Compaction of Commercially Pure Titanium Powder by Friction Powder Compaction Process

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A new friction powder compaction (FPC) process is introduced for compacting commercially pure titanium powder. The FPC process is very simple and energy-efficient since it requires only a rotating tool plunged into an aluminum plate with a hole filled with titanium powder, and no external heat source is necessary. The sintering of the powder is mainly achieved by the friction heat and pressing load generated by the rotating tool in the aluminum plate and powder. The microstructure and Vickers hardness of the compacted titanium powder were investigated. It was shown that titanium powder was satisfactorily compacted by the FPC process.

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1. Introduction

Titanium is expected to be used in automotive and aerospace components and medical implants owing to its light weight, high specific strength, high chemical stability and high biocompatibility. The powder metallurgical route is one of the most promising processes for fabricating near-net-shaped titanium components. The compaction of titanium powder has been conducted by various processes such as pressing and sintering,1,2) metal injection molding (MIM),3–5) spark plasma sintering (SPS),6,7) high-pressure torsion,8,9) equal-channel angular extrusion10,11) and compression rotation shearing system at room temperature (CROSS-RT).12) However, a process with higher productivity and lower environmental impact without the need for special equipment that can be used to manufacture low-cost components has been desired.

Recently, friction powder compaction (FPC) has been developed as a process for compacting a mixture of aluminum and sodium chloride powders to fabricate aluminum foam.13) The compaction of the powders is conducted by simply plunging a rotating tool into an aluminum plate with a hole filled with the powders. Namely, sintering of the powders is mainly achieved by the friction heat and pressing load generated by the rotating tool in the aluminum plate and powders. The FPC process is simple and has an extremely short processing time. Also, it does not require an external heat source for the compaction of powders; therefore, the FPC process is more environmentally friendly. Furthermore, it can be conducted using a conventional milling machine and does not require the use of any special equipment, making it a low-cost manufacturing process.

In this study, the FPC process was used to compact commercially pure titanium powder, which has a higher melting point than aluminum. The microstructure and Vickers hardness of the compacted titanium powder were investigated to determine whether satisfactory compaction of the titanium powder was achieved by the FPC process.

Fig. 1 Schematic illustration of compaction of titanium powder by friction powder compaction (FPC) process.

2. Experimental Procedure

Figure 1 shows a schematic illustration of the FPC process for compacting titanium powder. First, as shown in Fig. 1(a), commercially available as-received titanium powder (99.9% purity, <45 µm) was placed in a hole (ϕ = 13 mm) that was previously drilled into a commercially available A1050 pure aluminum plate of 10 mm thickness. The titanium powder used in this study was supplied by Kojundo Chemical Lab. Co., Ltd. (Sakado, Japan). Figure 2 shows a scanning electron microscopy (SEM) image of the titanium powder particles, which revealed that individual particles have an irregular and angular shape. Next, as shown in Fig. 1(b), a rotating tool (ϕ = 25 mm) was pressed into the A1050 plate and the hole filled with the powder. The center axis of the rotating tool corresponded to the center of the hole in the A1050 plate. An SUS304 stainless-steel cylindrical bar with a flat bottom was used as the tool. The rotation speed was 1000 rpm and the tool was lowered at a rate of 1 mm/min. The depth of tool indentation was set as 5 or 7 mm below the surface of the A1050 plate. The holding time was set as 10 s, namely, after the tool reached the specified depth, the tool remained at that depth for 10 s before it was moved upward.

The microstructure of cross section A–A shown in Fig. 1(b) was observed by SEM. The Vickers hardness of the cross section was measured using an MVK-H1 Vickers hardness testing machine manufactured by Mitutoyo Corporation (Kawasaki, Japan) with an applied load of 300 g for 5 s.
3. Results and Discussion

Figure 3 shows the indentation time \( t \)-indentation load \( P \) and indentation time \( t \)-powder temperature \( T \) relationships during the compaction process for the indentation depth of 7 mm. The indentation time \( t \) was the elapsed time from when the tool first touched the surface of the Al050 plate and powder. The powder temperature \( T \) was measured by a thermocouple placed on the bottom of the plate at a distance of 1 mm from the circumference of the hole. The curves can be divided into three regions. First, the indentation of the tool caused an increase in load and a rapid increase in temperature until \( t = 100 \) s. In this region, it is considered that plastic deformation of the Al050 plate occurred. Next, the load remained approximately constant at 5 kN until \( t = 300 \) s and the temperature increased slightly from \( T = 650 \) to 690 K. In this region, it is considered that the spacing between particles decreased and that contact and bonding between particles started to occur. In the final region, the load rapidly increased to above 10 kN and the temperature increased to a maximum of approximately \( T = 750 \) K. In this region, it is considered that intense plastic deformation of the powder occurred and the bonding between particles proceeded. After the indentation depth reached 7 mm (\( t = 420 \) s), which was followed by a holding time of 10 s, the tool was moved upward and unloaded, causing the temperature to decrease rapidly.

