Fabrication of a Dense Long Rod through Pulse Discharge Sintering Assisted by Traveling Zone Heating*

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A traveling zone heating technique was examined with the aim of creating a long dense rod by means of pressurized pulse discharge sintering. Based on local heating enabled by electric power supplied perpendicular to the loading axis through a terminal board, a one-direction continuous sintering process was successfully achieved, in which the terminal board was able to slide along the side wall of the cylinder in the direction of the loading axis while remaining in continuous contact with it. The heating zone was limited to within the range corresponding to the thickness of the board. Aluminum powder of 9.54 g, 99.9% in purity with an average size of 20 μm was placed in a graphite cylinder of inner diameter 15 mm. Electric power was then supplied through a 12 mm thick terminal board to sinter the powder. Power supply was provided to the board, which was moved upwards three times by a distance of 8 mm. A 20 mm long rod with a relative density of 99.2% was successfully sintered by means of this procedure. Similarly, four successive local heatings over a range 30 mm wide while moving the heating zone distances of 20 mm led to the production of a 55 mm long aluminum rod with a relative density of 99.7%. The present results suggest that much longer rods can be sintered well by expanding the moving range of the heating zone. The new sintering technique proposed in this study will enable the production of long rods with high density.

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1. Introduction

A pressurized sintering technique based on the direct supply of electricity to compacted material powder, called pulse discharge sintering, PDS, (also known as spark plasma sintering, SPS), has the great advantage of saving both manufacturing energy and time compared with both casting processes and conventional sintering methods such as hot press and hot isostatic pressing. Numerous efforts have been devoted to the application of the PDS process to the fabrication of machine components, especially engine parts that require high heat resistance to improve efficiency. For example, the application of titanium aluminide, an intermetallic compound, to engine valves has been studied. In the PDS process, however, there are problems with precise temperature control since this process supplies electricity simultaneously to all over the powder particle and the loading punches at once; these electric resistances are normally different. Heat loss to the surroundings, to cooled electrodes in particular is another problem which is aggravated when the electric current path within the product body gets longer, sometimes leading to uneven and unsatisfactory temperature distribution within the product body during sintering. Consequently, it may be very difficult to use the PDS process to produce high-density rod shape components such as engine valves and piston pins.

In order to apply the PDS process to the above rod-shaped products, the continuous zone sintering technique was examined. This is a form of one-directional solidification technique. A narrow heating zone is used so that the temperature range is held within the allowance. Electricity is supplied to a limited zone near one end of the powder material, and then the zone is moved along the axis. In this study, based on the above approach, we produced several rods from aluminum particles which appeared to be sintered at relatively low temperatures. The densities of the sintered rods were measured to evaluate their quality.

2. Experimental Procedure

2.1 Material and mold

The material used was 99.9% pure atomized aluminum powder with an average diameter of 20 μm.

Two types of graphite molds were set up for the present study. They are illustrated in Fig. 1. Mold A consists of a cylinder and a terminal board. The cylinder has dimensions of 30 mm and 15 mm as outer and inner diameter, respectively. A terminal board to which electric power is supplied was attached to a cylinder such that it was able to slide along the outer side wall of the cylinder in the direction of the loading axis while maintaining contact with it. The thickness of the terminal board employed was between 10.0 mm and 13.2 mm. The heating zone is defined as the range corresponding to the thickness of the board. Mold B, on the other hand, was a 100 mm long hollow pillar with a 40 mm square section with a hole 15 mm in diameter. Instead of a terminal board, spacers were used to transmit electricity to this mold. The contact area of the spacer with the pillar delineated the heating zone in this mold.

All pieces of mold were made from ISO-68 grade graphite distributed by Toyo Tanso Co., Ltd.

2.2 Sintering

Three variations of sintering experiments have been carried out in the present investigation. In the first experiment, 3.82 g of aluminum powder was placed in mold A and electric power was supplied to the powder compactor through the terminal board as shown in the heating diagram illustrated in Fig. 2. Five series using maximum temperatures of 853 K,
873 K, 893 K, 903 K and 913 K were examined. The thickness of the terminal board was 10.0 mm for the first two and 13.2 mm for the others. Temperature was monitored by a thermocouple inserted into a hole 7 mm deep drilled on the side wall of the cylinder through the terminal board.

The second experiment involved successive sintering by moving the heating zone. As shown in Fig. 3, 9.54 g of aluminum powder was placed into the same mold. A terminal board 12.0 mm thick was then attached to the initial position, setting the center of the board 72 mm from the bottom of the cylinder. Electric power was supplied three times to the powder compactor through the terminal board, which was moved sequentially at intervals of 8 mm. Temperature was monitored by a thermocouple, as in the previous experiment, but at a different depth to move a terminal board. It was inserted so as to just touch the sidewall of the cylinder. The same heating setup was used but the maximum temperature was set at 973 K. The details of the reasons for this will be described later.

Both the first and the second experiments were carried out under a load of 10 kN in an inert gas atmosphere under nitrogen gas flow.

