Producing High-Quality Titanium Alloy by a Cost-Effective Route Combining Fast Heating and Hot Processing

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Powder Metallurgy (PM) is a very attractive method for producing titanium alloys, which can be near net shape formed and have freedom in composition selection. However applications are still limited due to product cost affordability. In this paper, we will discuss a possible cost-effective route, combining fast heating and hot processing, to produce titanium alloys with similar or even better mechanical properties than that of ingot metallurgy titanium alloys. Two titanium alloys, Ti-5Al-5V-5Mo-3Cr (Ti-5553) and Ti-5Fe alloy, were successfully produced from HDH titanium powder and other master alloy powders using the proposed processing route. The effect of processing route on microstructural variation and mechanical properties were discussed.

INTRODUCTION

Titanium alloys have a lot of advantages comparing to steels which are commonly used in manufacturing industry, such as high specific strength and excellent corrosion resistance.[1-4] However, titanium alloys are very expensive because of their high materials costs, high buy-to-fly ratio for making the components and high manufacturing costs. These factors limited the titanium alloys to be widely used in manufacturing industry, especially for civil industry, despite their properties were very attractive.[5] Powder metallurgy (PM) method is able to nearly-net-shape form intricate products, leading to significantly reducing the manufacturing cost and saving materials. The drawbacks for PM method are that it is easy to introduce impurities into the final product during the processing and hardly make high dense materials without post-processing, this has a potential to deteriorate the mechanical properties of PM titanium alloys. Furthermore, the cost of PM titanium alloy products is still not affordable as a results of using the prealloyed titanium powders as starting materials. Hot processing of the blended elemental titanium powder mixtures, such as powder compact extrusion, is a potential cheap and effective method to produce high-dense Ti-6Al-4V alloy, which have comparable mechanical properties with that of ingot metallurgy counterparts.[6,7] In this paper, we will discuss a possible route that combines fast heating (induction and microwave heating) and hot processing to produce complicated and non-standard titanium alloys from cheap powders, such as Ti-5Al-5V-5Mo-3Cr (Ti-5553) (wt.%) and Ti-5Fe (wt.%) alloys. This process is a possible cost-effective method to produce titanium alloy parts with comparable mechanical properties with that of ingot metallurgy titanium products, and the microstructures and mechanical properties of Ti-5553 and Ti-5Fe alloys prepared are discussed.

EXPERIMENTAL PROCEDURE

Two titanium alloys, with nominal composition of Ti-5Al-5Mo-5V-3Cr (Ti-5553) and Ti-5Fe (wt.%), were prepared by a process combining of fast heating (induction heating and/or microwave heating) and hot processing, from elemental powders and master alloy powders. The starting materials were Ti powder produced by a hydride-dehydride (HDH) process (-200mesh), Al powder (purity: 99.9%), pure carbonyl Fe (-45μm) and Al35V65, Al15Mo85 and Al3070Cr (wt.%) master alloy powders (the particle size was smaller than 75μm, commercial purity) supplied by Dalian Rongde Company, PR China. The powder mixture, with a designed composition, was mixed for 20 hours using a roller mill at a speed of 200rpm. Then the mixed powder mixture was warm compacted at 230°C under a pressure of 400MPa into a cylindrical shape using 100-ton hydraulic press in air. The relative density of the compacts was about 83-85%. After the compaction, the Ti-5553 powder compact was induction heated (heating rate was about 180ºC/min) to 1300ºC and holding the temperature for 10min, and then hot pressed, and the Ti-5Fe powder compact was consolidated by microwave sintering (heating rate was about 28ºC/min) at 1300ºC for 22min. Then the as-consolidated billets were reheated using induction furnace to the desire temperature (950ºC for Ti-5553 and 1050ºC for Ti-5Fe) for extrusion in air. The extrusion ratio was about 9:1, and the extrusion processes were performed by 300-ton hydraulic press (XJ 300, produced by Wuxi Yuanchang Machinery Co. Ltd, PR China).

