fiber ceramic. This results in small differences for the heating and for the cooling rate. The dotted line indicates the temperature used for the additional conventional heating in the microwave sintering process (950°C). Heating above this temperature is only done by microwave radiation. With the intensity of the MW radiation the temperature was increased up to sintering temperature and hold for 20 minutes. The typical microwave density is about 0.02-0.03 W/cm³. Experiments with higher field strengths are difficult, because the generation of plasma cannot be avoided.

For samples which were sintered by microwave assisted heating without ceramic casketing, the maximum rise in temperature by microwave radiation is about 10 to 20 °C for sample temperatures above 600 °C. For microwave field strengths above 0.08 W/cm³ the generation of plasmas occurred. For temperatures below 600°C heating with microwave radiation is not detectable. The use of a fiber alumina casket shows no significant effect at these low temperatures.

3.2 Mechanical properties

The density of the sintered Distaloy AE-steel samples is varied in the range of 7.0-7.1 g/cm³. These values are independent for the sintering method (conventional and microwave assisted heating).

The tensile strength and the ultimate strain of the samples prepared at different sintering temperatures are given in figure 5 and 6 respectively. The mechanical properties show no significant difference between the conventionally and microwave sintered samples at temperatures above 1050°C. Below these temperatures, the microwave sintered samples show better ductility and higher tensile strength. For the sintering with the aid of the microwave radiation, the scattering of the data is much larger than for the conventional sintered ones. We think that the reason for this may be the non-uniform heat distribution of the sintering material.

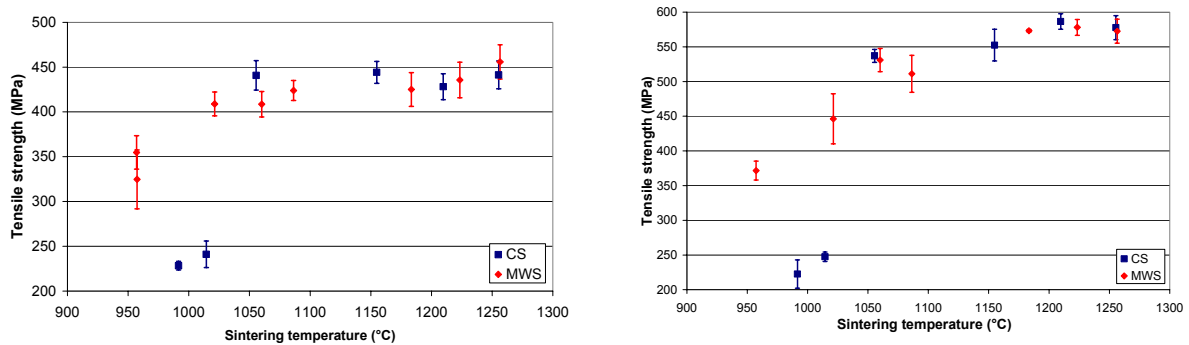


Figure 5: Effect of varying sintering temperature on the tensile strength of Distaloy AE +0.2 %C (left) and +0.5 % C (right) steel powders compacted with 500 MPa. The samples were sintered conventionally (CS) and with microwave radiation (MWS).

