On microstructural evolution and mechanical properties of Ti-5Al-5V-5Mo-3Cr alloy synthesised from elemental powder mixtures

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Abstract

In this paper, we explored to prepare a multi-compositional titanium alloy, Ti-5Al-5V-5Mo-3Cr (Ti-5553), by powder compact extrusion from elemental powder mixtures, and investigate the microstructure variation during the synthesising process and after post heat treatments and the changes of mechanical properties. XRD, OM and SEM were used to analyse the phase constitutions and microstructures of the Ti-5553 alloy at different processing conditions, and tensile tests were conducted to examine their mechanical properties. The results showed that a homogeneous Ti-5553 alloy was successfully produced by powder compact extrusion from the powder mixtures, β phase was mainly contained in the hot-pressed and 1200°C-extruded Ti-5553 alloy and had an equiaxed microstructure. Different types of α phase precipitated from the β matrix after heat treatment, and this significantly changed the microstructures and improved the mechanical properties of the extruded Ti-5553 alloy, with yield strength of 1250 MPa and ultimate strength of 1300 MPa for the alloy treated at 675 ºC for 2 h, and the ductility of about 6.1% for the alloy treated at 780 ºC for 2 h.

Keywords: *Titanium alloy, Powder compact extrusion, Heat treatment, Microstructures and mechanical properties*

1. Introduction

Ti-5553 (Ti-5Al-5V-5Mo-3Cr, wt.%) alloy was a recently developed near β titanium, and it had a very good hardenability, good ductility and high strength, and a larger process window and higher working strength than Ti-10-2-3 (Ti-10V-2Fe-3Al) alloy[1-6]. These characteristics made Ti-5553 alloy had a great potential to replace Ti-10-2-3 alloy in almost all the aerospace applications and provided to use for making landing gear in Boeing 787 aircraft[1, 2]. Also the low modulus, low forging temperature and high oxidation resistance of the Ti-5553 alloy made it suitable for fabrication of even smaller aircraft parts[2].

The traditional ingot route for fabrication of titanium alloy parts usually included double- or even triple-vacuum remelting of titanium and other alloying additions, this made the titanium alloy production technology complex and expensive. Powder Metallurgy (PM) method was regarded as a feasible and cost-effective route for manufacturing milled products and near-net shaped parts, because PM route allowed significant cost reduction due to the application of a relative simple set of technological operations, including compaction and sintering, and minimal metal waste upon near-net-shape fabrication. Researchers have been used different PM approaches to prepare Ti-5553 alloy [1, 4, 7-10], such as Press-Sintering [7, 9], Hot Isostatic Pressing (HIP) [4, 8], and Selective Laser Melting (SLM) [1], etc., and the mechanical properties of Ti-5553 alloy parts produced by Press-Sintering, HIP and SLM methods were comparable to those of the alloys prepared by Ingot Metallurgy route. However, the product cost would be expensive due to the long process and high-cost facilities used. In addition, extensive research have indicated that the heat treatment has significant effects on the microstructures of near β alloy, and this further affect the resulting mechanical properties. Sadeghpor [11] investigated the heat treated Ti-5553 and Ti-4733 alloys and reported that the maximum elongation was obtained in the alloys with microstructures having lamellar-alpha phases and the microstructure containing a combination of globular and acicular alpha phase rendered the alloy showing a balance between strength and ductility. Chen et.al [12] reported that great number of very fine alpha precipitations were obtained in the new β titanium alloy, rendering the alloy to have high yield strength of 1624MPa, and Wang et.al [13] claimed that grain boundary alpha (GBα) at prior beta grain boundaries (GBs) would affect the subsequent transformation pathway and alpha texture development, eventually affecting the resulting mechanical properties.

It has been demonstrated that a fast consolidation approach, powder compact extrusion of powder mixtures of hydride-dehydride titanium powder, elemental aluminium and Al35V65

master alloy powders, could be used for consolidating the elemental powder mixtures to rapidly produce Ti-6Al-4V (wt.%) alloy with a relatively high mechanical properties [14, 15]. In this paper, we would explore the feasibility of synthesising homogeneous Ti-5Al-5V-5Mo-3Cr alloy, of which composition was more complex than that of Ti-6Al-4Valloy, by powder compact extrusion of elemental powder mixtures, and investigate the microstructural evolution of Ti-5553 alloy during the synthesising process and after post heat treatment and the variation of mechanical properties.

