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Sintering Behavior of Aluminum Powder by Spark Plasma Sintering

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Aluminum powders were sintered by spark plasma sintering (SPS) process. The densification effect in the sintering process and the properties of powder compacts were investigated, and the microstructure was observed. The results showed that aluminum powders were abruptly densified by SPS process at sintering temperature of about 573 K. Increasing in sintering temperature or loading pressure, relative density and tensile strength of powder compacts increased, and the electrical resistivity decreased. At sintering temperature of above 873 K, loading pressure of over 23.5 MPa, main bonding mechanism of the powder compacts was considered to be metal-metal contact, and the properties of compacts were similar to those of base aluminum metal. The difference existed in bonding interfaces among powder particles for compacts by various sintering conditions. The better bonding could be obtained for the compacts with large loading pressure or at high sintering temperature.
Key words: spark plasma sintering, densification, electrical resistivity, tensile strength, microstructure

1. INTRODUCTION

Spark plasma sintering (SPS), as a newly developed process, is regarded as a rapid sintering method. In SPS process, pulse electric current flows directly in the sintered materials. The specimens are sintered by the Joule heating generated in the sintered materials and by heating transfer from the graphite dies and punches. A very high heating efficiency is offered. It can easily consolidate a high quality compact that is sintered at a lower sintering temperature and in a shorter time than the conventional sintering methods such as hot pressing (HP) and hot isostatic pressing (HIP), even to those materials that are very difficult to be sintered by the conventional processes [1-6].

On the other hand, Al powder is difficult to be sintered by normal and hot pressing, because the surface on Al powder particles is covered with the tenacious oxide film that inhibits metal-to-metal contact, and the oxide film cannot be broken and/or removed by heating [7,8]. SPS can partially eliminate the oxide films and the adsorbed gases, and activate the surface of powder particle [9]. It makes the sintering of Al powder to be easily. Recently, it has been reported that aluminum alloys and its compounds powders were sintered by SPS process [4, 10-12]. Since the investigation of the sintering mechanism in the SPS process is difficult, up to now, it is still unclear about the densification and cleaning effects in the sintering process.

In the present study, aluminum powders were sintered by means of SPS process. The densification effect in the sintering process was analyzed. The effects of sintering parameters on mechanical properties, electrical resistivity and microstructure of powder compacts were investigated.

2. EXPERIMENTAL METHODS

Commercial grade Al powders were used in this study. The powder particle sizes and distribution are as follows: below 44 μm, 42.5%; 44~62 μm, 28.1%; 62~74 μm, 14.0%; 74~104 μm, 15.4%; and the chemical compositions are: Al, 99.798 mass%; Si, 0.060 mass%; Fe, 0.140 mass%; Cu, 0.002 mass%. The sintering was carried out in a vacuum using SPS system (SPS-520 model; Izumi Technology Company, Ltd.). Figure 1 shows a schematic diagram of the system, and Fig. 2 gives the temperature and loading pressure control model of the SPS process. The pulse frequency of 40 kHz was used in the sintering process. The uniaxial pressure model was conducted using the top and bottom graphite punches. The powder compacts obtained by SPS process were the tensile specimen, with length of 20 mm and width of 5 mm at the parallel part.

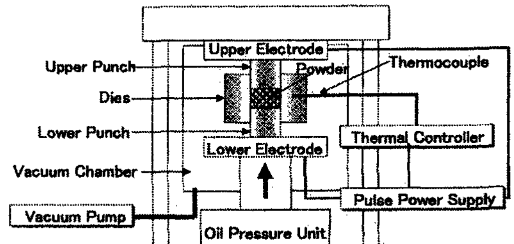

Fig. 1 Schematic diagram of SPS apparatus.

Density of the powder compacts was determined by measuring the weight and size. The electrical resistivity was obtained using four points probe method by measurement of the voltage between two probes of the specimen when the compact flows the certain direct current. Tensile test of the powder compacts was carried out by means of AG-250KNG autograph tester. The tensile velocity of 2 mm/min was used.

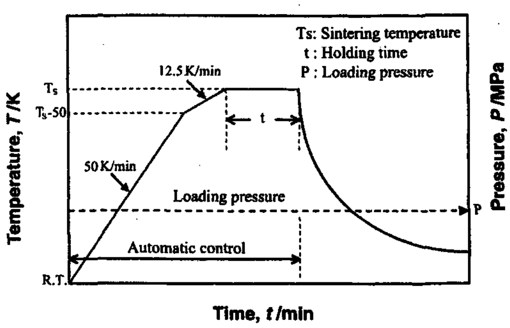

Fig. 2 Temperature and loading pressure control model of the SPS process.

