研 究

Effect of Sintering Temperature on Density and Tensile Properties of Titanium Compacts by Metal Injection Molding

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Received April 9, 1999

SYNOPSIS

The preparation of pure-Ti compacts by metal injection molding (MIM) process was described in this paper. To reduce contamination at sintering process, the sintering was performed at low temperature range from 1198K to 1348K. On this sintering condition, the properties of sintered compacts were investigated.

When HDH powder (average particle size is $23\mu\text{m}$) was used as the material, the compacts sintered at more than 1298K had high tensile strength (630MPa) in spite of low density (92.5~94.5%). When gas-atomized powder (average particle size is $15\mu\text{m}$) was used, the sintered compact had high density (~95%) and high elongation (15~20%) at low sintering temperature range (1248K~1298K).

KEY WORDS

metal injection molding, pure-Ti, sintering temperature, density, tensile properties

1 Introduction

Ti-based alloys have excellent characteristics such as low density, high strength, high corrosion resistance and high biocompatibility. However, in the respect of fabricating complicated shaped parts, low machinability may be a barrier to practical uses. Therefore, it is considered to be very available that metal injection molding (MIM) process is applied to fabrication of Ti-based alloys parts.

When MIM process is applied to Ti-based alloy, contamination by interstitial light elements such as oxygen and carbon is serious problem because they have much influence on the mechanical properties of Ti-based alloys¹⁻³⁾. Therefore, it is necessary that debinding and sintering conditions are controlled strictly.

In this study, preparing of pure-Ti compacts are performed by MIM process. To reduce contamination at sintering process, the debound compacts are sintered at low temperature less than 1373K. On this sintering condition, the properties of sintered compacts such as density, tensile strength are mainly investigated.

2 Experimental Procedure

Two types pure-Ti powders made from HDH and gas-atomization processes were used in this study. The chemical composition and mean particle size of both powders are shown in table 1. The mean particle sizes of HDH and

gas-atomized powder were $24.1\mu\text{m}$ and $15.2\mu\text{m}$, respectively. Both powders hardly contain carbon, nitrogen and hydrogen, while contain substantial amount of oxygen. Fig.2 shows the SEM images of both powders. From these photographs, it was clarified that the HDH and gas-atomized powders had irregular and spherical shape, respectively.

These powders were mixed with organic binder consisting of wax and resin for about an hour at 383K~393K by a planetary rotation type kneading machine. The content ratio of organic binder to powder was 43.1vol% for HDH powder and 33.3vol% for gas-atomized powder, corresponding to the shape of powders.

The compounds obtained were injection-molded into the die of tensile test-piece ($110\text{mm (l)} \times 7.5\text{mm (w)} \times 4\text{mm (t)}$) using screw type injection molding machine.

Thermal debinding was performed to 648K in vacuum ($\sim 10^2\text{Pa}$) atmosphere with Ar-gas current. The debinding speed is $1.4 \times 10^{-3}\text{K/sec}$ between 423K to 573K. The binder removal ratios of the specimens of both powders were about 90% on this debinding condition. Then, they were sintered in vacuum (10^{-2}Pa order) at various temperatures from 1198K to 1348K. The heating rate and holding time were $5.56 \times 10^{-2}\text{K/sec}$ and 7.2ksec, respectively.

Fig.2 shows the schematic illustration of sintering the specimens in this study. Both heater and vessel of vacuum furnace were made from graphite. The specimens were set

in alumina (Al_2O_3) vessel and embedded in alumina powder. Fig.3 shows the X-ray diffraction patterns of surface of sintered compacts both embedded in alumina powder and put on alumina powder during sintering. The TiC peaks were detected remarkably as well as Ti peaks on the surface of sintered compact put on alumina powder. As this result, the surface became black on this sintering condition. This fact shows that the surface are carbonized by extricated carbon from graphite heater and vessel. By embedding in

Table 1 Chemical compositions and mean particle sizes of Ti powders.

	Chemical Comp.(mass%)				Mean particle size
	O	C	N	H	
HDH powder	0.23	0.01	0.01	0.06	24.1 μm (45 μm >)
Gas-atomized powder	0.17	0.01	0.01	0.02	15.2 μm (25 μm >)

Fig.1 SEM images of Ti powders. (a) HDH powder (b) Gas-atomized powder

alumina powder, carbonization on the surface are almost prevented.

3 Results and discussins

Fig.4 shows the relationship between relative density and sintering temperature in the specimens of both powders. The densities were measured by the underwater gravimetric method, then they were converted into relative valuesby theoretical density of titanium (4.51Mg/m^3). The density of HDH powder specimen was low value of 82.4% at 1198K. However, it rapidly increased with increasing sintering temperature, and reached to 94.5% at 1348K. On the other hand, gas-atomized powder specimen had comparatively high density at low sintering temperature

Fig.2 Schmatic illustration of sintering.

Fig.3 X-ray diffraction patterns of the surface of two sintered specimens; one embedded in alumina powder and one put on alumina powder during sintering.

such as 92.4% at 1198K and 94.8% at 1248K. Gas-atomized powder is finer than HDH powder. Moreover, powder loading of gas-atomized green compact is higher than that of HDH green compact. These are considered to be the reasons that the gas-atomized powder specimen had higher density than the HDH powder specimen after sintering. However, the increasing rate in density was small more than about 1248K in case of the gas-atomized powder specimen. The relative density was 95.8% at 1348K finally.