2. Experimental procedure

The starting materials used for making Ti-5Al-5V-5Mo-3Cr (Ti-5553) alloy by powder compact extrusion were hydride-dehydride (HDH) Ti powder (purity of 99.6% and -75μm), atomised Al powder (purity of 99.9%, -40μm), Al35V65, Al15Mo85 and Al30Cr70 master alloy powders (the particle size was smaller than 75μm, commercial purity, in weight percentage (wt.%)), supplied by Dalian Rongde Company, PR China. The powder mixture of HDH Ti, elemental Al, Al35V65, Al15Mo85, and Al30Cr70 master alloy powders, with a nominal composition of Ti-5Al-5V-5Mo-3Cr (wt.%), was first mixed for 20 hours by a roller mill at a speed of 200 rpm, then was compacted into a powder compact at 230-260 °C in air using 100-ton hydraulic press. For mixing, the ball-to-powder ratio was 2:1 and about 10mm-diameter stainless ball was used. The compaction pressure was about 400 MPa and the powder mixture was pressed to produce a cylindrical powder compacts, 56 mm in diameter and 60mm high. The relative density (the density which is relative to the theoretical density of solid materials) of the compact produced by warm compaction was about 85%. After compaction, the Ti-5553 powder compact was heated to 1300 ºC in an argon atmosphere protective chamber using induction furnace, and the protective chamber had an oxygen content of below 200 ppm. Then, the Ti-5553 compact was held at the temperature for 10 min, followed by feeding the compact into the pressing system to press into a cylindrical billet in 1-3 minutes, and the applied pressure was about 400 MPa. The measured relative density of the hot-pressed Ti-5553 alloy billet was about 97%. After that, the hot-pressed Ti-5553 billet was removed from the pressing system, reheated to 1200 °C in air using induction heating within 5 minutes, and then extruded in air to produce a Ti-5553 alloy rod. The extrusion ratio used was 9:1 and extrusion speed was about 68mm/s. The extrusion was carried out by 300-ton hydraulic press (XJ 300), produced by Wuxi Yuanchang Machinery Co. Ltd, PR China. After extrusion, the as-extruded Ti-5553 alloy was heat treated at (1) 675 °C for 2 h and (2) 780 °C for 2 h, respectively, using muffle

furnace. The heat rate for the heat treatment was about 6°C/min, and the temperature was held for 2 hours once the as-extruded Ti-5553 alloy samples were heated to the desired temperature, then the heat treated samples kept staying in the muffle furnace and were cooled to room temperature with furnace.

X-ray diffraction (XRD) analyses were conducted using Philips X'Pert machine with Cu Kα radiation ($\lambda = 0.154157$ nm) to determine the phase constitution of the hot pressed, as-extruded and as-heat-treated Ti-5553 alloy materials. Optical microscopy (OM) (Olympus BX60) and scanning electron microscopy (SEM) (HITACHI S4700) were used to examine the microstructures of the hot-pressed billet, as-extruded and as-heat-treated alloys. The ground and polished metallographic surfaces of the samples were etched in a modified Kroll's reagent consisting of 2 vol% HF (35vol% concentration, Sigma Aldrich), 4 vol% HNO_3 (70% concentration, Ajax Finechem) 94 vol% H_2O. Tensile tests were conducted at room temperature using an Instron (INSTRON 4204) universal testing machine, and dog-bone shaped specimens were cut from the hot-pressed, as-extruded and as-heat-treated alloys and had a rectangular cross section of 2 mm×2 mm and gauge length of 20 mm. The strain was measured using an extensometer with a gauge length of 10mm. The strain rate of the tensile testing was $1\times10^{-4}s^{-1}$.

3. Results and discussions

Fig. 1 shows x-ray diffraction patterns of the Ti-5553 alloys processed at different conditions. For the 1300 ºC-hot-pressed Ti-5553 billet and the 1200 ºC-extruded Ti-5553 rod from the hot-pressed billet in air, only β peaks appeared in the XRD patterns, indicating that the phase constitution for the hot-pressed billet and the as-extruded rod were mainly composed of β phase, and no α phase or only very small amount of α phase existed in both of the hot-pressed billet and as-extruded rod. Because the β-trans temperature for Ti-5553 alloy was about 845-870 ºC [16-18], the temperature for hot pressing (at 1300 ºC) and extruding (at 1200 ºC) of the Ti-5553 compact and billets were much higher than the β-trans temperature. Moreover, the cooling rate of hot pressing (cooling in flow argon) and extrusion (cooling in air) was faster than that of furnace cooling (close to equilibrium condition), the precipitation of α phase from β phase was significantly supressed during the process of hot pressing and extrusion. This led to the hot-pressed billets and the extrusions were mainly composed of β phase. The phase transformation was triggered by the heat treatment, both α and β peaks were appeared in the XRD patters of the 675 ºC- and 780 ºC-heat-treated Ti-5553 alloys, as shown in Fig. 1b and c.