Microstructure of powder compacts was carried out using a JSM-6400 scanning microscope. The specimens were cut out from the compacts by a diamond saw, and polished to be mirror plane by emery paper and buff polishing, then corroded with a mixing solution of nitric acid (1.38) 5 ml, hydrochloric acid (1.19) 3 ml, fluorhydric acid (40%) 2 ml, and distilled water 190 ml.

3. RESULTS AND DISCUSSION

3.1 Densification of sintering process

Figure 3 shows the apparent relative density and linear shrinkage of the sintered powders as a function of sintering temperature and holding time at a loading pressure of 23.5 MPa. The linear shrinkage of the sintered powders is equal to the measured Z-axis displacement after deducted the displacement of the graphite punches in the sintering process. The apparent relative density was determined by the linear shrinkage of the sintered powders. As the comparing, the real relative densities of specimens sintered at several temperatures were also shown in Fig. 3. It can be seen that the apparent relative density increases abruptly when the sintering temperature is higher than 573 K, and then increases slowly at sintering temperature above 673 K. The tendency of change on the real density shows good consistency with the change of apparent relative density. It indicates that the observation of linear shrinkage is very useful to understand the consolidation behavior of powder compacts during SPS process.

Figure 4 shows the changes of the densification rate as a function of sintering temperature at various heating rates. Densification rate was determined by calculating the slope of the linear shrinkage curve (taken off the displacement of the graphite punches) of the sintered Al powders in the sintering process. It can be seen that the densification rates are different according to each heating rate, but the maximum of densification rate for each heating rate is observed at the similar sintering temperature (near 573 K). It is suggested that the maximum in the densification rate curve should be related to the neck formation in the powder compacts. Kim et al. showed the similar results from SPS process of Fe and Ni powders [13, 14].

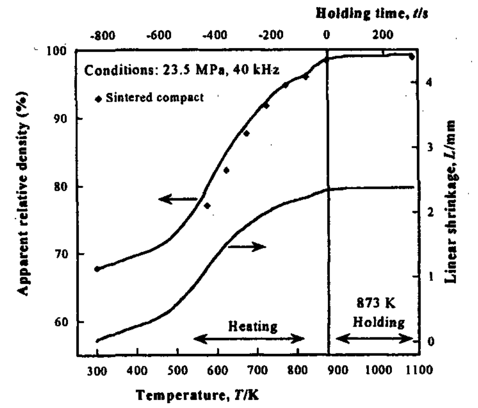

Fig. 3 Relationship between relative density, linear shrinkage of specimen and temperature, holding time.

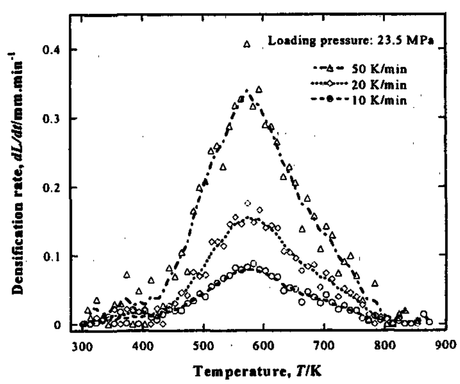

Fig. 4 Change of densification rate with temperature at various heating rate.

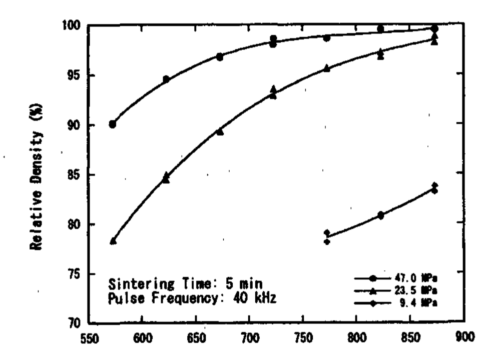

Fig. 5 Effect of sintering temperature and loading pressure on relative density of Al powder compacts.

Figure 5 shows the effect of sintering temperature on relative densities of Al powder compacts with various loading pressures for holding time of 5 min. Increasing in sintering temperature at the same loading pressure, or increasing in loading pressure at the same temperature, the relative densities of powder compacts increase. The relative density of compacts is above 97% when the temperature is above 823 K and the loading pressure is over 23.5 MPa.

