Optimization of Parameters for Pressureless Sintering of Titanium and Ti-6Al-4V Powders by a Taguchi Method

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This work describes the effects of various parameters on the pressureless sintering of Ti and Ti-6Al-4V alloy powders. Specific sintering trials were carried out based on a Taguchi method using four parameters (particle size, compaction pressure, sintering temperature and holding time). Each parameter was studied at three different levels. The effect of sintering parameters on densification is discussed for three different materials (Ti-200mesh, Ti-6Al-4V-200mesh and Ti-6Al-4V-100mesh). Confirmation experiments were carried out to validate the optimum parameters obtained from the original design experiments. Phase constituents and microstructure were characterized using X-ray diffraction (XRD) and environmental scanning electron microscopy (ESEM). Optimum sintering parameters for achieving the highest density were 1250℃ and 8 hours of holding time for Ti powder, and 1400℃ and 6 hours for Ti-6Al-4V powder. Validation trials confirmed the optimized parameters with Ti-6Al-4V-200mesh samples having density close to 95%. The additional analysis showed differences between the surface and centre of the samples, in terms of different phases, morphology and porosity.

Keywords: *Titanium, Ti-6Al-4V, taguchi method, sintering parameters*

1. Introduction

Although titanium and titanium alloys have high strength to weight ratio, good corrosion resistance, and excellent biocompatibility[1-4], the application of titanium products is still limited to a few specialized areas such as aerospace industry, primarily because of high cost and difficulties associated with forming, e. g. difficult machining[4,5]. Powder metallurgy (P/M) technology has the potential to reduce the price to affordable levels when compared with wrought and casting methods[6,7]. Current research works related with powder pressureless sintering of Ti P/M parts are mostly carried out in vacuum or inert atmosphere, however most experiments have been conducted under only a limited range of conditions. This work focuses on the optimization of sintering parameters for both Ti and Ti-6%Al-4%V powders through a Taguchi statistical method and suggests the optimal sintering parameters based on this method[8,9].

2. Experimental Design

Three different batches of hydride-Dehydride (HDH) powders were used: elemental titanium powders (−200 mesh), Ti-6Al-4V alloy powders (−100 mesh) and Ti-6Al-4V alloy powders (−200 mesh). The differences in the two Ti-6Al-4V powders are the impurity level and particle size. The as-received powders were further sieved into two size ranges: 0-75 μm and 75-150 μm. The powders were pressed into compacts using different compaction pressure, followed by cold isostatic pressing (CIP) at 200MPa to achieve good uniformity. The pressed compacts were sintered in a high temperature vacuum furnace under various sintering conditions. Experiments were designed based on the Taguchi statistical method, which involves choosing three different levels for each of the four experimental variables: particle size distribution, compaction pressure, sintering temperature, and sintering time. The advantage of using this design method is that it allows one to investigate the effects of an individual parameter without having to carry out every possible combination of all four parameters. The four parameters and their levels can be determined by carrying out a minimal number of experiments as described in Table 1. Other invariable sintering conditions were set as argon atmosphere of 1atm and heating rate of 10℃/min. Note that each of these runs includes the compacts from all three of the powder types described above.

The density was measured by Archimedes' principle according to ASTM B962-08 standard. The phase structure and morphology was determined by X-ray diffraction (XRD) and environmental scanning electron microscopy (ESEM) respectively.

Table 1. Orthogonal array L9 (3^4) for Optimization of Sintering

No.	Pressure (mPa)	Temperature (℃)	Time (hrs)	Particle Size (μm)
1	500	1100	8	0-75
2	500	1250	4	75-150
3	500	1400	6	As Received
4	400	1100	4	As Received
5	400	1250	6	0-75
6	400	1400	8	75-150
7	300	1100	6	75-150
8	300	1250	8	As Received
9	300	1400	4	0-75

3. Results and Discussion

3.1 Density

The sintered density results for each condition are illustrated in Figure 1. Each value was the average obtained from experiments carried out under three conditions where one parameter is kept constant and the

others varied. The error bars in Figure 1 represent the spread of data from the three experiments. From Figure 1(a), it can be seen that for a given powder, the sintered density was not significantly affected by the compaction pressure. In general a higher pressing pressure can lead to more contact areas, which is advantageous for the diffusion-controlled densification process. However, the gas trapped in the compact under higher compaction pressure may be more difficult to eliminate, which in turn adversely affect densification[7]. The holding time had an effect on the sintered density for the Ti-200mesh powders, achieving the highest density when holding for the longest time of 8 hours as seen from Figure 1(b). However this effect was not observed for the Ti-6Al-4V powders which had similar densities at all holding times.