Figure 4(a) shows an image of the compacted titanium powder in the case of an indentation depth of 5 mm taken from cross section A–A in Fig. 1(b). This figure corresponds to the end of the second region in the time–load relationship (an approximately constant load of 5 kN) shown in Fig. 3. The upper part of the figure corresponds to the vicinity of the surface in direct contact with the rotating tool, and the lower part corresponds to the bottom of the hole subjected to the FPC process. There were differences in appearance between the upper part and lower part. Figures 4(b)–4(e) show SEM images observed from points A to D indicated in Fig. 4(a), respectively. At point A, as shown in Fig. 4(b), the titanium powder was not compacted and the particles resembled those in Fig. 2. As shown in Figs. 4(c) and 4(d), although the titanium powder was compacted from the upper part at point B, some voids were observed between the particles and the initial shapes of individual particles remained discernible. At point D, as shown in Fig. 4(e), the size of voids was much smaller and the initial shape of individual particles could seldom be observed.

Figure 5 shows the Vickers hardness \( H_V \) of the compacted titanium powder in Fig. 4(a) as a function of \( y \), the distance below the upper surface where the rotating tool was directly in contact during the FPC process. Each value of \( H_V \) is the average value at three points, except for that at \( y = 2.0 \) mm, which is the value at one point because \( H_V \) could not be measured owing to the powder not being compacted. It can be seen that \( H_V \) is approximately 150 in the vicinity of the upper surface, corresponding to point D in Fig. 4. This value is approximately the same as that of pure titanium, and therefore it was shown that titanium powder can be compacted by the FPC process. In contrast, \( H_V \) decreased
with increasing distance below the upper surface to a lower value than that of pure titanium. This is because the number of voids increased with increasing distance as shown in Fig. 4. This is considered to be due to the temperature distribution in the powder; the maximum temperature was achieved in the vicinity of the rotating tool where friction heat was generated, and the temperature decreased with increasing distance below the upper surface. Therefore, the powder was not compacted in the lower part.

Figures 6(a) and 6(b) show typical SEM images of the compacted titanium powder in the case of an indentation depth of 7 mm taken from cross section A–A in Fig. 1(b). The Vickers hardnesses of compacted titanium powder at the upper part (0.8 mm from the surface) and lower part (1.2 mm from the surface), respectively, in the case of an indentation depth of 7 mm taken from cross section A–A in Fig. 1(b). These figures correspond to the end of the third region of the time–load relationship (a load of 10 kN) shown in Fig. 3. The individual titanium particles and the voids shown in Fig. 4 observed at the indentation depth of 5 mm cannot be observed. This tendency was observed in the entire cross section of the compacted titanium powder. The Vickers hardnesses $H_V$ of the upper part (near Fig. 6(a)) and lower part (near Fig. 6(b)), each of which was the average value at 10 points, were $248 \pm 11$ and $199 \pm 9$, respectively. Therefore, it was shown that the titanium powder was compacted by the FPC process. The higher Vickers hardness $H_V$ in the upper part than in the lower part is considered to be caused by the work hardening of titanium and the decrease in grain size due to the high temperature and high shear plastic deformation caused by the rotating tool. Clearly, many more systematic studies are necessary to investigate the relationship between the FPC process conditions, such as the tool rotation speed and tool indentation depth, and the compaction of titanium powder.

The greater Vickers hardness for the indentation depth of 7 mm than for the indentation depth of 5 mm is due to the greater densification of titanium powder. Therefore, all voids were removed by applying a higher load and higher temperature for a longer time.

4. Conclusions

In this study, a new FPC process was introduced for compacting commercially pure titanium powder. The FPC process is very simple and energy-efficient, namely, it requires only a rotating tool plunged into an aluminum plate with a hole filled with titanium powder, and no external heat source is necessary. From SEM observations and Vickers hardness tests, it was shown that titanium powder can be compacted by the FPC process. A maximum Vickers hardness of approximately $H_V = 250$ was obtained at a maximum load of 10 kN and a maximum powder temperature of 750 K. It is considered that the FPC process is promising for the low-cost and environmentally friendly fabrication of titanium compacts and has great potential for a wide range of industrial applications.

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REFERENCES