Scanning electron microscopy (SEM) (HITACHI S4700) were used to examine the microstructure of the hot pressed, microwave sintered and extruded titanium alloy billets and samples. The ground and polished surfaces of samples for OM examination were etched in a modified Kroll's reagent consisting of 2vol% HF, 4vol% HNO_3 and 94vol% H_2O. The tensile tests were conducted at room temperature using an Instron (INSTRON 4204) universal testing machine with dog-bone shaped specimens, having a rectangular cross section of 2mm×2mm and a gauge length of 20mm. The strain was measured using an extensometer with a gauge length of 10mm. The strain rate used for tensile testing was 10^{-4} s^{-1}. The oxygen contents of powder mixture and the solid parts were measured using Inert Gas Fusion Method by the standard of ASTM-E-1019 at Durkee Testing Laboratories, Inc, USA.

RESULTS AND DISCUSSION

Microstructure and properties of Ti-5553 alloy

Fig.1 shows the XRD patterns of the Ti-5553 alloys processed at different conditions. It exhibits that only β phase peaks appeared in Fig.1a, and this means that the Ti-5553 powder compact was fully alloyed during the process of induction heating to 1300ºC, holding the temperature for 10 minutes and hot pressing, forming Ti-5553 alloy materials. The microstructures of Ti-5553 alloy processed at different conditions are presented in Fig.2. From the SEM microstructure of the hot-pressed Ti-5553 alloy billet (Fig.2a), it can be clearly seen that the microstructures of the hot-pressed billet were mainly composed of equiaxed grain structure, with a grain size of up to 50µm. The microstructure also suggests that the master alloy powder particles were completely dissolved into the titanium matrix and the porosity was less obvious, with a few closed pores left inside of the equiaxed grains. The relative density of the hot-pressed Ti-5553 was measured to determine a value of about 97%, this confirms that the hot-pressed billet had less porosity than the compact. BSE microstructure (Fig.2b) suggests that only small amount of α phase, with acicular morphology, precipitated from the β matrix and located in grain inside and at grain boundaries. Due to the amount of α phase was very small, there is no α peaks appeared in the XRD pattern (Fig. 1a). The reason why master alloy powder particles could be dissolved into titanium matrix in the short process of induction heating, temperature holding and hot pressing, it could be explained from the pressure-assisted sintering theory, applying pressure (about 400MPa) on the 1300ºC-induction-heated Ti-5553 compact would:[6,7] (1) enlarge contact area of powder particles; (2) cause plastic flow through dislocation gliding; (3) promote grain boundary diffusion; and (4) accelerate volume diffusion from the grain boundary. All these factors would significantly accelerate the master alloy powder particles to dissolve into the titanium matrix.

After extrusion at 950°C, both of α and β peaks are observed in the XRD pattern (Fig.1b), the number and intensity of α peaks were quite small, indicating that the amount of α phase in the as-extruded Ti-5553 alloy was not very high. However, more α peaks were appeared, as shown in Fig.1c, when the extruded bar was double-aged at 450°C for 6h and then 675°C for 30min. The intensity of α peaks was significantly increased, compared to that of the 950ºC-extruded alloy. This was because the sample was cooled in flow argon after extrusion and the cooling rate was faster than that of furnace cooling (close to equilibrium condition), and the precipitation of α phase from β phase was significantly suppressed during and after the extrusion, leading to the extruded bar were mainly composed of β phase. The phase transformation of β/α was triggered by the heat treatment, so that substantial α phases could precipitate from β matrix, as a result that much more α peaks with high intensity were observed in the XRD pattern of the double-aged sample. SEM images clearly show that the microstructure of the 950ºC-extruded Ti-5553 alloy have strip microstructures (Fig.2c), where dark strips are composed of equiaxed grain structure with dimension of 5-20µm (Fig.2d), and bright strips show a lot of acicular α precipitations inside the original β equiaxed grain (Fig.2e and f). After double-aging (450ºC for 6 h first and then 675ºC for 30 min),