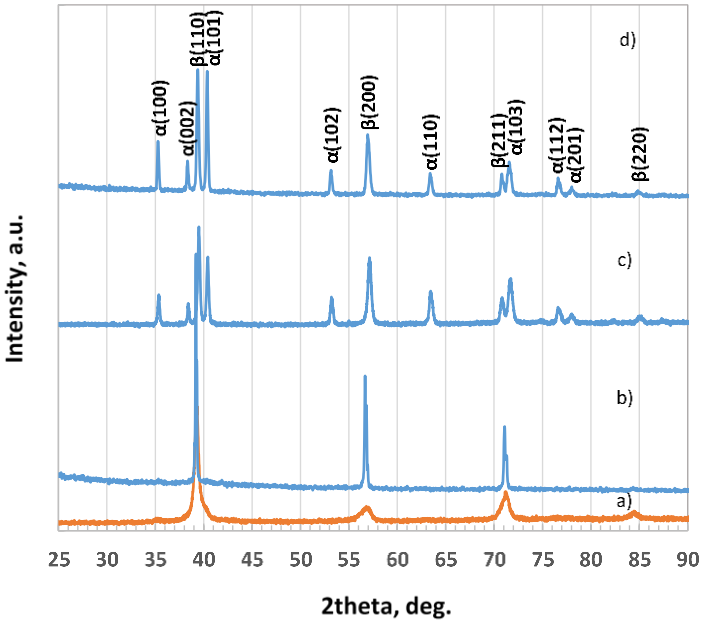


Fig. 1. XRD patterns of Ti-5553 alloy: a) hot-pressed at 1300 ºC after holding the temperature of 10 min, b) extruded the hot-pressed billet at 1200 °C in air, c) heat treated the extruded material at 675 °C/2 h, and d) heat treated the extruded at 780 °C/2 h.

In addition, the intensity of α peaks was higher and stronger in Fig. 1 d than that in Fig, 1c, indicating that the amount of α phase was higher in the 780 ºC- heat-treated Ti-5553 alloy rod than in the 675 ºC- heat-treated alloy. This was because α phase nucleation and growth was significantly dependent on the heat treatment temperature, and the higher temperature, the faster nucleation and growth, and the more α phases will be formed in the heat treated Ti-5553 alloy.

No Al-Mo, Al-Cr and Al-V master alloy peaks appeared in the XRD patterns, this indicated that there was a very low possibility for the master alloy powder particles left in the hot-pressed billet after holding the Ti-5553 compact at the temperature of 1300ºC for 10min and hot pressing. This speculation was further confirmed through microstructure observation and EDS analyses, as shown in Fig.2 and 3, respectively. It can be clearly seen, from Fig. 2, that the microstructures of the hot-pressed billet were mainly composed of equiaxed grains surrounded by small amount of flake-shaped structure. The size of the flake-shaped structure was very small compared to that of the equiaxed grains, with dimensions of 1-2 µm in length and about 0.5µm thick. No obvious undissolved master alloy powder particles were observed. Also, the porosity was less obvious and just a few closed pores were left and located inside of the equiaxed grains.

