Calibration of Temperature and Pressure in the Electroconsolidation Process

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The electroconsolidation (EC) process is a proprietary method (Superior Graphite Co.) for pressure assisted densification of powder preforms. Granular graphite is both the pressure-transmitting and heating medium as direct current is passed through. The present research was conducted to determine the temperature and the pressure distribution in the die. Alumina pellets and slices of Ni-C eutectic pellets were used as probes. The temperature distribution in the die was inferred by observing the melting behavior of the Ni-C eutectic slices using an X-ray imaging system. Then the pressure-assisted master sintering surface was used to infer the pressure distribution, given the temperature distribution.

Keywords: *electroconsolidation, densification, temperature distribution*

1. Introduction

Electroconsolidation is a process for densifying complex shaped parts using electrically conductive graphite particles as a pressure transmitting medium. The schematic of the process is shown in Figure 1. The preform surrounded by the pressure transmitting medium within graphite die. The graphite medium is a materials to take advantage of rapid heating, greater than 1000℃ per minute and heats the preforms up to 2500℃.

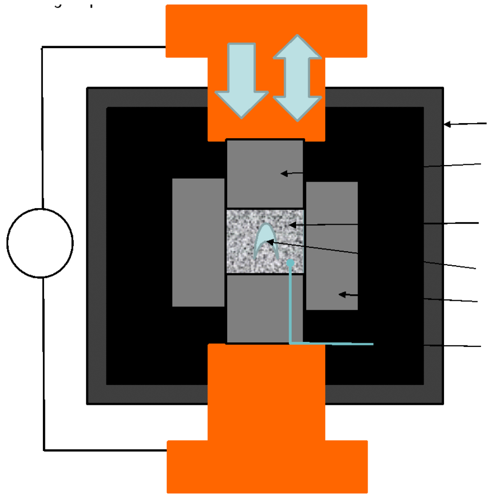

Figure 1 Schematics of Electroconsolidation process

However, a certain degree of uncertainty in densification behavior of preforms in this process was raised due to difficulties in determination of exact temperature and presser throughout the cavity of die and punches assembly. Correct measurement of temperature is one of the primary issues to determine densification behavior during Electroconsolidation(EC) process. Calibration of temperature in EC can be carried out with well-known melting point. Then, pressure distribution inside the cavity could be determined from the profiles of density and temperature inside cavity.

2. Experimental procedure

Nine pieces of Ni-C alloy as temperature probes having 1318℃ melting point and twelve alumina pellets as density probes were installed in graphite die cavity (76mm in diameter and 76mm in height) of EC system as shown in Figure 2. Ni-C alloy pieces were placed at 3 different height levels such as 19, 0 and -19mm and 3 different radial direction such as 19, 0 and -19mm. Twelve alumina pellets (9.5mm in diameter and 2mm in thickness) were used to determine final densities at the same height levels of Ni-C alloy as well as 2 additional levels between them.

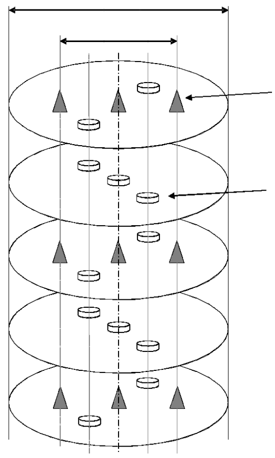

Figure 2 Locations of temperature and density probes in EC (76 x 76mm cavity)

Pressure of 13.8Mpa was applied at room temperature and the furnace was heated up to 1200℃. At the temperature point of 1200℃, electric power was shut down to take x-ray image and then the furnace was reheated up to 1250℃ (Figure 3). The same procedure was conducted with increment of 50℃ up to 1300℃, followed by increment of 25℃ up to 1400℃. At each temperature step where the electric power shut down, x-ray images was taken to observe the melting point of Ni-C alloy.

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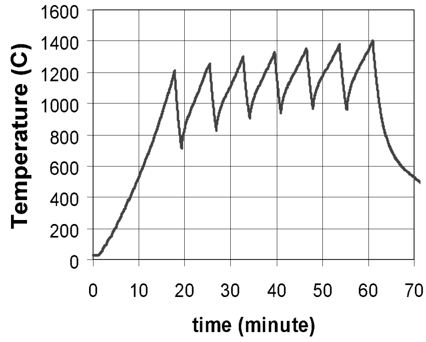


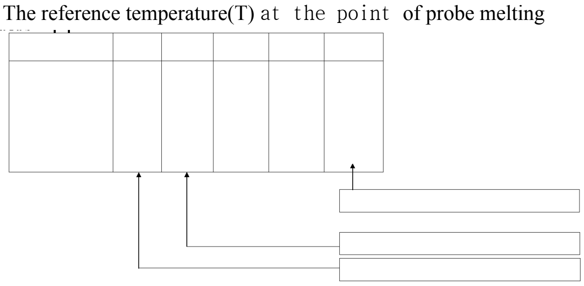
Figure 3 Heating steps in EC process

The final densities of alumina pellets after EC process was determined by the Archimedes method.