The third experiment also comprised continuous sintering but was carried out using mold B to produce a longer sample. In this experiment, spacers that delineated the heating zone were substituted for a terminal board. The height of the spacers was 30 mm. Mold B filled with 26.0 g of aluminum powder was placed on the table and the table was set at the initial level so that the center of spacer met the level of 80 mm from the pillar bottom. Electric power supply was
repeated four times while elevating the pillar at intervals of 20 mm. In this experiment, the level of the table was changed so as to move the heating zone instead of moving the terminal board. In the fourth sintering experiment, the pillar was set upside down due to limited space in chamber. The same heating setup as in the previous experiment was employed but the maximum temperature was set at 923 K. A thermocouple was inserted into a hole 12 mm in depth drilled in the side wall of the pillar at the center level of the spacer. These sinterings were carried out under a load of 9 kN in a vacuum.

The properties of the sintered samples were evaluated by examining their density using Archimedes’ principle.

3. Result and Discussion

Before actual sintering, the effect was examined on the temperature distribution in the loading direction of the introduction of a terminal board to the sintering system. Temperatures were monitored at three points: the center level of a board and 10 mm or 20 mm above that point. The thickness of the board was 10 mm. The results are shown in Fig. 4. The highest temperature was attained at the center and decreased with increasing distance from that point. These results show that only a specific zone, corresponding to the thickness of a terminal board, can be successfully heated.

Figure 5 shows the change of relative density of sintered samples with sintering temperature in the first experiment. It is obvious that the density increased at higher sintering temperatures, reaching a ceiling of 99.4% when sintered at 913 K. This value may guarantee that the sintered body is successfully densified. That is to say, even if electric power is supplied perpendicularly to the loading axis assisted by a terminal board, the powder compact can be fully sintered.

In contrast to the above experiment, aluminum powder could not be densified sufficiently at 913 K in the second experiment, in which the insertion location of thermocouple was different. In the conventional PDS process, electricity is supplied through the punch; consequently the punches themselves generate heat, so most heat loss takes place through the sidewall of the mold. Accordingly, temperature increases towards the center of the rod. Therefore, setting temperature should be decreased when thermocouple is moved outside. In the present zone heating system, however, generation of heat occurs only within the range corresponding to the thickness of the terminal board, not in the punches. Consequently, more heat is likely to be lost in the loading direction through the punches rather than radially, with the result that temperature decreases towards the center of the rod. For the above reasons, the sintering temperature was finally set at 973 K, which appears to be equivalent to 913 K in the previous experiment.

Figure 6 indicates the shrinking curve in traveling-zone sintering carried out in the second experiment. The degrees of shrinkage were significantly different between the first and second sintering, as can be seen in the need for the use of different scales for each sintering in the figure. In the first sintering, shrinkage increased with rising temperature but leveled off at around 770 K, then accelerated again at nearly 920 K. On the other hand, some expansion was observed at the primary stage in the second sintering. This behavior was caused for the reason that the thermal expansion of the punches outweighed the shrinkage of the raw material powder. Aluminum powder, however, began shrinking rapidly when the temperature reached about 920 K. We conclude that this behavior was equivalent to the final densification in the first sintering. Similar shrinking behavior was also observed in the third sintering. The above results suggest that zone heating while moving the terminal board promotes the densification of parts of the powder compact.
that have not yet been densified in the previous process. A sintered aluminum rod 19.4 mm long with a relative density of 99.2% was successfully obtained by application of this procedure although some weight loss was caused due to polishing.

The third experiment was carried out aiming to produce a longer rod. This experiment was done under a similar procedure to the second experiment except for the number of times the terminal board was moved and the distance it was moved each time. In this experiment, shrinking behavior that was closely equivalent to the former experiment in that further shrinkage appeared even in the second and the later sintering was observed. This sintering resulted in the successful production of an aluminum rod 55.1 mm long with a relative density of 99.7% after polishing.

In the present investigation, a heating zone was manually moved in stepwise fashion. Real continuous automatic sintering of rods, however, will be enabled provided that the table possesses an up and down function. While the longest densified Al rod obtained in this study was 55.1 mm long, the potential length of this sintering process can be extended, since the moving range of the heating zone is not restricted except by space. Also, this advanced pulse discharge sintering process can be adapted to the variation in electrical resistance at separate parts of the product during sintering. This special feature can ensure equal temperature conditions over the entire product body even if it has an irregular cross section, provided that a sufficiently narrow band is employed as a heating zone. In other words, this new process is extremely effective for sintering not only long rods but also products with irregular section such as tapered pins.

4. Concluding Remarks

In order to provide a long dense rod through the pressurized pulse discharge sintering method, an advanced sintering system assisted by traveling zone heating was examined in present study. Our principal results are summarized as follows.

1) A local heating system was developed by supplying electric power perpendicularly to the loading axis. The heating zone could be moved in the direction of the loading axis assisted by the use of a terminal board. A one-direction successive sintering process was successfully established.

2) Our traveling zone sintering process has enabled the production of a highly densified aluminum rod 55 mm long with a relative density of 99.7%. It is highly likely that much longer components can be sintered using the PDS process providing the range of movement is extended.

3) The new technology developed in the present study makes possible precise temperature control of any part of the object. Establishing the appropriate heating zone size and moving rate brings the advantage of allowing even components having an irregular heating zone to be produced to a high level of density through the pulse discharge sintering process.

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REFERENCES