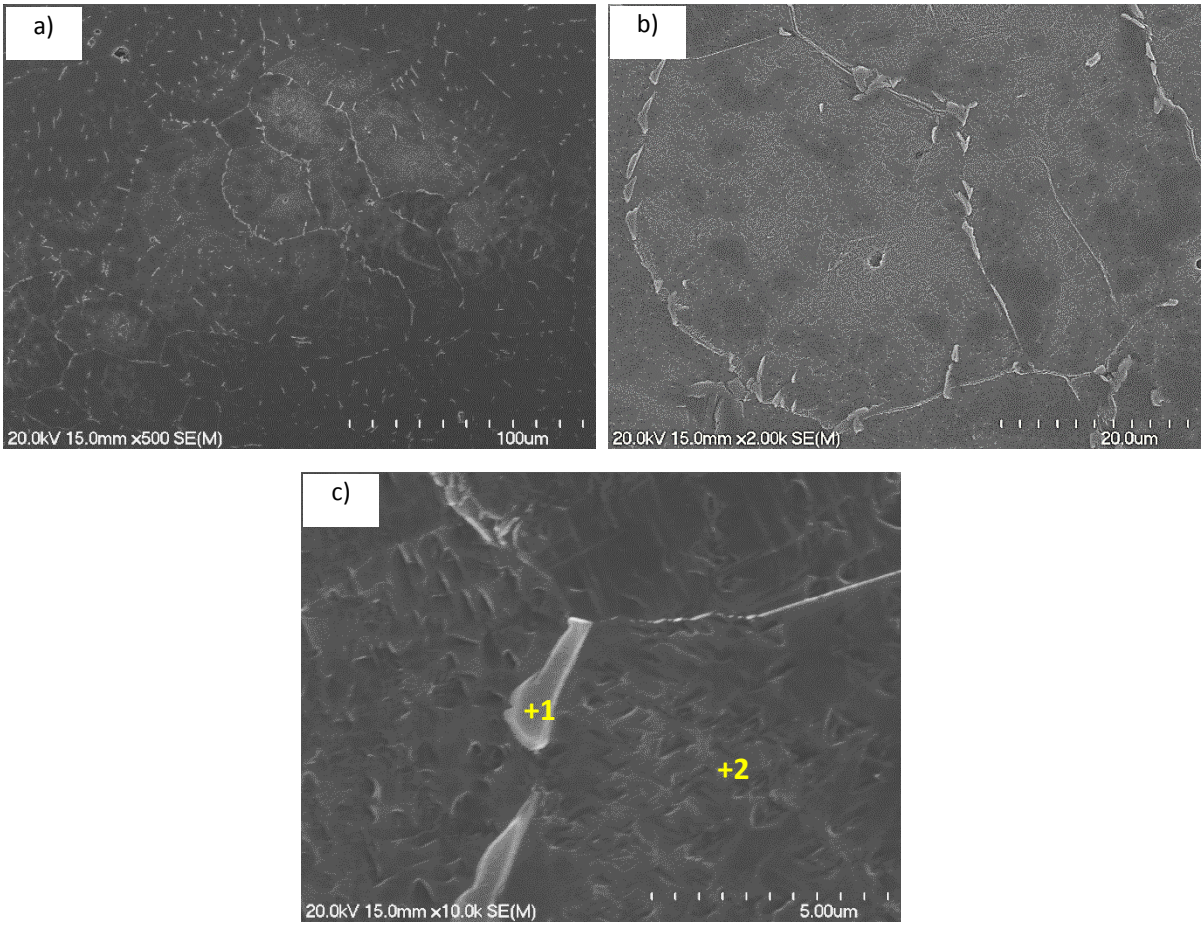


Fig. 2. SEM microstructures of the hot-pressed Ti-5553 billet at 1300 ºC after holding the temperature of 10 min.

EDS mapping analysis showed that the distribution of chemical composition of Ti, Al, V, Mo and Cr of the hot-pressed Ti-5553 billet was relatively homogeneous in general, as shown in Fig.3, this further indicated that no obvious undissolved master alloy powder particles remained in titanium matrix. However, the results of EDS point analysis performed at the different positions in Fig.2c, as listed in Table 1, showed that the chemical compositions were slightly different for the flake-shaped structure compared to that of the equiaxed grains, although both of them were β phases. The contents of V, Cr and Mo were relatively higher in the flake-shaped structure, with the value of 8.01wt%, 2.26wt% and 4.39wt%, and the content of Al was lower, having the value of 2.46wt%. This may be caused by localised chemical element rich in the microstructure of the hot-pressed Ti-5553 billet. Because the whole alloying process of forming Ti-5553 alloy from the powder mixtures during the process of hot pressing included: (1) Al-V, Al-Mo and Al-Cr master alloys dissolving in the titanium matrix, and (2) Al, V, Mo and Cr diffusing from higher content positions, where were close to the original

dissolved position of the relevant master alloy powder particles in titanium matrix, to low content positions.

