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Elimination of Surface Porosity in Ti-6Al-4V Alloy Powder Compacts

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Abstract:

How to eliminate surface porosity in titanium alloy sintered powder compacts to improve their fatigue strength was studied, using internal defect-free Ti-6Al-4V alloy powder compacts. Surface pores to a depth of about 100μm in sintered compacts can be completely eliminated by shot peening with steel grits having high cutting power, and the surface smoothness of the sintered compacts after the first peening can be improved by second peening with steel balls. This double shot peening method increases the fatigue strength of the compact by about 150 MPa from the hot isostatically pressed (HIP) condition. If a fine titanium powder of 45 μm or lower in particle size is blended more than 40%, the maximum depth of surface porosity can be reduced to a quarter or less, which can be completely eliminated by subsequent light peening with steel balls.

1. Introduction

Nippon Steel focused attention on powder metallurgy (PM) as one of the near net shape technologies in its effort to expand the titanium alloy market and introduced basic powder metallurgy technology from Dynamet Technology Inc. of the United States in 1987. The technology is called the CHIP process (combination of CIP, short for cold isostatic pressing, and HIP, for hot isostatic pressing) (see **Fig. 1**). The company improved on the technology introduced, found that compacts totally free from internal defects and having mechanical properties equivalent to those of wrought titanium can be produced from extra-low-chloride (≦ 0.005%) titanium powder by appropriately control ling the CIP, sintering, and HIP conditions, and succeeded in commercially producing some titanium powder metallurgy products[1)].

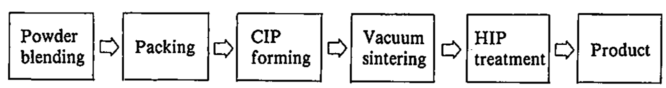

Fig. 1 Manufacture of titanium powder compacts by CHIP process

Titanium powder compacts are claimed to be lower in fatigue strength than comparable forged or rolled materials. The low fatigue strength can be attributed mainly to the presence of internal defects, such as sintering porosity, inclusions and impurities, and such surface defects as pores. Internal defects can be completely eliminated by the combination of the low-chloride titanium powder and CHIP process as noted above. The surface pores (pores in the surface layer) are remnants of open pores formed in or directly below the surface of compacts during sintering, and reach as deep as 200 to 400 μm, depending on manufacturing conditions. The surface pores tend to nucleate fatigue cracks and significantly lower the fatigue strength of powder compacts[2)]. Since titanium powder compacts are often used after surface machining, surface pores have not been studied so extensively as internal defects.

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The surface pores in titanium powder compacts may be rendered harmless by:

(1) Removing them; or

(2) Reducing their size at origin.

Among methods of the first group are machining and shot peening with steel grit high in cutting power. Shot peening is an easy and effective method. Depending on its conditions, however, shot peening is known to increase the surface irregularities of wrought titanium products and reduce its fatigue strength[3,4]. Eylon et al.[2] examined the effect of shot peening on the fatigue strength of titanium sintered powder compacts, but they used specimens with surface defects removed by mechanical grinding. The second group includes the canning treatment and reducing the particle size of the powder used. Few reports are available that have quantitatively investigated the formation behavior of surface pores in titanium compacts made using a finer powder.

The present report describes the results of various studies conducted on the effects of shot peening and particle size reduction on surface porosity and fatigue strength, using internal defect-free Ti-6Al-4V titanium alloy powder compacts.

2. Experimental Results

2.1 Elimination of surface porosity by shot peening

To investigate the effect of peening with grit of high cutting power on the amount of surface porosity elimination, the change in the surface layer of titanium powder compacts produced by the CHIP process was studied by changing the shot peening density.