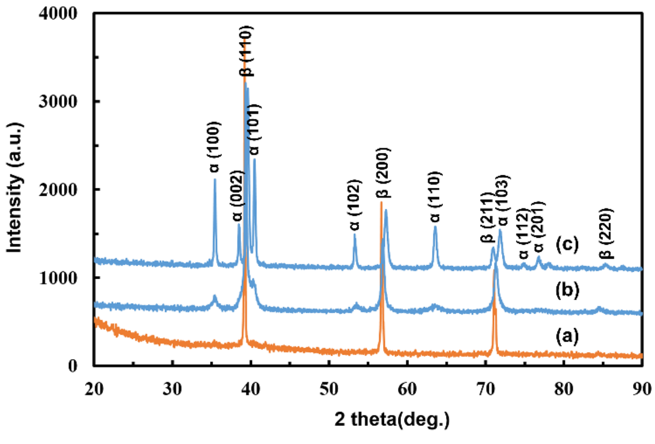


Fig. 1. XRD patterns of the Ti-5553 alloys: (a) hot pressed at 1300ºC and with a holding time of 10min, (b) extruded from the hot-pressed billet at 950ºC, and (c) double-aged (450ºC/6h + 675ºC/30min) the extruded alloy.

the strip microstructure is almost disappeared and becoming more homogeneous than that of the as-extruded Ti-5553 alloy, as shown in Fig.2g. In the high magnification SEM images (Fig.2h, i, j and k), α precipitations are clearly observed, which are composed of acicular α, fine particle α and grain boundary α. Since the Ti-5553 alloy was extruded at high temperature and followed by air cooling to room temperature, the stability of the as-extruded Ti-5553 was quite low and the driving force for the nucleation and growth of α phase was high during aging. Thus, α precipitation could form more homogeneously throughout the microstructure, evidenced by the formation of large number of α precipitations within β grains and discontinuous α phases at the original β boundaries.[8]

The mechanical properties of Ti-5553 alloys processed at different conditions are listed in Table I. It could be concluded that the hot-pressed Ti-5553 alloy had very poor mechanical properties, no yield occurred and the ultimate strength was about 800MPa. The mechanical properties of the as-extruded Ti-5553 alloy was significantly improved compared to that of the hot-pressed alloy, with a yield strength of about 1270MPa, a ultimate strength of about 1370MPa and the elongation to fracture of about 3%. The double-aging heat treatment had an important impact on the as-extruded Ti-5553 alloy and made significant contribution to increase its mechanical properties: the yield strength reached 1435MPa, the ultimate strength was about 1530MPa, and the elongation to fracture was increased to 8.5%. This was about 13% increase in yield strength, about 11% increase