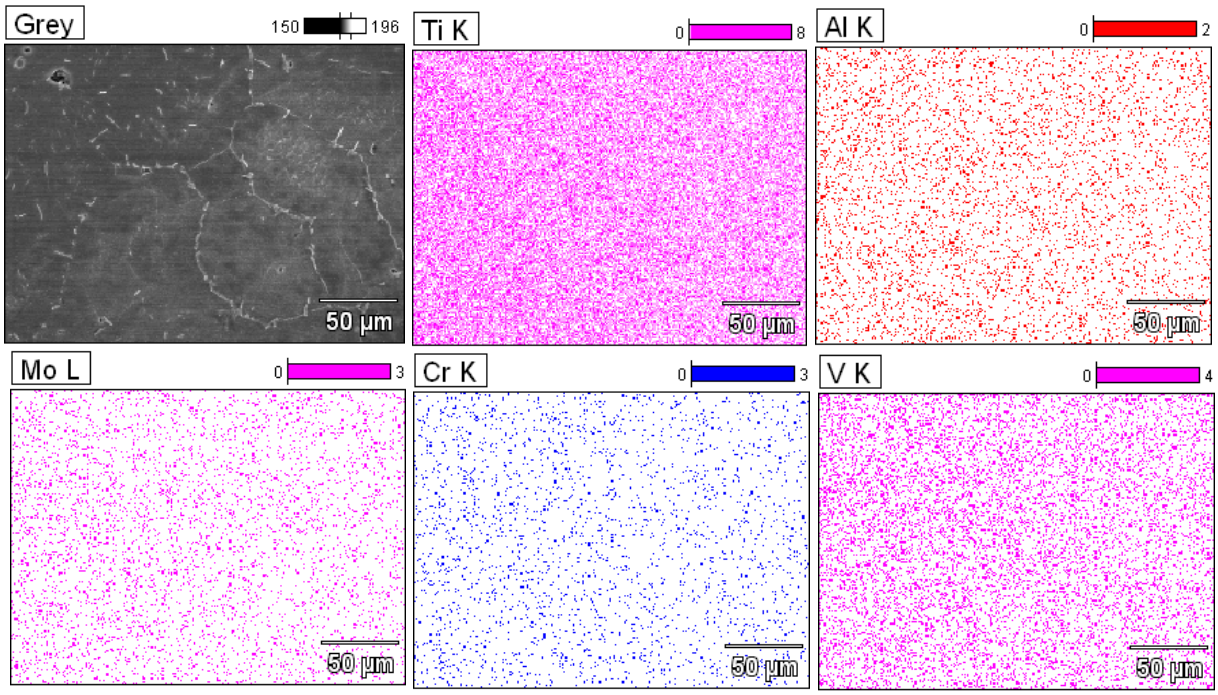


Fig. 3. EDS mapping analysis of the hot-pressed Ti-5553 billet at 1300 ºC after holding the temperature of 10 min.

Table 1. Chemical composition of different positions in Fig.2c

Position	Ti (wt.%)	Al(wt.%)	V(wt.%)	Mo(wt.%)	Cr (wt.%)
1	82.88	2.46	8.01	4.39	2.26
2	84.27	3.22	7.02	4.13	1.36

Master alloy particles were unlikely to completely dissolve in the titanium matrix during the period of rapid induction heating the Ti-5553 compact to 1300 °C (less than 5min) and holding the compact at the temperature of 1300 °C for 10min. Because the atomic diffusion, for dissolving the master alloy particle and homogenising the chemical composition, was a slow process in pressureless sintering, and it was dependent on both of sintering temperature and sintering time. Extensive research have been started to make titanium alloys through pressing and pressureless sintering of blended elemental powder mixtures. It suggested that the sintering temperature should be higher than 1200 °C and sintering time should be no less than 2 hours (excluding the heating time) in order to ensure the master alloy and/or elemental powder particles to completely dissolve into the titanium matrix [7, 19-21]. From the pressure-assisted sintering theory, applying pressure (about 400 MPa) on the 1300ºC-induction-heated Ti-5553

compact would: (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. This was the reason why there was no obvious undissolved powder particles left after hot pressing the Ti-5553 compact at 1300 °C. Because the holding time for the Ti-5553 powder compact at 1300 °C was only 10min, the chemical composition may not be completely homogenised in such short time, although the master alloy powder particles were able to completely dissolve into the titanium matrix in 10 minutes [14, 15]. Thereby it was supposed that the position of the flake-shaped structure were close to where the master alloy powder particles were completely dissolved in titanium matrix. Fig. 4 shows the microstructures of Ti-5553 alloy extruded at 1200 °C from the hot-pressed billet in air. After extrusion, the microstructures were mainly composed of equiaxed β phase grains, the flake-shaped structure surrounded the equiaxed grains were completely disappeared, and the equiaxed β grain size was in the range of 30-150μm.