2.1.1 Experimental methods

Table 1 gives the chemical composition of the experimental material. The starting titanium powder was a blend of low-chloride titanium powder with an average particle size of 80 μm (Cl: ≦0.002%, O: 0.11%, particle size: 45 to 150 μm) and 60% Al-40% V master alloy powder with an average particle size of 30 μm (particle size: ≤ 150 μm). The experimental material was fabricated by the CHIP process into 100 mm diameter and 30 mm long cylindrical specimens. **Fig. 2** shows the manufacturing conditions for each process. The CIP mold was made of ordinary urethane rubber (Shore hardness = 90). The peening material was 0.7 mm size grit (Rockwell hardness = 65), the peening velocity was 73 m/s, and the peening density was varied from 1,000 to 4,000 kgf/m².

Each specimen was shot peened with some portion masked, the thickness difference between the shot-peened area and the non-shot-peened area was measured with a stylus-type surface roughness measuring instrument, and the amount of the surface layer removed by shot peening was calculated from the measured data. The surface roughness of the specimen was also evaluated by the center line height (Ra) of the roughness curve obtained with the stylus-type surface roughness measuring instrument in the longitudinal direction (about 10 mm). **Photo 1** shows the cross section through the surface layer of a specimen before shot peening. The maximum depth of the surface porosity is about 100 μm, and the surface roughness (Ra) is 7.5 μm.

2.1.2 Experimental results

The effect of the peening density on the amount of surface layer removal is shown in **Fig. 3**, and its effect on the surface roughness in **Fig. 4**. The amount of surface layer removal by shot peening increases with increasing peening density, is about 100 μm when the peening density is 2,000 kgf/m² or more, and gradually levels off as the peening density increases further. **Photo 2** shows the surface cross sections of specimens grit peened at different densities. When the peening density was 2,000 kgf/m²

Table 1 Chemical composition of experimental material (mass%)

Al	V	O	N	Fe	C	H	Cl
6.40	4.21	0.12	0.007	0.06	0.03	0.0022	<0.003

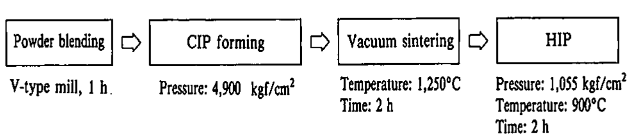


Fig. 2 Manufacturing process conditions

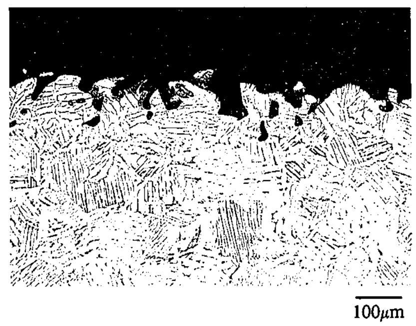


Photo 1 Cross section through surface layer of Ti-6Al-4V alloy HIP compact

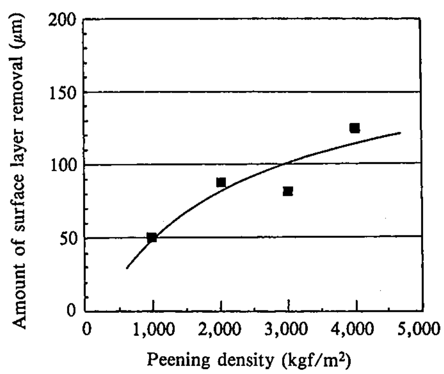


Fig. 3 Relationship of grid peening density with amount of surface layer removal (grit size: 0.7 mm, H_{RC} = 65)

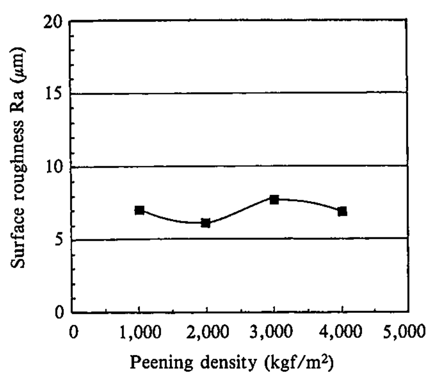
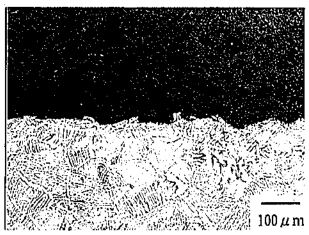