Fig.5 shows the relationship between tensile properties in the specimens of both powders at room temperature and sintering temperature. The strain rate in tensile test is 7.0×10^{-4} (/sec). The tensile strength of the gas-atomized powder specimen increased slowly with increasing sintering temperature. The difference in strength between the compacts sintered at 1198K and 1348K was only 60MPa. On the other hand, the HDH powder specimen showed rapid increase with increasing sintering temperature. The difference in strength between the compacts sintered at 1198K and 1348K was 210MPa. These tendencies are considered to reflect the changes of density in Fig.3. Especially, it is worthy of remark that the HDH powder specimen has higher strength than the gas-atomized powder specimen above 1298K in spite of its low density.

The behaviour of 0.2% proof stress of the specimens of both powders showed similar to that of tensile strength. The stress of the HDH powder specimen was higher than that of the gas-atomized powder specimen when sintering temperature was more than 1248K.

The behaviour of elongation (plastic elongation) in the specimens of both powders show quite different from that of tensile strength and 0.2% proof stress. The gas-atomized powder specimen showed high elongation of 15~20% when

sintering temperature was from 1223K to 1298K. However, the elongation went down abruptly to about 5% when sintering temperature was more than 1323K.

The HDH powder specimen did not had much variation with the change of sintering temperature compared with the gas-atomized powder specimen. As a whole, the elongation of the HDH powder specimen was lower than that of the gas-atomized powder specimen. The elongation of the HDH powder specimen was comparatively high value from 6 to 7% at the sintering temperature of 1273K and 1298K.

As mentioned at introduction, it is the problem that the specimen was contaminated by oxygen and carbon at MIM process. The results of chemical analysis of the sintered

Fig.4 Relationship between relative density and sintering temperature.

Fig.5 Relationship between tensile properties at room temperature and sintering temperature.

specimens are shown in Fig.6. The carbon contents of HDH and gas-atomized powder specimens were about 0.06~0.07 mass% and 0.05~0.06 mass%, respectively. These values were a little more than those of original powder (0.01 mass%), but they were hardly affected by the sintering temperature. The increase in carbon content hardly influence on the mechanical properties of sintered specimen^{1,2)}.

On the other hand, the oxygen content in the specimens of both powders increased substantially from that of original powder. The HDH powder specimens had high oxygen of 0.45~0.46 mass%, which were almost constant with changing the sintering temperature. The tensile properties of high strength and low elongation in the HDH powder specimen was considered to be caused by this high oxygen content¹⁻³⁾. As for the gas-atomized powder specimen, the oxygen content was influenced by the sintering temperature. The sintering temperature showed the minimum oxygen

content (0.28 mass%) was 1248K, and at this temperature the elongation became the maximum (~20%).

The increase in oxygen content of the sintered specimens from that of the original powder is considered to be caused by trapping from organic binder or debinding atmosphere. It is difficult to explain precisely the cause of the difference in oxidation behaviours of the specimens of both powders in Fig.6.

However, the increase in oxygen content of the gas-atomized powder specimen sintered at more than 1323K, as shown in Fig.6 (0.33 mass% <), is due to the reaction to alumina powder embedding the specimen at sintering^{4,5)}. In other words, the influence of this alumina powder on the sintered specimen was considered to be disregarded when sintering temperature was less than 1298K.

Fig.7 shows the SEM photographs of fracture surface after tensile test of the HDH powder specimens at various sintering temperatures. Since the specimen sintered at 1198K had low density of 82.5%, a lot of large irregular-shaped pores were observed on its fracture surface. Moreover, the shape of original powder still remained⁶⁾. These surface condition fitted in low tensile properties as shown in Fig.5. Rising the sintering temperature to 1298K (relative density is 92.5%), the shape of pores changed to spherical, and dimple patterns peculiar to ductile fracture were observed. When sintering temperature was 1348K (relative density is 94.5%), cleavage planes suggesting brittle fracture were observed on the fracture surface.

Fig.8 shows the SEM photographs of fracture surfaces of the gas-atomized powder specimens. As a whole, the surface condition was similar to that of the HDH powder specimen sintered at the same temperature. However, fracture facets of the gas-atomized powder specimens were smaller than those of HDH powder specimens. This fact is considered to be due to the difference of original powder size.

Fig.6 Relationship between carbon and oxygen contents and sintering temperature.

Fig.7 SEM images of fracture surfaces of HDH powder specimens. Sintering temperature: (a) 1198K (b) 1298K (c) 1348K

Fig.8 SEM images of fracture surfaces of gas-atomized powder specimens. Sintering temperature: (a) 1198K (b) 1298K (c) 1348K

4 Conclusions

To prepare pure-Ti compacts by MIM process, the sintering was performed at the temperature range between 1198K and 1348K. The following results were obtained from the investigation in properties of the sintered compacts.

- (1) When HDH powder (average particle size: $23\mu\text{m}$) was used as the material, the compact sintered at more than 1298K showed high tensile strength (630MPa) in spite of low relative density (92.5~94.5%).
- (2) When gas-atomized powder (average particle size: $15\mu\text{m}$) was used, the sintered compact showed high density (~95%) and high elongation (15~20%) at low sintering temperature range (1248K~1298K).

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