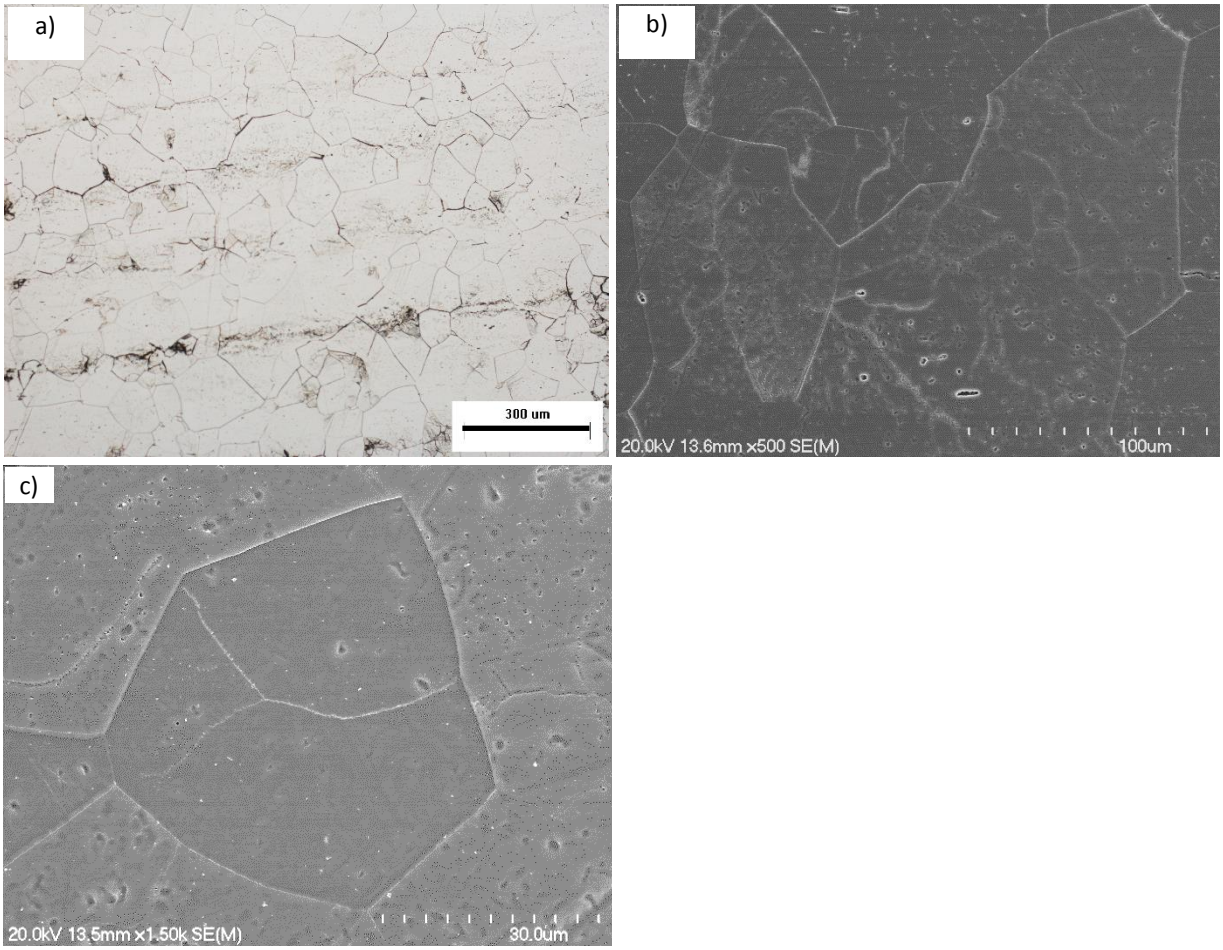


Fig. 4. Microstructures of the Ti-5553 alloy extruded from the hot-pressed billet at 1200 ºC in air: (a) Optical image and (b) and (c) SEM images.