Fig. 4 Relation of grid peening density with surface roughness (grit size: 0.7 mm, H_{RC} = 65)

Grit peened at 1,000 kgf/m^2

Grit peened at 2,000 kgf/m^2

Grit peened at 3,000 kgf/m^2



Grit peened at 4,000 kgf/m^2

Photo 2 Change in surface layer cross section with grit peening density

or more, there remained no surface pores. It is evident from **Fig. 4** that the surface roughness of the specimens is constant regardless of the peening density, and is practically the same as observed before the peening treatment.

2.2 Smoothening of shot-peened surface

Improving the surface smoothness of titanium powder compacts is said to be important in improving their fatigue strength[5]. As shown in **Fig. 4**, surface irregularities after grit peening are still large, and additional surface smoothing is necessary to secure sufficient fatigue strength. Low-intensity peening with steel balls was tested for its effect on improving the surface smoothness of titanium powder compacts.

2.2.1 Test conditions

Chemical composition and manufacturing conditions of the test material, and specimen geometry were the same as described in the preceding section. The specimen was first peened with grit to remove the surface layer and was then peened with steel balls at low speed to smoothen the surface. The shot peening conditions are given in **Table 2**. For steel ball peening, the shot material and shape were held constant, and the peening speed and time were changed over the ranges of 30 to 73 m/s and 30 to 60 s, respectively, and the surface smoothness of the specimens was evaluated by Ra of the roughness curve after shot peening.

2.2.2 Test results

Fig. 5 shows the effects of shot peening conditions on the surface roughness after the second steel ball peening treatment. The surface smoothness improves with increasing peening speed and time, and the surface roughness is reduced by about a half when the peening speed and time are 50 m/s and 20 s, respectively.

Table 2 Shot peening conditions

	Purpose	Shot peening conditions	
1	Surface porosity elimination	Medium	Grit (H_{RC}: 65, Size: 0.7 mm)
		Speed	73 m/s
		Density	2,000 kgf/m^2
2	Surface smoothening	Medium	Steel ball (H_{RC}: 55, size: 0.6 mm)
		Speed (m/s) × time (s)	30 × 30, 30 × 60 50 × 30, 73 × 30

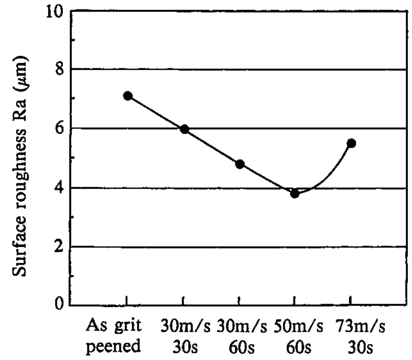
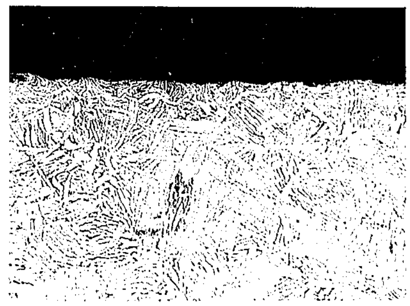
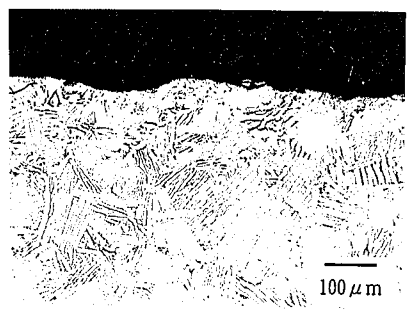


Fig. 5 Change in surface roughness with shot peening conditions



50m/s × 60s



73m/s × 30s

Photo 3 Change in surface layer cross section with second shot peening conditions

When the peening speed is increased to 73 m/s, however, the surface smoothness deteriorates. **Photo 3** shows the cross sections of specimens after the second shot peening treatment. When the peening speed is 50 m/s or less, the effectiveness of shot peening in improving the surface smoothness can be confirmed.