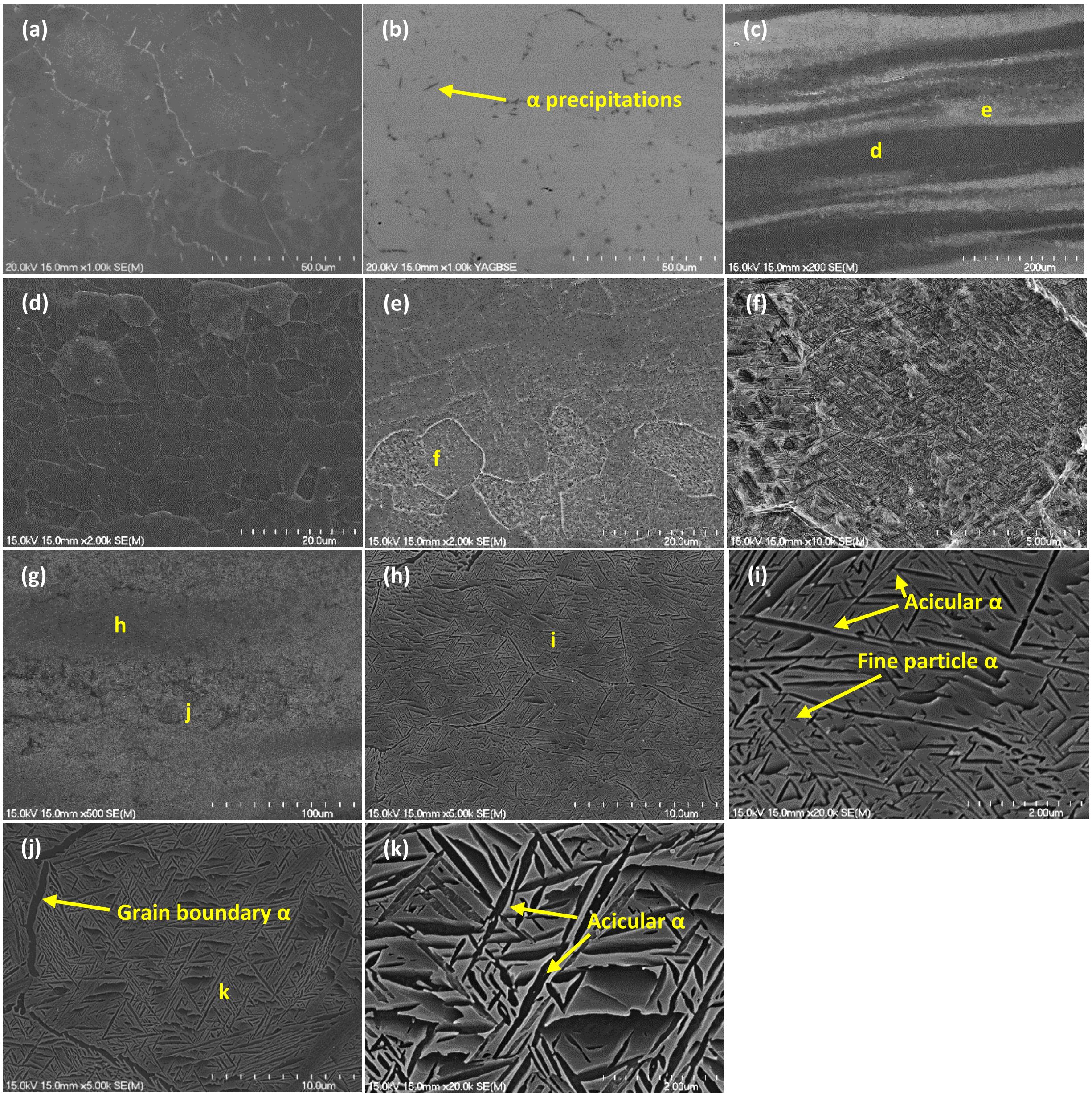


Fig. 2. Microstructures of Ti-5553 alloy: (a) and (b) hot pressed at 1300ºC and with the holding time of 10min, (c)-(f) extruded from the hot-pressed billet at 950ºC and (g)-(k) double-aged (450ºC/6h + 675ºC/30min) the extruded alloy.

in ultimate strength, and about 183% increase in the elongation, comparing to that of the as-extruded Ti-5553 alloy.