Figure 1(c) compares the density for each of the powders with different particle size distributions; as received, classified 0-75 μm and 75-150 μm. The as received Ti-6Al-4V (-100 mesh) powders had the lowest density, as might be expected from the coarser powder, since the larger particle size would result in lower surface contact area which is less favorable for the densification process. After sub-sieving into <75 μm, the sintered density significantly increased, compared to the as-received powder. For this powder there was also a significant effect of the classification with the smaller size range having higher density than the larger one again due to the effect of surface area. However, this was not seen in the classification groups for the Ti-200mesh and Ti-6Al-4V-200mesh powders. These-200mesh powders should theoretically be all less than 75μm and the 75-150μm size range produced by sieving is probably made up of agglomerates of smaller particles. Particle size analysis (not shown) indeed confirmed that the majority of particles were below 75μm and therefore it is not surprising that the two size ranges showed little or no difference in density.

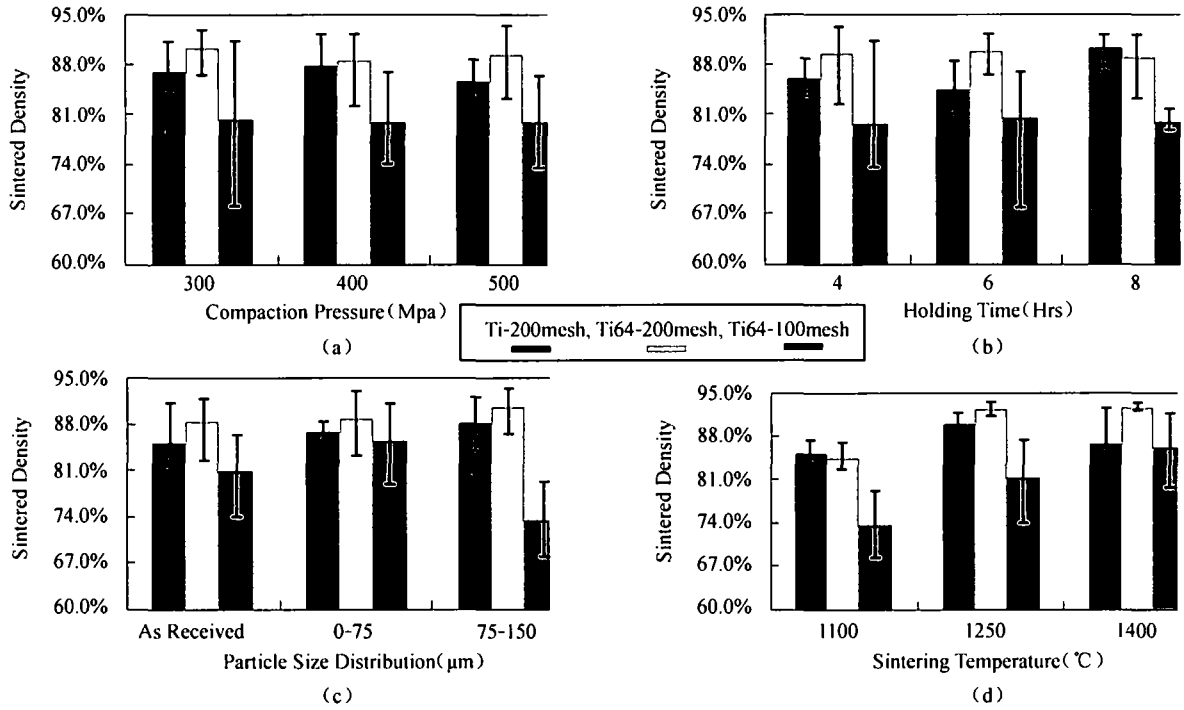

Figure 1. Statistic Relationship of Compaction Load, Holding Time, Particle Size Distribution and Sintering Temperature with Sintered Density

Figure 1(d) shows the effect of sintering temperature, where the Ti-200mesh powders achieved the maximum density at a sintering temperature of 1250℃ whereas for the alloy powders the density was improved by further heating to 1400℃. In general high temperature will benefit densification as it accelerates diffusion process[6]. The scattering of values when keeping the sintering temperature constant whilst varying the other parameters was smaller than those cases where the other variables were kept constant, implying that the temperature has a more important role in comparison with other parameters for the sintered density.

Through analysis of these results, it is possible to determine the optimal combination of parameters that would lead to the possibly highest density based on the Taguchi design, and these conditions are shown in Table 2 along with the resulting densities. These values are comparable with, or higher than, the best results obtained from the original runs and would thus seem to validate the choice of these parameters.