2.3 Fatigue property evaluation with simulated-surface fatigue test specimens

Now that the shot peening conditions for surface pore removal and surface smoothening were clarified, the fatigue strength of titanium sintered powder compacts after the surface pore removal was evaluated using small round fatigue specimens simulating the surface of actual sintered powder compacts.

2.3.1 Test conditions

The chemical composition and the manufacturing conditions for each process of the test material are the same as described in the preceding sections. The geometry of the fatigue test speci-

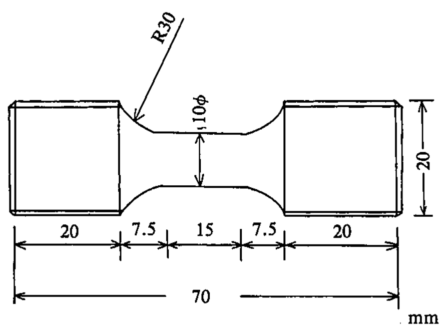


Fig. 6 Geometry of fatigue test specimen (simulating surface characteristics of titanium powder compact)

Table 3 Shot peening conditions of simulated-surface fatigue test specimens

	Medium	Speed	Density
1	Grit (size: 0.7 mm)	73 m/s	2,000 kgf/m²
2	Steel ball (size: 0.6 mm)	50 m/s	2,254 kgf/m²

Photo 4 Cross section through surface layer of shot-peened fatigue test specimen

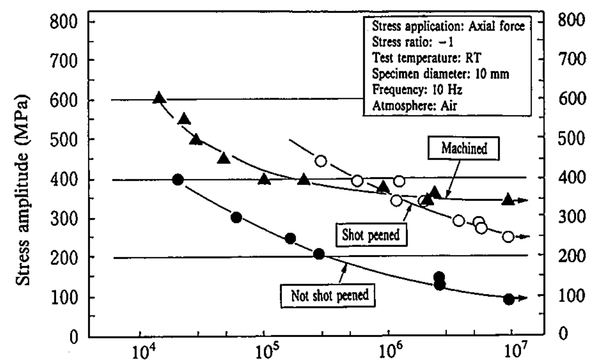


Fig. 7 Fatigue test results of simulated-surface specimens (Ti-6Al-4V)

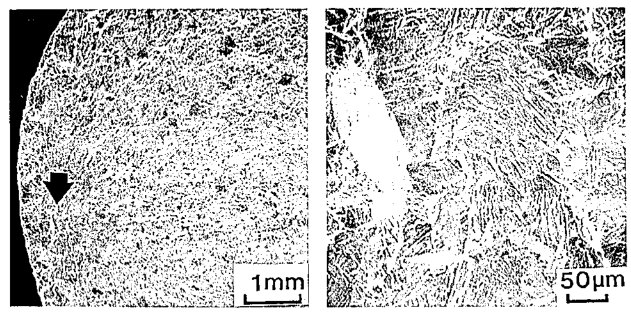


Photo 5 Fracture surface near fatigue crack initiation site (arrow) (double shot-peened specimen)

men is illustrated in **Fig. 6.** A urethane mold was fabricated to suit the geometry of the specimen so that the specimen would exhibit the same surface characteristics as the powder compact, and the powder compact was formed by the CIP process. After HIP, the powder compact was shot peened. The shot peening conditions are given in **Table 3**. The fatigue strength of specimens with as-HIPed surface (Ra ≈ 8 μm) and specimens with mirror-finished surface (Ra ≈ 0) was also evaluated for comparison. The fatigue test was conducted in air under the conditions of axial force, stress ratio R of −1, and maximum number of 10^7 cycles. After the test, the fracture surface of each specimen was examined by scanning electron microscopy (SEM).