As we discussed above, the existence of the flake-shaped structure was caused by localised chemical element rich after the hot pressing. Before extrusion, the Ti-5553 hot-pressed billet was reheated to 1200 °C first, and then the billet was transferred to the extrusion machine to extrude into a rod. Thus, the atomic diffusion was activated again and further promoted by the storied strain energy during the process of reheating the hot-pressed Ti-5553 billet. Furthermore, the significant plastic deformation was introduced during extrusion, the atomic diffusion was significantly accelerated, so that the chemical composition of the extruded Ti-5553 alloy was further homogenised. Therefore, the flake-shaped structure was disappeared in the microstructure of the as-extruded Ti-5553 alloy.

SEM microstructures of the heat treated Ti-5553 alloy are presented in Fig.5. When the as-extruded Ti-5553 alloy was heat treated at 675 °C for 2 h, α phases were precipitated from the β phase matrix, all the original β grains of the as-extruded Ti-5553 alloy were completely embedded with α precipitations, and there was no precipitation free zone throughout the microstructures of the heat treated Ti-5553 specimens. There were three different shapes of the precipitated α phases: (1) discontinued grain boundary α phase, with a thickness of less than 0.5μm; (2) acicular α phase, which were close to the grain boundaries, with the length of less than 5μm and the thickness of less than 0.5μm; and (3) fine particle α precipitations, with a size of smaller than 0.5μm. When the heat treatment temperature was increased to 780 ºC, the α phases which precipitated from β phase matrix were getting coarser and larger than that of the alloy heat treated at 675 ºC. Four different types of α phases could be observed: (1) discontinued grain boundary α phase, with a thickness of near 2μm; (2) lath α phase, with the length of up to 20μm and the thickness of up to 1μm; (3) acicular α phase, its dimension was similar with that of 675 ºC-heat-treated Ti-5553 alloy; and (4) globular α phase, with a dimension of about 5μm in diameter. 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 the post heat treatment. So that 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 [22]. As seen in Fig. 5, acicular structured α phases were close to the original β boundaries, and both fine particle α phases and acicular α phases were located within β grains. These types of microstructures would suggest that the fine particle α phase was first precipitated from β grain boundaries and within β grains, and then the fine particle α precipitations was acted as a precursor for nucleation and growth of the acicular structured α phase, the similar findings could be found in elsewhere [11].

