In Situ Observations of Sintering Process during Pulsed Current Sintering of Al_2O_3, ZnO and WC ALLOY

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Abstract

Pulsed Current Sintering (PCS) process possesses some problems that need to be resolved. We, therefore aims at understanding phenomena of PCS process by presenting some basic data on in situ sintering behavior of PCS. Special graphite mold equipped with thermo couple and electrodes were designed to measure the temperature, electric current and voltage inside the powder during PCS process. We apply three types of raw materials, especially for ZnO as semiconductor, Al_2O_3 as non-conductor and WC as good conductor. The electric current and voltage were measured for each powder during PCS process. In addition, their electric resistance properties were calculated.

Keywords : powder metallurgy, electric current-voltage, electric resistance, PCS, plasma

1. Introduction

Pulsed Current Sintering (PCS) process was developed recently. It can sinter materials at relatively low temperature and within a short time. PCS machines are widely used by many academic and research institutions, and private companies that are engaged in research and development activities on materials. However, there are some fundamental problems that need to be resolved. One of those problems is that the reason of rapid sintering occurs at a lower temperature is not well understood. Another problem is that the temperature of the sintering powder is different from that of mold, which is commonly used to control sintering process. There have been some attempts to identify the fundamental phenomena and tried to explain what occurs inside the sintering mold set. There are also some researches that were conducted to find out whether sintering plasma is generated or not. [1,2,3] In this and future study, we are main interested in understanding the mechanism behind PCS and the reasons why rapid sintering occurs at relatively low temperature. For its first step, in this paper, we investigate these fundamental phenomena of PCS process by using electrical measurement technique

2. Experimental and Results

In order to investigate fundamental phenomena of PCS process, we prepared three types of raw material powders. The first was ZnO thermoelectric material as a semiconductor material, the second was Al_2O_3 oxide ceramic material as a nonconductor material and the third was WC carbide ceramic material as a good conduct material. Those powders were filled into graphite mold sets, and sintered by PCS machine (SPS Syntex Inc. SPS-3.20MK-4). All PCS sinters were performed in vacuum (<10Pa) atmosphere for 10min. Sintering temperatures differed with each sample material (ZnO: 1423K, Al_2O_3:1473K, WC: 1673K). For ZnO thermoelectric material, we performed sintering test by ordinary electric furnace in air atmosphere in order to compare ordinary sintering with PCS.

Electric circuit diagram of the measurement system is shown in Fig.1. Pt electrodes were used and the electrode gap was 20 mm. Electrical voltages V_1 was measured as a voltage, and electrical current was measured by V_2. Then the electric resistance was calculated. R type thermocouple (R_TC) was also inserted 5mm into the mold set. Sintering temperature was measured and controlled by K type thermocouple (K_TC) for ZnO and Al_2O_3 or Infrared radiation thermometer for WC.

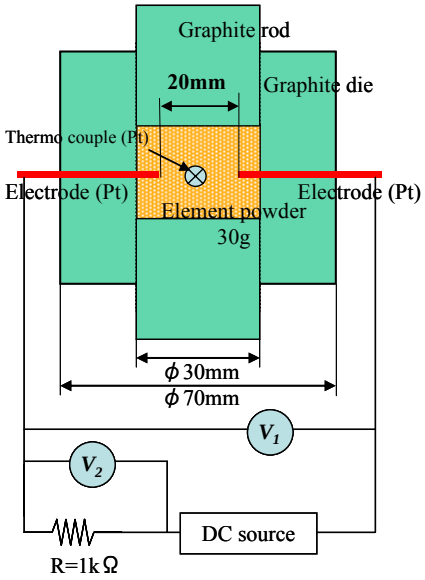

Fig. 1. Schematic diagram of sintering mold set and electric resistance measurement circuit.

Electric resistances and sintering temperature of Al_2O_3 were observed. Temperature change inside Al_2O_3 powder showed a similar tendency with outer mold temperature. However, at sintering temperature, a temperature of inside Al_2O_3 was about 100 degree higher than that of mold surface. At heating process, electric resistance decreased as sintering temperature rose. At cooling process, electric resistance rose and got back to original value.

Electric resistance change during PCS of WC was observed. As WC is a good electric conducting material, the electric resistance remained unchanged during the sintering process.

Electric resistances and sintering temperature of ZnO were observed both by PCS and by electric furnace. At a sintering temperature of PCS, a temperature of inside ZnO (R_TC) was about 170 degree higher than that of mold surface (K_TC). As a raw material powder, the electric resistance of ZnO was high about orders of 10^6 Ω and acted like a non-conductor. When sintering begins, the electric resistance decreased rapidly to orders of 10^4 Ω and acted like a good conductor. The electric resistance at 850K (R_TC) heating process was less than 10^1Ω. For PCS specimen, the low resistance was maintained throughout the entire sintering process and finally a low electric resistance sample was obtained. On the other hand, for electric furnace sintered specimen, its electrical resistance decreased at heating process until 750K, but over 750K, electric resistance started to increase and at 835K electric resistance decreased again. The electric resistance increased during the cooling process, and then it got back to original value of electric resistance finally.

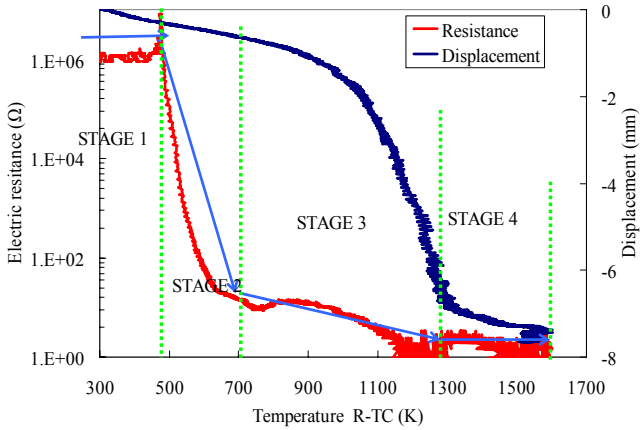

Fig. 2. Four stages of resistance change ZnO by PCS.

The sintering behavior of ZnO by PCS was thoroughly analyzed, and then four sintering stages as a relation of electric resistance change were recognized. Those sintering stages and displacement change are shown in Fig. 2. There was no change observed in Stage 1 (RT-500K). In Stage 2 (500-700K): The electric resistance decreased rapidly. In Stage 3 (700-1300K): The electric resistance decreased gradually and rapid shrinkage occurred. Lastly, in Stage 4 (1300-1600K), the electric resistance and displacement remained constant.

3. Summary

To get a better understanding how the mechanism of PCS process, we can develop an electrical measurement method to investigated the fundamental phenomena of PCS process. By using this method, we successfully investigated the temperature inside the sintering powder and the electric resistance property of non-conductor (Al_2O_3), good conductor (WC) and semiconductor material (ZnO). We thoroughly analyzed PCS process of ZnO and recognized four stages of electrical resistance change. We cannot find out obvious evidence of plasma generation.

4. References

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