2.3.2 Test results

Photo 4 shows the cross section through the surface layer of a specimen after shot peening. Surface pores are completely eliminated by shot peening, and no new defects like microcracks are observed. The fatigue test results are shown in **Fig. 7**. The as-HIPed material without shot peening exhibits the lowest fatigue strength at 100 MPa, whereas the material with surface porosity elimination by shot peening has a fatigue strength of 250 MPa, an improvement of 150 MPa. The mirror-finished material has a smaller fatigue strength than the shot-peened material in the low-cycle region of S-N curve, but has the highest 10^7-cycle fatigue strength of 350 MPa. **Photo 5** shows the fracture surface near the fatigue crack initiation site of a shot-peened specimen. The fatigue crack initiation site was located at 200 to 500 μm below the surface in each specimen and exhibited no defects at all nearby.

2.4 Elimination of surface porosity by use of finer powder

As already described, various manufacturing conditions influence the formation of surface porosity in titanium powder compacts. Among these conditions, the powder particle size has a direct and important bearing on the size of surface porosity. The surface porosity is considered to decrease in size with decreasing powder particle size. Decreasing the particle size of the starting titanium powder, however, is known to linearly increase the oxygen content of titanium compacts[6)] and reduce their ductility[7)]. A study was made of the formation behavior of surface porosity by changing the fine powder blend ratio.

2.4.1 Test conditions

The Ti-6Al-4V titanium alloy was used as test material in the same way as in the previous tests. A conventional titanium powder with a particle size range of 45 to 150 μm, average particle size of 80 μm, and oxygen content of 0.11% was screened with a 325-mesh sieve to obtain a fine powder with a particle size range of 45 μm and less, average particle size of 30 μm, and oxygen content of 0.23%. The blend ratio of the fine powder to the conventional powder was changed in six steps from 0% to 100%. A fine 60% Al-40% V alloy powder with an average particle size of 20 μm was used as the master alloy. Specimens were 10 mm diameter and 30 mm long cylinders, and their manufacturing process conditions from powder compaction to HIP were exactly the same as already described. The microstructure of the cross section through each HIPed specimen was examined by microscopy, and the surface roughness and maximum depth of surface porosity layer of the specimen were measured. The surface roughness was measured as described in the preceding sections. The surface layer of the specimen was observed over the entire circumference on three cross sections by optical microscopy, and the maximum depth of the surface porosity layer was adopted. To study the elimination of surface porosity by shot peening, 10%

40%

60%

100%

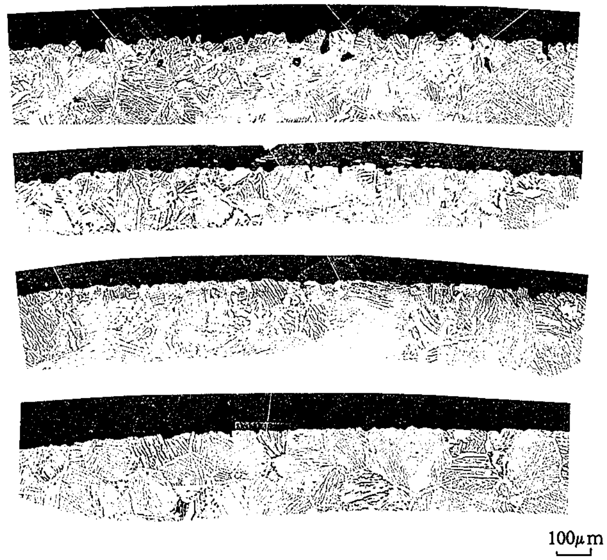


Photo 6 Change in surface characteristics with fine powder blend ratio (after HIP)

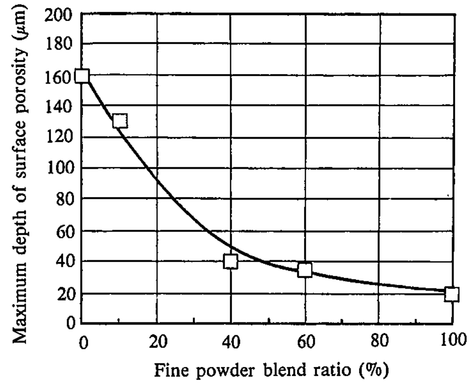


Fig. 8 Relation of fine powder blend ratio with maximum depth of surface porosity