3. Results and discussion

Table 1 shows the temperatures measured by a thermocouple installed at the cavity bottom, and the melted Ni-C alloy pieces from x-ray image photos taken at each step of power shut down.

Table 1 The reference temperature(T) at the point of probe melting



At 1318℃ eutectic point of Ni-C alloy, no melt was observed.

At 1326℃ which is 8℃ over eutectic point, the first melt piece was observed at the center of bottom layer. It suggests that Ni-C alloy melted at right over its melting point of 1318℃. Certain temperature gradient was observed vertically and radially because the other pellets were clearly seen as a solid shape.

At 1352℃, additional 2 pieces placed at the above two center levels were melted, which can be considered 25℃ lower at the area of those pieces placed than at the bottom center.

In the same manner, pieces at the bottom sides and the top sides were melted in sequence. Therefore, the temperature gradient was found to be existed in the cavity from the hottest point at the bottom center to the coldest at the die wall as shown in Figure 3.

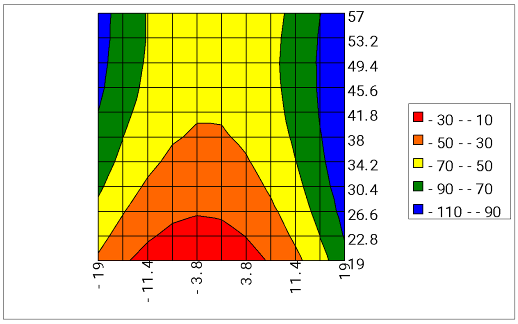


Figure 3 Temperature drop profile in the cavity of EC

Figure 4 shows densification curve during hot pressing of alumina at13.8MPa. The master sintering curve was used to predict the sintered density, in which density is a unique function of the integral of a temperature function over time.

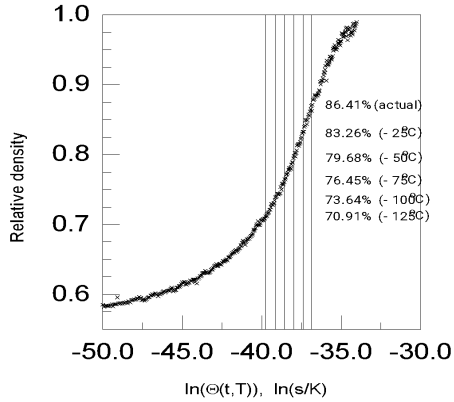


Figure 4 Densification curve of alumina at 13.8MPa

From the master sintering curve, one can predict the pressure if the density and temperature of a specimen are known. The final densities of pellets at varying location were determined by Archimedes method and their corresponding temperature drop are shown in Figure 3.

Figure 5 shows the profile of the effective pressure predicted from the temperature of Figure 3 and density of the pellets in Figure 4 using master sintering curve.

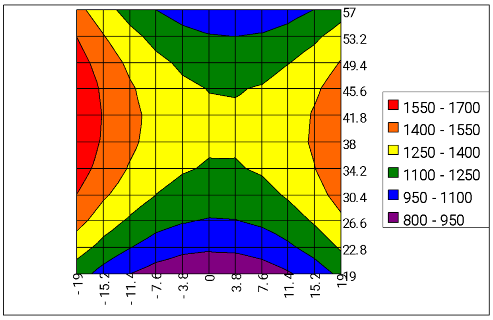


Figure 5 Profile of effective pressure in cavity of EC

The temperature and pressure drops resulted in density drop in sequence. The effective pressure might come from the friction between the granular medium and the die wall, as well as the frictional behavior of the granular medium.

4. Conclusions

The temperature distribution in the cavity of EC can be inferred by observing the melting behavior of Ni-C eutectic pellet slices, using X-ray imaging system.

The pressure-assisted master sintering surface can be used to infer pressure distribution, given the temperature distribution.

The applied pressure on specimen could be diminished due to the friction between the granular medium and the die wall, as well as the frictional behavior of the granular medium.

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