The low mechanical properties for the hot-pressed Ti-5553 alloy was mainly caused by the equiaxed microstructures and unstable single β phases resulted from fast cooling after hot pressing. For the as-extruded Ti-5553 alloy, the strength was significantly improved and yield happened, because of the fine microstructure and small percentage of α particles precipitated from the β matrix, which enhance the alloy strength through precipitation hardening. After double-aging heat treatment, a lot of α precipitations with different morphologies: fine particle α, acicular α and discontinued grain boundary α, were precipitated from the β titanium matrix, this contributed to both of the strength and ductility, because fine particles and/or acicular structure were harder than β phase and harder to be deformed; α/β interfaces pinned the movement of dislocations, increasing the strength;[9,10] and grain boundary α could be deformed by slipping and shearing mechanisms so that the dislocations could be activated and accumulated within them, improving the ductility.[11,12] Due to the finer microstructures and α precipitations formed in the as-extruded Ti-5553 alloy than that of the ingot Ti-5553 alloy, the strength of the studied Ti-5553 alloy was much more higher than that of the ingot metallurgy Ti-5553 alloy.[13] The oxygen contents for both of hot-pressed and extruded Ti-5553 alloy were 0.39wt.%, which were about 0.06wt.% increase comparing to the starting powder mixture (0.33wt.%). Unlike Ti-6Al-4V alloy, there was no critical value for oxygen content in β titanium alloys. It reported that the elongation of β titanium alloys was not significantly affected by the contained oxygen level due to beta titanium alloys were less sensitive to the influence of oxygen on the tensile ductility than alpha titanium alloys.[14] Yang et. Al found that the tensile elongation was more than 8% for as-sintered near beta Ti-10V-2Fe-3Al alloy with an oxygen level of 0.59wt.%.[15] Thus, the microstructure take more important role to render Ti-5553 alloy with better mechanical properties comparing to the oxygen.

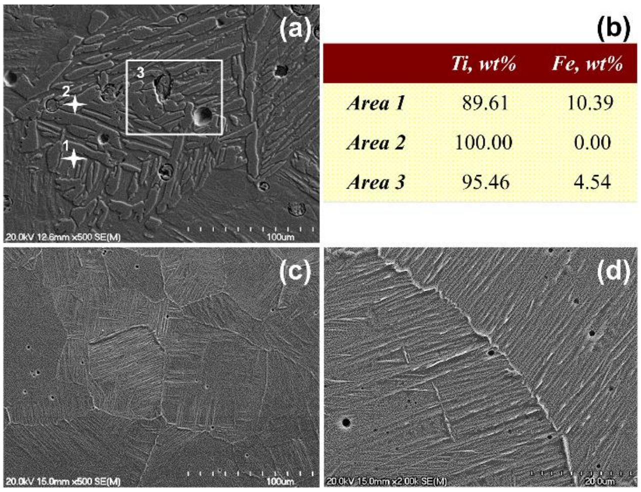
Table I. Mechanical properties of Ti-5553 and Ti-5Fe alloy processed at different conditions

	Ti-5553 alloy			**Ti-5Fe**	
	Hot pressing at 1300°C	**Extrusion at 950°C**	**Double-aging (450°C/6h+675°C/30min)**	**Microwave sintering 1300°C/2h**	**Extrusion at 1050°C**
Yield Strength (MPa)	—	1270	1435	—	1070
Ultimate Strength (MPa)	800	1370	1530	860	1090
Elongation (%)	1	3	8.5	—	1.5

Microstructure and properties of Ti-5Fe alloy

After microwaving sintering at 1300°C for 22 min, the relative density of the as-microwaved-sintered Ti-5Fe alloy was significantly improved, reaching a value of about 94%, which was slightly lower than that of the conventional vacuum-sintered titanium alloy billet.[16,17] The microstructures of Ti-5Fe alloy processed at different conditions are shown in Fig.3. For the as-microwave-sintered Ti-5Fe alloy, no undissolved Fe powder particles were observed and the microstructure consists of lamellar of α phase which is distributed in β-phase matrix (Fig.3a). The lamellar are coarser with average width of 10μm and length of 20 to 80μm. The porosity distribution shows large isolated pores with spherical appearance, confirming that significant densification occurred during microwave sintering, but the porosity level is higher than that of the hot-processed alloy. EDS analysis results indicates that Fe distribution was not homogeneous throughout the microstructure (Fig.3b). After extrusion, the plastic deformation led to elimination of the large pores left after microwave sintering (Fig.3c and d). As a result, the density of the extruded material reached almost to this of fully solid material. Although the overall grain size was still quite coarse, with a dimension of up to 120μm, the lamellar spacing was much finer than that of the as-microwave-sintered Ti-5Fe alloy. This was mainly caused by large plastic deformation induced during extrusion and fast cooling (air cooling) after extrusion.