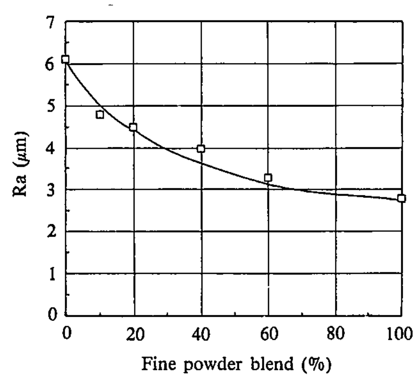


Fig. 9 Relation of fine powder blend ratio with surface roughness

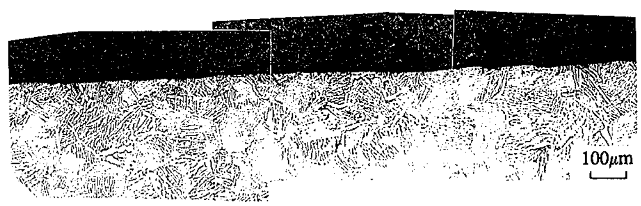


Photo 7 Cross section through surface layer of shot-peened titanium powder compact (40% fine powder)

some specimens were peened with steel balls (size of 0.6 mm and Rockwell hardness of 55) at a density of 2,254 kgf/m^2 and speed of 45 m/s.

2.4.2 Test results

Photo 6 shows the relationship of the fine powder blend ratio with the cross-sectional microstructures of the specimens. As the fine powder blend ratio increases, the surface porosity decreases in size, and the depth at which the surface porosity exists also decreases. **Fig. 8** shows the relation of the fine powder blend ratio with the maximum depth of surface porosity. The maximum depth of surface porosity decreases with increasing fine powder blend ratio. When the fine powder is blended at 40%, the maximum depth of surface porosity is about 25% of that when the fine powder is not blended at all. The change in surface roughness with the fine powder blend ratio is shown in **Fig. 9**. The surface roughness also decreases with increasing fine powder blend ratio. When the fine powder is blended at 100%, the surface roughness is less than a half of that when the fine powder is not blended at all. A specimen with 40% fine powder, a blend ratio at which the decrease in the depth of surface porosity begins to level off, was lightly shot peened after HIP. The cross section through the surface layer of the shot-peened specimen is shown in **Photo 7**. It is confirmed that surface pores are completely eliminated by steel ball peening. The surface roughness of the shot-peened specimen is 2.4 μm, an improvement on the initial surface roughness of 3.6 μm, and no new surface defects like microcracks are recognized.

3. Discussion

3.1 Elimination of surface pores by shot peening

Grit peening is generally used for the surface grinding of materials, and there are several published reports on its application to wrought titanium. Tosha et al.[8] studied the effect of grit peening conditions on the amount of metal removal, using commercially pure wrought titanium. They reported that the amount of metal removal increases with increasing grit peening speed and density and that the surface layer is removed by about 3.5 μm (converted value) when the grit size is 1 mm, the peening speed is 35 m/s and the peening time is about 22 s. Their results cannot be directly compared with the results of the present study. When compared at the 2,000 kgf/m^2 peening density of this study, a condition close to that reported by Tosha et al., the surface grinding depth of titanium powder compact is nearly 30 times greater than that of wrought titanium. This is probably because surface pores serve as notches to facilitate the surface grinding of titanium powder compact and because the surface layer of the powder compact is low in relative density. The amount of surface layer removal by grinding exhibited the tendency to level off at the peening density of 2,000 to 3,000 kgf/m^2. As described by Tosha et al.[8], increasing the peening density is considered to have promoted the work hardening of the surface layer and made it more difficult to grind.

When the peening speed and density are increased further, new microcracks may occur in the surface layer and become initiation sites for fatigue cracks, reducing fatigue strength. Considering this possibility, the peening density and speed must be properly controlled.

