CHARACTERISTICS OF HIGH PURITY TITANIUM POWDER

BY HDH PROCESS

E. Fukasawa, R. Murayama, and W. Kagohashi

Toho Titanium Co, Ltd., 3-3-5 Chigasaki, Chigasaki-shi, Kanagawa-ken, Japan

1. INTRODUCTION

Recently, the powder metallurgy(P/M) of titanium has been considered as a cost effective processing with emphasis on near-net shape[1-2]. Among titanium P/M techniques, the blended elemental(BE) method has been reported in many applications because of its flexibility of alloy composition and economic advantage[3-4]. In many applications, the quality of titanium powder as a starting material is very critical. Especially, oxygen and chlorine as impurities in the titanium powder can strongly influence mechanical qualities of P/M parts. In order to meet the demand for high quality powder, an extra low impurity titanium powder has been needed.

We have developed an extra low chlorine and low oxygen titanium powder by the hydride-dehydride(HDH) process which is highly optimized under high quality control.

In this paper, the above titanium powder was evaluated about its characteristics such as flowability, compressibility, green strength and so on.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The powders used were produced commercially by the mentioned HDH process. Chemical compositions of the powders studied are tabulated in Table 1.

Table 1. Chemical compositions and bulk densities of the titanium powders used.

<table>
<thead>
<tr>
<th>Type</th>
<th>Chemical Composition(wt%)</th>
<th>Bulk Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ti</td>
<td>Fe</td>
</tr>
<tr>
<td>TC-150</td>
<td>bal.</td>
<td>0.02</td>
</tr>
<tr>
<td>TC-151</td>
<td>bal.</td>
<td>0.04</td>
</tr>
<tr>
<td>TC-152</td>
<td>bal.</td>
<td>0.07</td>
</tr>
</tbody>
</table>
In Table 1, Type TC-150 is a typical high purity titanium powder having an extra low chlorine and low oxygen content. This oxygen content is close to that of sodium reduced titanium sponge fine. Type TC-151 and TC-152, having higher oxygen content, were especially produced to compare with TC-150 in characteristics of hardness of powder particles, compressibility of powders, and green strength. The oxygen content level of TC-151 had seemed to be typical as a standard HDH powder before TC-150 appeared.

A sieve analysis and flow rate of the titanium powders used are listed in Table 2. Also, Figure 1 shows SEM micrograph of the powder(TC-150).

### Table 2. Sieve analysis and flow rate of the powders used.

<table>
<thead>
<tr>
<th>Type</th>
<th>+150</th>
<th>-150+106</th>
<th>-106+75</th>
<th>-75+45</th>
<th>-45µm</th>
<th>Flow Rate (seconds/50gs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TC-150</td>
<td>0.1</td>
<td>33.3</td>
<td>32.3</td>
<td>29.5</td>
<td>5.0</td>
<td>41</td>
</tr>
<tr>
<td>TC-151</td>
<td>0.2</td>
<td>37.6</td>
<td>33.1</td>
<td>24.8</td>
<td>4.3</td>
<td>-</td>
</tr>
<tr>
<td>TC-152</td>
<td>0.2</td>
<td>32.0</td>
<td>33.7</td>
<td>24.2</td>
<td>9.9</td>
<td>-</td>
</tr>
</tbody>
</table>

As described in Table 2, these powders have a similar particle distribution. Also powder particles of TC-150 are angular in shape like other HDH powders.

### 2.2 Methods

As fundamental characteristics of the powders, flowability, compressibility, green strength and densification by sintering were investigated. The hardness of the powder particle itself was also measured in order to confirm its effect on these characteristics. Each characteristic was measured by the following procedure:

1. **Flowability of powder**
   Flowability was determined by Carr's method[5]. Along the method, flowability of the powder(TC-150) was totally evaluated by using four properties, angle of repose, compression rate, angle of spatula and coefficient of uniformity. Each property was measured by using an all-in-one type apparatus, as shown in Figure 2.

2. **Hardness of powder particles**
   Micro-Vickers hardness of powder particles was measured. Each measurement was carried out by pressing the central portion of twenty powder particles ranging from -150+106µm diameter, molded in a resin. The hardness was determined by averaging the twenty-particles hardness measured.

3. **Compressibility of powders**
   Compressibility was evaluated in accordance with The Japan Society of Powder and Powder Metallurgy standard (JSPM1-64), "Compressibility Test of Metal Powders," by the use of floating die pressing at given pressures of 98MPa to 686MPa. Compressibility was expressed as relative density of the theoretical. In this method, the green compact is cylindrical and has a diameter and height of 11.3mm, respectively. When pressing the powders, a die wall lubrication(lubricant, zinc stearate) was adopted.
Green strength was evaluated by the Rattler test which provides information on the abrasion resistance of a green compact and the ability to retain its shape (edge stability). The Rattler test is specified in JSPM4-69, "Method for Determination of Green Strength by Rattler Test," using five green compacts pressed at same given pressure. In the test, the green strength is reported as weight loss (%). The green compacts were pressed at 294 MPa to 686 MPa of applied pressures as the compressibility test above.