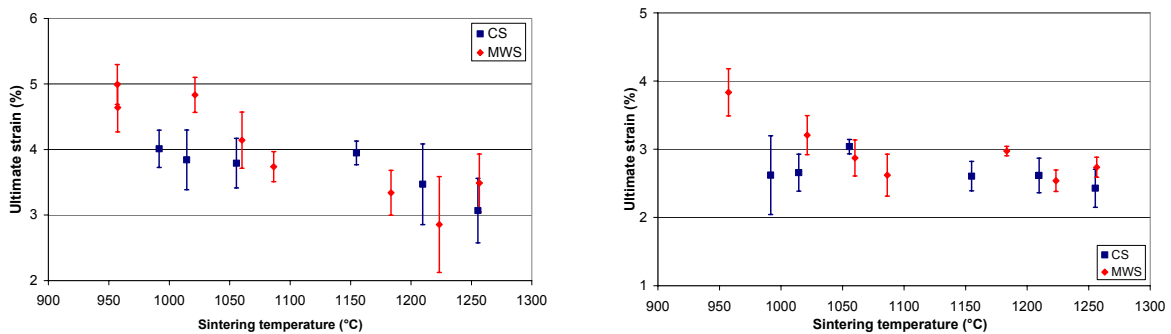


Figure 6: Effect of varying sintering temperature on the ultimate strain of Distaloy AE + 0.2% C (left) and 0.5 % C (right) steel powders compacted with 500 MPa. The samples were sintered conventionally (CS) and with microwave radiation (MWS).

The mechanical properties also depend on the field strength of microwave radiation. These can be varied by the conventional preheating temperature of the molybdenum resonator and thus of the samples. A lower preheating temperature required a higher microwave intensity to get the same temperature. Table 1 shows representative values for two different temperatures according to figures 3 and 4.

Table 1:

Mechanical properties of Distaloy AE + 0.2 % C steel powders compacted with 500 MPa depending on the conventional preheating temperature while microwave assisted sintering in hybride technique.

Sintering temperature (°C)	Preheating temperature of the samples (°C)	Tensile strength (MPa)	Ultimate strain (%)
957	700	354(19)	5.0(3)
957	850	324(33)	4.6(3)

4. Conclusion

The microwave assisted sintering of metallic materials is still an interesting field of materials processing. But the mechanism behind the microwave absorbing nature of metal powder compacts with good electrical conductivity is still unsolved. Additionally the influence of the ceramic casketing has not been understood. We think that a significant contribution of the microwave heating originates from additional heating of the casket. At higher temperatures alumina, especially fiber ceramics with an amount of silica act as a susceptor. The temperature of an empty casket rise slightly while placed in a microwave field. The absorption of microwaves by the alumina casket may be higher then the absorption in the metallic compact. Up to now, the ratio of the specific contributions to the heating of the sample (microwave absorption, inductive heating, heating by susceptors (e.g. casket), ...) are unknown.

Nevertheless, there is a measurable influence of the microwave radiation to the mechanical properties at low temperatures. Our metallographic investigations show no differences in the microstructure between both methods. It is assumed in [3], that the pore size and pore surface distribution measured by Hg-porosimety is different for microwave and conventional sintering of P/M steel. These investigations show that microwave sintered samples have less and smaller pores, possibly originating in the effect of the electromagnetic field. Other authors found smaller and rounder pores with a more uniform distribution compared with conventionally sintered samples [6]. This may be an explanation for the observed results. Further work is still in progress to answer this question. It seems that the effect is not conserved in the completely sintered state. The “activating” effect of the radiation has no permanent effect on the mechanical properties after the sintering at recommended sintering temperature for Distaloy AE (1120°C) was performed. A significant reduction of the optimal sintering temperature by microwave assisted heating is also not detectable.

The summary of these results shows that the microwave sintering of metallic materials needs further investigations. In future we will complete this work with other P/M steel powders. First results show, that the sintering of these powers is more influenced by the microwave radiation.

5. Literature

- [1] R. Roy, D.K. Agrawal, S. Gedevarishvili, J. Cheng, *Nature* 399(17), 1999, 668
- [2] M. Willert-Porada, H.-S. Park, *Ceram. Trans.* 111, 2001, 459
- [3] M. Willert-Porada, H.-S. Park, F. Petzoldt, B. Scholz, *Pulvermetallurgie in Wissenschaft und Praxis*, H. Kolaska (Hrsg.) Bd. 18, 2002, 103-133
- [4] *Thermal Radiation Properties Survey*, Honeywell Research Center
- [5] H. Oertel, W. Bauer, *Gaswärme International* 48(4/5), 1999, 282
- [6] R.M. Anklekar, D. K. Agrawal, R. Roy, *Powder Metallurgy* 44(4), 2001, 355