The mechanical properties of the as-microwave-sintered and the as-extruded Ti-5Fe alloy are listed in Table. I. No yield happened for the as-microwave-sintered Ti-5Fe alloy and its ultimate tensile strength is about 860MPa. This testing results were similar to those reported in literature for vacuum sintered alloy with similar composition,[14] while the other study,[15] where controlled faster cooling was used to control the size of the α-phase lamellar, showed similar ultimate strength but significantly higher elongation to fracture of 10%. This suggests that the combination of low sintered density (about 94%), large pores, coarse lamellar microstructure and inhomogeneous Fe chemical distribution were the possible reason for lower tensile strength and no yield and ductility for the as-microwaved-sintered Ti-5Fe alloy. Furthermore, the oxygen content of the as-microwaved Ti-5Fe alloy was 0.70wt%, which was higher than the critical oxygen value of 0.33wt% for Ti-6Al-4V alloy.[14] Yan et.al [ref 14] reported that the elongation for Ti-6Al-4V (α+β) alloy was significantly reduced when the oxygen content was higher than 0.33wt% due to the formation of specific microstructure features: (1) fine α precipitates in β-Ti, (2) α_2-type cluster in α-Ti, and (3) α-β-α layered grain boundary. Ti-5Fe was a type of α+β titanium alloy, thus, another important reason to cause the as-microwave-sintered Ti-5Fe having lower elongation was the as-microwaved-sintered Ti-5Fe alloy had high oxygen content. For Ti-5Fe alloy extruded from the microwaved-sintered billet, its oxygen content was about 0.56wt.%, and the yield strength was about 1070MPa and the ultimate strength was about 1090MPa, and the elongation to fracture was about 1.5%. The strength was comparable with that of ingot Ti-6Al-4V alloy. The higher strength for the as-extruded Ti-5Fe alloy was mainly attributed to finer lamellar spacing and higher relative density comparing to that of the as-microwave-sintered Ti-5Fe alloy. The high tensile strength also demonstrated the valuable strengthening effect of the alloying element Fe. Nevertheless, the ductility of the as-extruded Ti-5Fe alloy was low, and this was mainly attributed to the high oxygen content contained in the as-extruded alloy.



	Ti, wt%	*Fe, wt%*
Area 1	89.61	10.39
Area 2	100.00	0.00
Area 3	95.46	4.54

Fig. 3. Microstructures and EDS analysis of Ti-5Fe alloy at the different conditions: (a) as-microwaved-sintered, (b) EDS analysis of the as-microwave-sintered Ti-5Fe alloy, and (c) and (d) the extruded Ti-5Fe alloy from the microwave-sintered billet along the extrusion direction.

CONCLUSION

Titanium alloys with homogeneous microstructure could be fast produced by both of induction heating plus hot pressing and microwave sintering from the elemental powder mixtures. After extrusion, the extruded Ti-5553 alloy had a higher mechanical properties, with a yield strength of 1270MPa and an ultimate strength of 1370MPa and the elongation to fracture of 3%. The double-aging significantly improved both of strength and ductility for the extruded Ti-5553 alloy. The elongation to fracture could reach 8.5% and meanwhile the strengths were increased to 1435MPa for the yield strength and 1530MPa for the ultimate strength. Microwave sintering plus extrusion could produce Ti-5Fe alloy with high strength, but the elongation was quite low because of high oxygen content contained in the microwave-sintered and post-extruded materials.

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