The Continuous Pressurized Sintering of a Metallic Powder Assisted by a Stepped Mold*

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A new industrial technique for the continuous pressurized sintering of metallic powders was proposed. A unique open mold with a step in its sintering room was employed in order to make the continuous production of sintered rods possible. This step added pressure to a powdered material in the mold, and the amount of pressure was controlled by a sectional reduction ratio at the step. Tin powder was extruded repetitiously into the mold, which was heated by a band heater attached near the step. It was confirmed that the above procedure successfully generated a stable pressure on the powdered material during sintering when it was fed into the mold. This sintering resulted in the production of a tin rod with a relative density of 99%. The longest sample produced in this study was about 70 mm, since in the case of longer samples the push rod was broken in the middle of the sintering by the friction due to the powder in the gap between the push rod and the mold. This failure must be avoided by improving the strength and precision of this system. It was indicated that the proposed sintering system has the potential for the continuous production of dense metallic rods.

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1. Introduction

Pressurized sintering under the direct supply of electric power, commonly called pulse discharge sintering, PDS, or spark plasma sintering, SPS, has many industrial advantages in both processing time and product density compared with conventional sintering methods with no pressurizing. Many studies have been conducted on this sintering process regarding the fabrication of machine components, particularly engine parts that require high heat resistance.

The productivity of this method, however, is not as high as other methods because this process requires powdered material to be placed in a mold for every sintering process. Pressurizing is another problem. The load transmission within the product body gets worse as it becomes longer due to an increase in the friction between the powder and the walls of the mold, resulting in an insufficient and uneven density at the deep section of the product.

In order to improve the quality of long products sintered using the PDS process, the authors proposed a traveling zone sintering method, in which electric power was supplied to a narrow zone perpendicular to the loading axis through a movable electrode. In this method, a desired pressure can be applied to any sintering section, since the friction between the powder and the mold must be much reduced after the powder is solidified. However, the potential length of a product is still limited to the size of the mold used, and the productivity remains at the same level for this proposed method.

In the present investigation, a new pressurized sintering technique to improve productivity is proposed. A prototype machine with an open-end mold was designed for a continuous pressurized sintering process. This open mold was equipped with a step in its sintering room in order to achieve a suitable pressure for the powdered material fed into the mold. Tin powder was sintered by the new system designed here, and the industrial validity of this system was evaluated by examining the density of a sintered rod.

2. Continuous Pressurized Sintering

Figure 1 schematically illustrates an image of the continuous pressurized sintering method assisted by a stepped mold. The one end of the stepped mold is kept open while the powder is supplied from the hopper located on the other side of the mold and is extruded into the mold by a push rod. When the powder passes through the step, the pressure is applied to the powder. The amount of pressure is mainly determined by the sectional reduction ratio of the step. Continuous pressurized sintering is carried out by extruding powder into the mold repetitiously, while the step section is heated to a suitable temperature for sintering of the material.

3. Experimental Procedure

In this study, a band heater was employed as a heating instrument. Due to the restrictions on the performance of this heater, tin powder with a low melting point was selected for the sintering experiment. That was atomized, 99.9% pure, 38 μm under tin powder.

Both the sintering mold and the push rod were made of graphite. Two sets of cylindrical diameters in the mold were examined for continuous pressurized sintering. The first one, as shown in Fig. 2, was the set of φ13.8 mm and φ15.0 mm as the outlet and the intake of the sintering mold, respectively. The height of the step was 0.6 mm. In the other mold, only the outlet diameter was reduced to 13.5 mm to raise the pressure applied to the powder. The height of the step increased to 0.75 mm. The angle of the step was fixed at 60 degrees for both experiments. The mold was heated by the band heater, which was attached to the mold with its center lying on the step. The width of the band heater was about 48 mm. The temperature was monitored by a φ1.6 mm stainless sheath thermocouple inserted into a hole 7.0 mm deep drilled at the step. After establishing a stable temperature of 493 K, tin powder was extruded repetitiously into the mold by a push rod. The powder was extruded at a constant speed of 2 mm/min. The amount of supplied powder was about 6.2 g per repetition. Also at this point, a pre-compacted head of the same tin powder with the same diameter as the intake was added to the mold in advance to prevent powder from passing through the step without compacting at the beginning stage of the continuous sintering process.