Densification by sintering
Sintered densities were measured by sintering green compacts in a vacuum at 1300°C for two hours. The green compacts were pressed from 98 MPa to 490 MPa by a die pressing. Densification by sintering was expressed as sintered density with green density.

3. RESULTS AND DISCUSSION

3.1 Flowability of powder
Table 3 lists results of flowability evaluation. Consequently, the flowability of TC-150 is fairly good. This means that this powder requires no special attention in its handling.

Table 3. Flowability of the high purity titanium powder (TC-150).

<table>
<thead>
<tr>
<th>Angle of Repose (degree)</th>
<th>Compression Rate(%)</th>
<th>Angle of Spatula (degree)</th>
<th>Coefficient of Uniformity</th>
<th>Flowability</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>17</td>
<td>48</td>
<td>2</td>
<td>fairly good</td>
</tr>
</tbody>
</table>

3.2 Hardness of powder particles
Figure 3 gives hardness of powder particles adopted. As shown in Figure 3, the higher the oxygen content in the powder, the higher linearly the hardness. It is well known that oxygen in commercially pure titanium has a strong effect on hardness of the titanium itself. Kusamichi et al. reported that oxygen as an impurity in titanium could affect approximately twice in content (ppm) as strong as iron [7]. According to this result, increase of the hardness in Figure 3 can be explained mainly by the effect of oxygen in the powders. About work hardening of the particles, there was no evidence because of no significant change in hardness after annealing the powders in an inert gas at 670°C for ten hours. As a result, TC-150 containing 0.11 wt% oxygen is a softer powder. This will reflect good effects on other characteristics, for example, compressibility.

3.3 Compressibility of powders
Figure 4 shows the comparison of compressibility among three kinds of powders used. Through 98 MPa to 688 MPa of compacting pressure, TC-150 showed better compressibility. At 495 MPa, the relative density of TC-150 was 86% of the theoretical. This compressibility will be close to that of sodium reduced titanium sponge fine.
Generally, the compressibility of a powder is influenced by several factors, inherent hardness of the metal, particle shape, internal porosity, particle size distribution, and so on. Among three powders used, the most affecting factor will be the inherent hardness of the titanium particles. As described above, the inherent hardness of TC-150 particle is lower. Therefore, the powder would be more compressible during pressing.

3.4 Green strength
Figure 5 illustrates the effect of compacting pressure on green strength of the compacts. As TC-150 gave weight loss of less than 1% at 490MPa, it would be satisfactory to maintain size and shape during handling prior to sintering. Because the strength of green compacts results mainly from mechanical interlocking of particle surface irregularities, particle shape will be the most important factor contributing to green strength. Accordingly, in comparison with the powders having different oxygen content, no significant difference in green strength was seen.

3.5 Densification by sintering
Figure 6 gives sintered densities versus compacting pressures. With increase in the compacting pressure, the sintered densities also increased similar to the green densities. At 490 MPa, the sintered density was 4.1g/cm³. From this result, it will be understood that TC-150 can reach higher density after sintering in a vacuum only. In order to increase the sintered density of the TC-150, fine powder(-45µm) was added to the TC-150. The fine powder had also an extra low chlorine(<0.002wt%) and low oxygen(0.23wt%). Figure 7 shows the effect of the fine powder addition on the sintered densities of the mixtures. As illustrated in the figure, the sintered density increased considerably by adding the fine powder. In particular, adding the fine powder up to 30wt% resulted in remarkable increase of the density. At 30wt% of the fine powder addition, the sintered density was 94% of theoretical density. On the other hand, the green density of the mixed powder decreased significantly with the addition of the fine powder. At 100wt% of the fine powder, the green density was only 80% of the theoretical. However, up to 30wt% of the addition, the green density was decreased slightly. The green density of the mixture was about 85% at 30wt% of the addition.

Though there are numerous variables that can affect sintered density, the better compressibility of TC-150 will reflect this characteristics also.

4. CONCLUSIONS
(1) The high purity titanium powder including the extra low chlorine and low oxygen is being produced commercially by the HDH process.
(2) The powder(TC-150) gives satisfactory flowability and green strength.
(3) This powder is more compressible than other HDH powders. By using this powder as a starting material, denser green and sintered compacts can be obtained.
REFERENCES


Fig. 1 Scanning electron micrograph of the powder (TC-150).
Fig. 2 All-in-one type apparatus for flowability evaluation.

Fig. 3 Hardness of particles versus oxygen contents in powders.
Fig. 4 Comparison of compressibility among the powders used.

Fig. 5 Effect of compacting pressure on green strength of the compacts.
Fig. 6 Effect of compacting pressure on sintered densities.

Fig. 7 Effect of fine powder addition on sintered densities.