3.2 Electrical resistivity and mechanical properties of powder compacts

Figure 6 shows the relationship between electrical resistivity of powder compacts and sintering temperature with various loading pressure, and Fig. 7 shows the change of tensile strength of compacts with sintering temperature and loading pressure for holding time of 5 min. Increasing in sintering temperature, or increasing in loading pressure, the electrical resistivity of powder compacts decreases, and the tensile strength increases. It can be seen that at loading pressure of 23.5 MPa with temperature of 873 K, or 47.0 MPa with over 823 K, the tensile strength and the electrical resistivity of powder compacts are similar to those of base aluminum metal. Figure 8 shows the change of elongation of powder compacts with sintering temperature and loading pressure. It can be seen that the elongation of compacts increases with increasing in sintering temperature or in loading pressure. When the sintering temperature is above 873 K, and the loading pressure is over 23.5 MPa, the elongation of compacts is similar to that of the base aluminum metal. The reduction of area is also shown the same result as the elongation of powder compact. Therefore, we can determine that pure Al powder compacts with the similar properties as base aluminum metal can be obtained by SPS process with the sintering temperature of above 873 K, loading pressure of above 23.5 MPa.

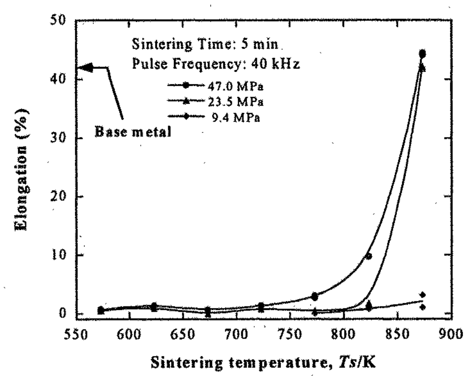

Fig. 8 Effect of sintering temperature and loading pressure on elongation of Al powder compacts.

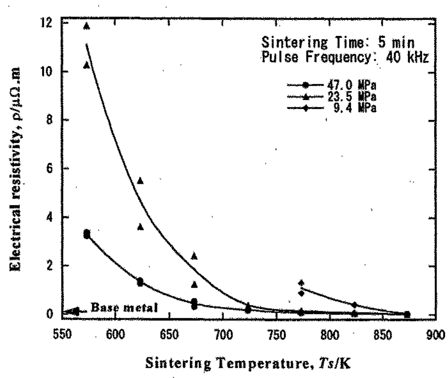

Fig. 6 Effect of sintering temperature and loading pressure on electrical resistivity of Al powder compacts.

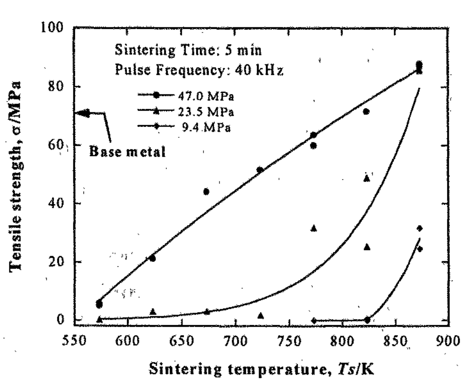

Fig. 7 Effect of sintering temperature and loading pressure on tensile strength of Al powder compacts.

3.3 Microstructure of powder compacts

Figure 9 shows scanning electron microscopy (SEM) observation results of the compacts using several typical sintering conditions by SPS process for holding time of 5 min. Figure 9(a), (b), (c) and (d) give the results of the compacts by 623 K, 23.5 MPa; 873 K, 9.4 MPa; 623 K, 47.0 MPa and 773 K, 23.5 MPa, respectively.

For the compacts shown in the Fig. 9(a) and (b), there are the similar relative density, namely, there are the similar contact areas among powder particles, but the large difference in their electrical resistivity and in the tensile strength exists (as shown in Fig. 6 and Fig. 7). It can be seen that there is no obvious difference in the pore density and the particles size, while the difference in microstructure is obvious. It shows the better bonding for the powder compacts with the low electrical resistivity and the high tensile strength. Therefore, it is suggested that the cause of the difference in properties for powder compacts are mainly subject to the bonding behavior at the interface between Al powder particles.