Table 2. Density Data for Confirmation Experiments

	Ti-200mesh	Ti64-200mesh	Ti64-100mesh
Load (mpa)	300	300	300
Temp. (℃)	1250	1400	1400
Time (hrs)	6	8	8
Size (μm)	0-75	0-75	0-75
Density	90.22%	94.32%	92.36%

3.2 Morphology Analysis

Morphological examination of surface and cross sections of the samples produced from the preliminary trials were carried out by ESEM. Figure 2 highlights the contrast between the surface and cross section for Trial No. 7 of Titanium-200mesh and the cross section of as-polished raw titanium powder.

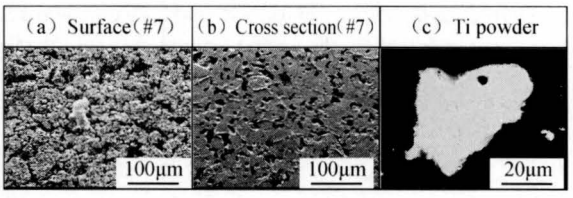

Figure 2. Surface and Cross Section of Sintered Titanium Specimen and Cross Section of As-polished Ti Powder

The surface morphology is different from that of the cross section, and is thought to be due to reaction between titanium and contaminants in the furnace atmosphere such as carbon and/or oxygen. Since the density calculations were taken from the entire sample, this porous surface would lead to lower overall density values, and therefore the densities presented above may be higher if this surface layer is removed. There are some pores pre-existing in the raw titanium powders (Figure 2(c)). These pores are difficult to remove during sintering unless they are closed up during pressing, and therefore partially are responsible for the existence of macropores observed in the grain interiors of the etched specimen (Figure 3).

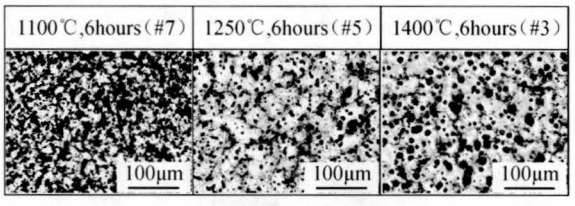

Figure 3. Metallographic Cross-section Pictures for As-etched Ti-200mesh Specimens

Figure 3 shows the metallographic pictures of as-etched Ti-200mesh sintered specimens when sintered at different temperatures. The average pore size increased and the pore shape became more spherical when sintering temperature increased from 1100℃ to 1400℃.

The pores were observed mainly at grain boundaries, junctions and grain interiors.

3.3 Phase Determination

The ESEM images demonstrate that there are differences in porosity between the surface and the centre, thus, it is interesting to investigate whether this has any effect on the phase structure or composition within the bulk material. Figure 4 shows XRD patterns of surface and cross section for the trial (#3) for titanium-200mesh as being representative of all of the samples. The spectrum of the cross section shows only single alpha phase.

In contrast, the surface XRD shows that the major phase is one which might be a solid solution of TiC and TiO, as the peaks lie in between those expected for TiC or TiO. It is therefore indexed as TiCO[10] in Figure 4.

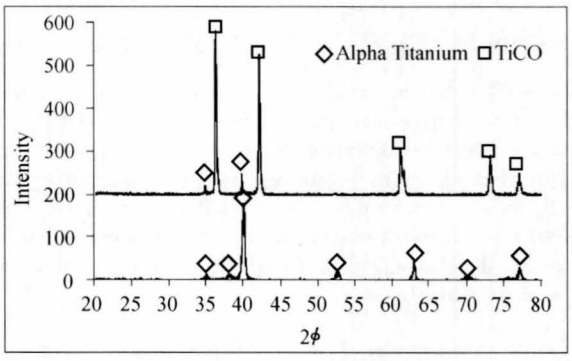

Figure 4. XRD Patterns of the Surface and Cross-section for Ti #3

4. Conclusions

Initial sintering trials have been completed for Ti and Ti alloy powders based on a Taguchi design method, and validation runs carried out under the optimum conditions. Sintering at 1250℃ and holding for 8 hours resulted in the highest density for the Ti-200 mesh powders; whereas for the alloy powders the best sintered density was achieved at 1400℃ with a holding time of 6 hours. Surface analysis by ESEM and XRD showed the presence of a porous surface, lower Ti concentrations and a crystalline phase that is identified as a solid solution of TiO and TiC. It is believed that these surface features caused by contamination have led to a decrease in the final measured density.

Acknowledgements

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