MICROWAVE SINTERING OF METAL POWDER COMPACTS

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Microwave sintering technology has been developed in the field of ceramics and composite materials in the last decades, because microwaves can penetrate into the entire sample body realizing instantaneous volumetric heating. It allows more rapid heating in comparison to the conventional heating process and in general reveals an enhanced sintering kinetics. This allows a reduction of process temperature and/or processing time and therefore an increased productivity and a reduction of energy consumption. The enhanced sintering is believed to be due to a non-thermal microwave effect caused by material interaction with the microwave electro-magnetic field. Microwave sintering of metal powders is a new activity with growing interest since R. Roy, D. Agrawal and co-workers at Penn State University published surprising results[(1)] some years ago. It is well known that metals reflect microwaves rather than absorbing them. Therefore volumetric heating could not be expected. Nevertheless it has been demonstrated that for sintering of metal powder compacts microwave technology can be beneficial as well.

At the Forschungszentrum Karlsruhe experimental investigations on sintering of Cu-powder compacts in nitrogen atmosphere have been performed in a compact gyrotron system operating at 30 GHz with a maximum power level of 15 kW. This system can be equipped with a dilatometer set-up, which allows in-situ measurement of the linear shrinkage of the powder compact during the sintering process. This information can be used to optimize the process parameters as well. In parallel, measurements of temperature gradients within the powder compacts were performed by use of two shielded type S thermocouples. One was fixed at the sample surface and the second put into a hole drilled into the green body. A comparison of these two temperatures allows distinguishing between volumetric heating and surface heating. Bar like samples 40 mm x 10 mm x 4 - 6 mm and 10mm x 5 mm angle in size with different green densities in the range from 40% to 85% of the theoretical density 8.93g/cm^3 and 80 mm x 20 mm x 5 mm with green density 65% were prepared by uni-axial pressing of copper powder type PC100 from Fukuda Metal Foil Powder Co., Ltd., Japan.

These samples have been placed into a box of mullite fiber boards for thermal insulation and sintered in N_2 atmosphere. The temperatures of the samples were reaching 1000 °C within 10 minutes and were held at this temperature for 10 minutes. In case of low green density and temperatures below 400 °C, the temperature inside of the sample was found to be higher than at the sample surface. This demonstrates that microwave can permeated even into a metallic powder sample and indicates that there is not sufficient metallic contact between the metal particles possibly due to oxide layers on the particle surfaces. So microwave shielding eddy currents are suppressed to some extend. On the other hand, in case of high green density, the temperature on the sample surface was higher than inside the sample volume during all the heating cycle. The maximal temperature gradient with heating rates of 100 °C/min. reached values of about 300 °C on a distance of a few millimeters only. This phenomenon appears, because the microwave is shielded by the skin effect and cannot penetrate into the sample volume. These experimental results show the dependence of the penetration depth of microwave on the density of a sample.

To check if there exists a correlation between the changes in temperature gradients, as they have been observed with samples of lower green density, and the sintering kinetics, the linear shrinkage was measured with a dilatometer setup. The samples were heated with a heating rate of 100 K/min. and 30 K/min. up to the sintering temperature of 1000 °C followed by 10 min. isothermal dwelling. The shrinkage was found to start in the range of about 400 °C. When sintering starts, oxygen layers at the grain surfaces are expected to disappear and mechanical as well as metallic contact between the grains improve by neck formation and subsequent sintering. But this increasingly suppresses microwave penetration, causing the observed change of the sign in temperature gradients.

Finally the tensile strength was measured with copper samples sintered at 950°C by microwave and conventionally, and sintered at 1050°C by the conventional method, respectively. The microwave method used N_2 atmosphere and the conventional method used an atmosphere of H_2+N_2 gas or N_2 gas. The sintered sample size was 70×18×4.5 mm^3. The tensile strength of the samples sintered at 950 °C by microwaves is 174 N/mm^2. This value is similar to the value obtained by the conventional method in N_2 gas at 1050 °C. In addition, it is similar to the sample sintered at 950 °C in N_2+H_2. There is little difference between the microwave method in N_2 and the conventional method at 1050 °C in H_2+N_2. As a result, the millimeter wave method allows a reduction of sintering temperature of about 100°C in comparison to the conventional method.

REFERENCES

1. R. Roy, D. Agrawal, J. Cheng, and S. Gedevanishvili, "Full Sintering of Powdered-Metal Bodies in a Microwave Field," *Nature*, Vol. 399, p.p.668-670 (1999).