Comparing between Fig. 9(a) and (c), or Fig. 9(a) and (d), it can be observed that the powder compacts with higher loading pressure at the same sintering temperature, or those with higher sintering temperature at the same loading pressure, can obtain the better bonding in which the disappearance partially of grain boundaries is observed, and the metal-to-metal contact is shown. The powder compacts are provided with the large tensile strength and the low electrical resistivity.

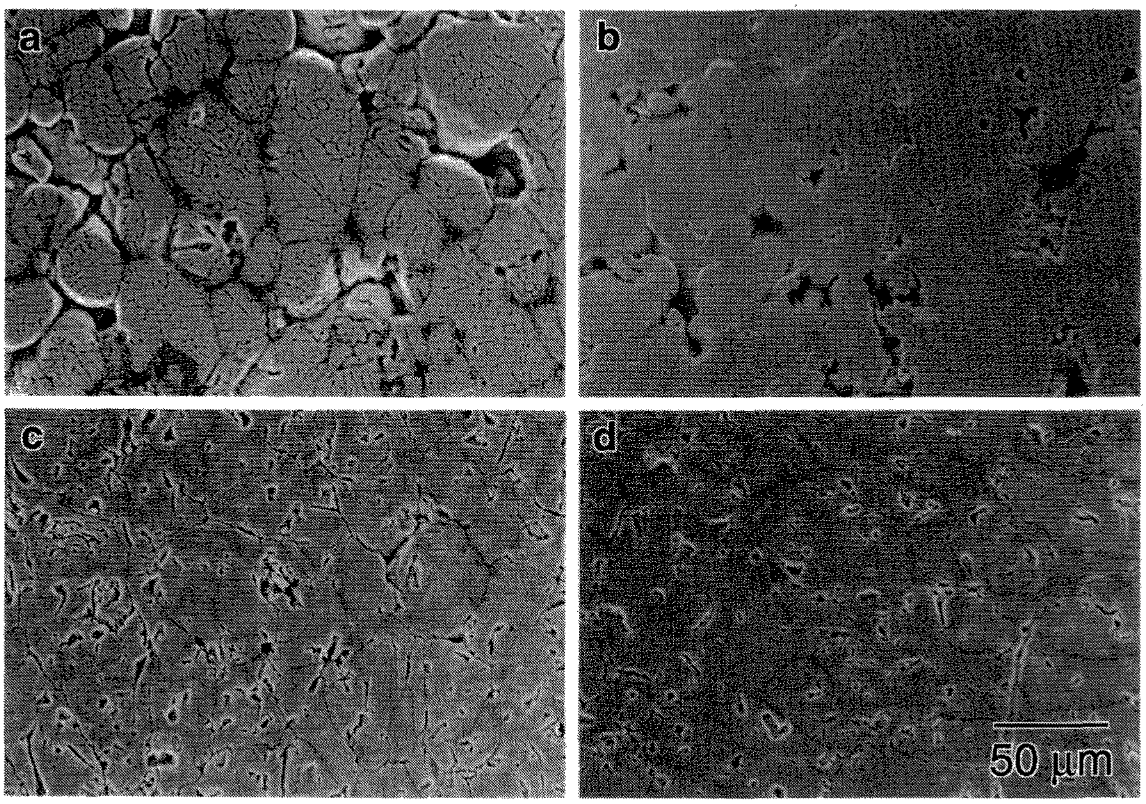

Fig. 9 Scanning electron microscopy (SEM) images of Al powder compacts for the typical conditions by SPS process. (a) 623 K, 23.5 MPa; (b) 873 K, 9.4 MPa; (c) 623 K, 47.0 MPa; (d) 773 K, 23.5 MPa.

4. CONCLUSIONS

Aluminum powders were sintered by SPS process. The densification effect in the sintering process and the properties of powder compacts were investigated, and the microstructure was observed. The results obtained are summarized as follows.

(1) The difference existed in bonding interfaces among powder particles for compacts by various sintering conditions. The better bonding could be obtained for the compacts with large loading pressure or at high sintering temperature.

(2) Increasing in sintering temperature or loading pressure, relative density and tensile strength of powder compacts increased, and the electrical resistivity decreased. At SPS process conditions above 873 K, above 23.5 MPa, the metal-to-metal bonding was mainly in the interface among powder particles, and the properties of compacts were similar to those of base aluminum metal.

(3) Aluminum powders were abruptly densified by SPS process at sintering temperature of about 573 K.

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