4. Results and Discussion

Figure 3 indicates the transition of the load applied to the push rod in the first experiment (outlet diameter φ13.8 mm). The load peak increased with each extrusion at the early stages, but reached a plateau after the 8th extrusion. Figure 4 shows a tin rod produced through this process. It seems that powder was insufficiently added to the mold room. The sintered body looks like a bellows in the early extrusion stages in which the load peak had not stabilized. The sintered product, however, was satisfactory in appearance in the middle of its body, where no bellows-like defects were observed. It is believed that this turning point matches the time at which the load peaks stabilize in the repetitious sintering.

In order to reduce the initial defects in the continuous sintering process, it is necessary to stabilize the peak loads as soon as possible. Therefore, in the following experiment, the outlet diameter was reduced to φ13.5 mm to enlarge the sectional reduction ratio at the step, and a thicker pre-compacted head (8 mm, twice the previous one) was employed. Figure 5 indicates the load transition during this sintering. It is clearly observed that the load peak increased with the extrusion cycle in the early stages of the sintering, but reached a stable level sooner than in the previous experiment. The photograph of a product in the second experiment is shown in Fig. 6. It appears that the initial defects caused in the primary stages of the continuous process were reduced and most of this product had a well-sintered body.

Figure 7 shows the positional dependence of the relative density of a sintered tin rod with a diameter of φ13.5 mm. Densities were measured for seven fractions cut from the sintered tin rod with a width of about 7 mm each. Each fraction was assigned a serial number 1 through 7 from the back end. It is seen that the relative density increased as the section moved to the back end of the rod, reaching 99% at the back end. This value guarantees that tin powder was
successfully solidified by the continuous sintering method proposed in this study. It was expected that further extrusion repetitions would produce a longer tin rod with a high density, but the sintering experiment was not able to produce rods beyond a certain length.

In this method, the load applied to a push rod mainly consists of three factors: a) the passing resistance of the powder at the step, b) the friction between the powder and the mold and c) the friction between the sintered body and the mold, as schematically shown in Fig. 8. The passing resistance of the powder at the step seems to be constant provided that the sintering conditions such as the temperature, the extrusion rate and the shape of the step are not changed. The friction between the powder and the mold, on the other hand, depends on the length of the filled powder $L$. Because this friction increases as the length $L$ gets longer, the load applied to a push rod increases when its tip is located far from the step, while the load decreases as it approaches the step. The effect of the friction between the sintered body and the mold is ignored here since it seems to be much smaller than the other two factors. These factors are summarized in the loading diagram shown in Fig. 8. As the powder supplied from the hopper is fed forward by the push rod, the powder is compacted and the load applied to the push rod increases (1). The compacted powder then begins passing through the step at a certain load level (2). Further extrusion invites a reduction in the length $L$, resulting in a decrease in the load applied to the push rod (3). Finally, the load drops to zero when the push rod arrives at the forward end (4) and is pulled back (5).

The above logic is compared with the actual behavior during the continuous sintering shown in Fig. 5. It is obvious that the actual load increased to the peak value and then began decreasing. The load, however, increased again during extrusion. This behavior seemed to be induced by the sticking of powder around the push rod. This powder caused a strong frictional drag on the push rod. As a result, the push rod was broken at its threaded hole, prepared for a connection to the power train, when it was pulled back.

For these reasons, the sintering experiment was stopped. However, the production of a longer rod with a density of more than 99% is promising if sintering can be continued by improving the strength of the push rod. There is also room to devise a better powder supply system to inhibit powder from entering into the gap between the push rod and the mold. The introduction of a vertical mold is another way to get supplied powder to first land on the previous compactor before being pressurized. In addition, tin powder was selected as the material to be sintered due to the restrictions regarding the amount of heat in this study. The PDS process, however, should excel when applied to refractory materials or hard machining materials such as intermetallic compounds and ceramics. Improvements on the design for the push rod and powder supply system to prevent the push rod from breaking and an examination of a suitable heating method for refractory materials are the future tasks for practical application of the continuous pressurized sintering method.

5. Concluding Remarks

In this study, a new pressurized sintering system that enables the continuous production of a metallic rod assisted by a stepped mold is proposed. It was confirmed that this new system could pressurize a powder fed into the mold even if one end of the mold was kept open as a product outlet. The proposed method resulted in the successful production of tin rod with a relative density of 99%.
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REFERENCES