Several reports are available concerning the effect of shot peening on the fatigue strength of rolled titanium. Wagner et al.[5] cited surface smoothness, surface residual stress, and surface dislocation density as factors that change the fatigue strength of rolled titanium. They also reported that when rolled titanium is shot peened at a high energy level, there may be cases where the drop of fatigue strength due to the deterioration of surface smoothness more than offsets the gain of fatigue strength from the rise of compressive residual stress by shot peening[2]. In the present experimental work, too, the effect of surface smoothness is more pronounced than that of surface residual stress. The fatigue strength difference of 245 MPa between non-shot-peened titanium powder compacts and mechanically polished titanium powder compacts may be explained as loss of fatigue strength due to the presence of surface pores, because both titanium powder compacts have no effect of residual stress. The shot-peened titanium powder compacts have lower fatigue strength than the mechanically polished titanium powder compacts, probably because the difference of surface smoothness had a greater effect than the difference of surface compressive residual stress.

In this experimental work, fatigue cracks always initiated not at the surface, but in the subsurface of specimens. Usually, in wrought titanium fatigue cracks initiate at the surface under the stress ratio R of -1[9]. The crack initiation site of shot-peened titanium powder compacts is located below the surface, probably because the compressive residual stress introduced by shot peening changed the surface stress distribution and brought the maximum tensile stress point to just below the surface, as reported by Wagner et al.[10] When a fatigue crack occurs at the internal site of the specimen, the mechanism whereby the surface roughness of the specimen affects the fatigue crack initiation and fatigue strength is a future subject of research.

3.2 Elimination of surface pores by particle size reduction and shot peening

Reducing the particle size of the starting titanium powder is very effective in reducing the depth of surface pores and improving the surface roughness of titanium powder compacts, as can be seen from the results of **Figs. 8** and **9**. Use of fine powder at a blend ratio of 40% provides the same surface roughness as when the surface pores are eliminated by the double shot peening method. As shown in **Photo 6**, however, there still remain 20 to 30 μm surface pores. These surface pores can cause a decrease in the fatigue strength of titanium powder compacts. Even if the surface roughness is improved by the particle size reduction, it seems necessary to completely remove surface pores by shot peening after HIP in order to secure fatigue strength comparable to that of titanium powder compacts shot peened twice.

This study adopted the maximum particle size of fine powder as particle shape parameter. The particle size distribution also influences the size and depth of surface pores. It is considered possible to further reduce the size and depth of surface porosity by using a powder blend with the particle size distribution controlled so that gaps between relatively large powder particles are filled by small powder particles, although the fine powder blend ratio is the same. In that case, it should be noted that the relationship between the fine powder blend ratio and the oxygen content increase also changes.

In the present study, the maximum depth of surface pores was about 100 μm. When the size and complexity of titanium powder metallurgy products increase the maximum depth of surface pores is liable to increase. In that case, there is the possibility that surface pores may not be completely removed by the double shot peening method discussed here. The particle size reduction of powders and the double shot peening method will have to be employed in combination: to reduce the depth of surface porosity to about 100 μm after HIP by reducing the particle size, and then completely remove the surface pores by double shot peening.

4. Conclusions

How to eliminate surface porosity, a serious cause of a fatigue strength drop in titanium powder compacts, was studied, using Ti-6Al-4V alloy powder compacts free of internal defects. The following results were obtained:

(1) Shot peening with steel grits having high cutting power can remove surface pores 100 μm or more in depth, and subsequent peening with steel balls can approximately halve the surface roughness of titanium powder compacts.

(2) This double shot peening method improves the fatigue strength of specimens simulating the surface characteristics of titanium powder compacts by 150 MPa as compared with as-HIPed titanium powder compacts. The elimination of surface pores and the improvement in surface smoothness are considered to have the effect of raising the fatigue strength.

(3) Reducing the particle size of the titanium powder blend is effective in preventing the formation of surface pores. If 40% or more of the titanium powder blend is composed of 45 μm or finer particles, the maximum surface pore depth and surface roughness can be reduced by 75% and 50%, respectively. Steel ball peening after HIP can completely remove surface pores and improve the surface roughness of titanium powder compacts.

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