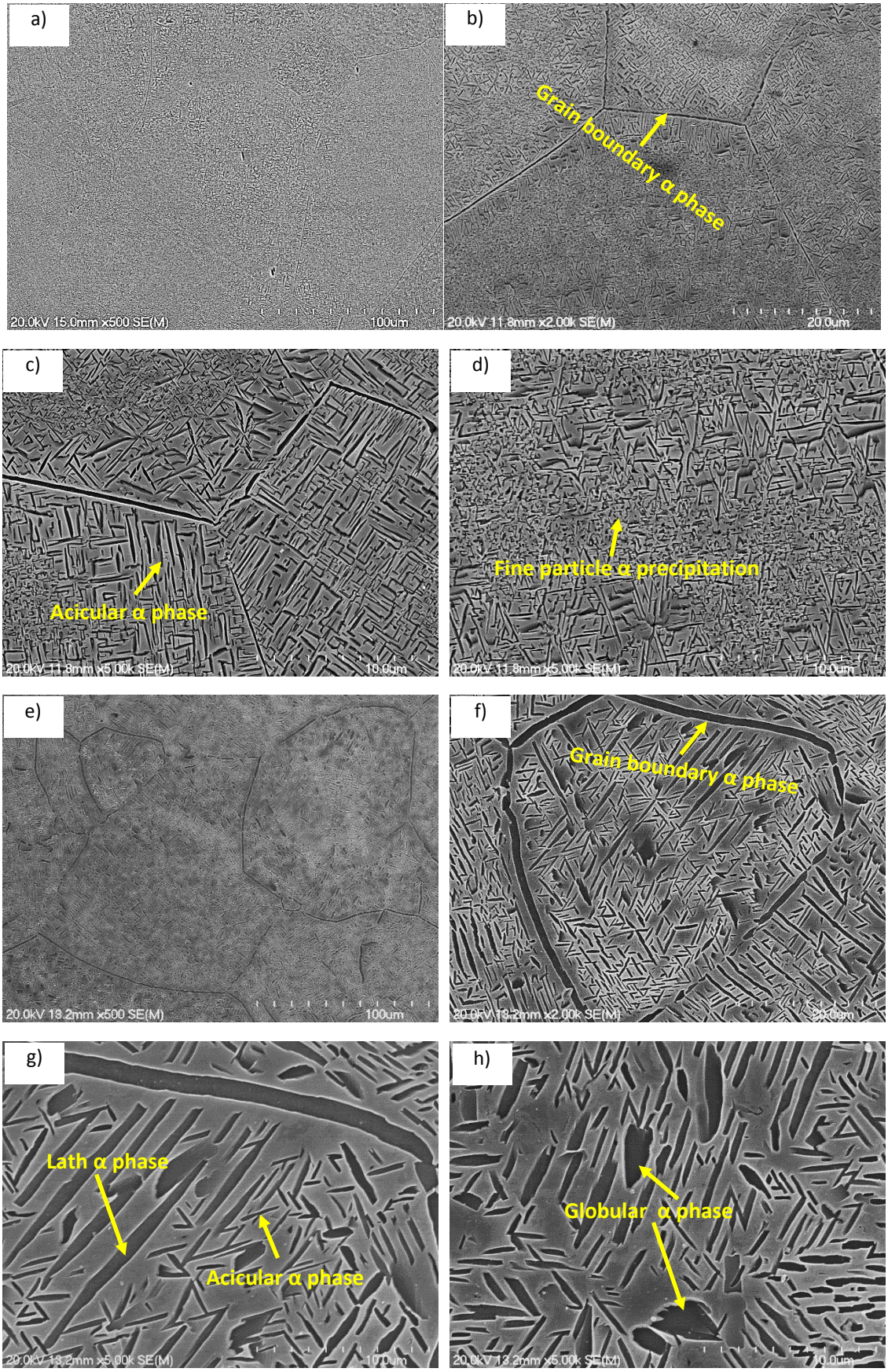


Fig. 5. SEM microstructures of the Ti-5553 alloy heat treated at: (a)-(d) 675 ºC for 2 h and (e)-(h) 780 °C for 2 h (dark regions are α phase).

The driving force for α precipitation nucleation and growth of the as-extruded Ti-5553 alloy was also dependent on the post heat treatment temperature. The higher the treated temperature, the faster nucleation and growth. This was the main reason why the thickness and dimensions of the grain boundary α phase and acicular structured α phase in the Ti-5553 alloy treated at 780 ºC were much thicker and greater than that in the alloy treated at 675 ºC. Furthermore, partial acicular structured α phase were getting to be coarsened and globularised to form lath α phase and globular α phase in the as-extruded Ti-5553 alloy when the heat treatment temperature was increased to 780 ºC. This was because: (1) the aspect ratio (width/thickness) of fine acicular structure α phase was large, and it was kinetically not very stable at higher temperature and tended to be coarsened during the heat treatment. When the aspect ratio of the coarsened α acicular was getting small, the globularisation would predominate according to lamellar globularisation mechanism dominated by fault migration and/or termination migration in titanium alloy [23, 24]; (2) a gradient in chemical potential/solute concentration existed between the curved edge of an acicular structure and the flat α/β interphase boundary, leading to form the driving force for atomic diffusion which associated with the temperature. Therefore, the completeness and the rate of globularisation increased with heat treatment temperature, and this explained why the globular α phase was only observed in the Ti-5553 alloy treated at 780 ºC, and rather than at 675 ºC.

Typical stress-strain tensile curves of the Ti-5553 alloys processed at different conditions are shown in Fig. 6. It can be clearly seen that the hot-pressed Ti-5553 alloy had very poor mechanical properties, and no yield occurred. The ultimate strength was about 800MPa and the elongation was just over 1%. After extrusion, the mechanical properties of the as-extruded Ti-5553 alloy was obviously improved compared to that of the hot-pressed alloy, with a yield strength of about 875 MPa and an ultimate strength of about 905 MPa, and the elongation was about 2.2%. The heat treatment had an important impact on the as-extruded Ti-5553 alloy and made significant contribution to increase the mechanical properties. When the as-extruded Ti-5553 alloy was heat treated at the condition of 675 ºC for 2 h, the yield strength was increased to about 1250 MPa, and the ultimate strength was about 1300 MPa. This was about 43% increase in yield strength and about 44% increase in ultimate strength. The elongation almost remained unchanged, with a value of 2.1%. Increasing the heat treatment temperature to 780 ºC, the yield strength and ultimate strength were decreased to 1120 MPa and 1235 MPa, respectively, comparing to that of Ti-5553 alloy treated at 675 ºC for 2 h, however, the elongation was increased to 6.1%.

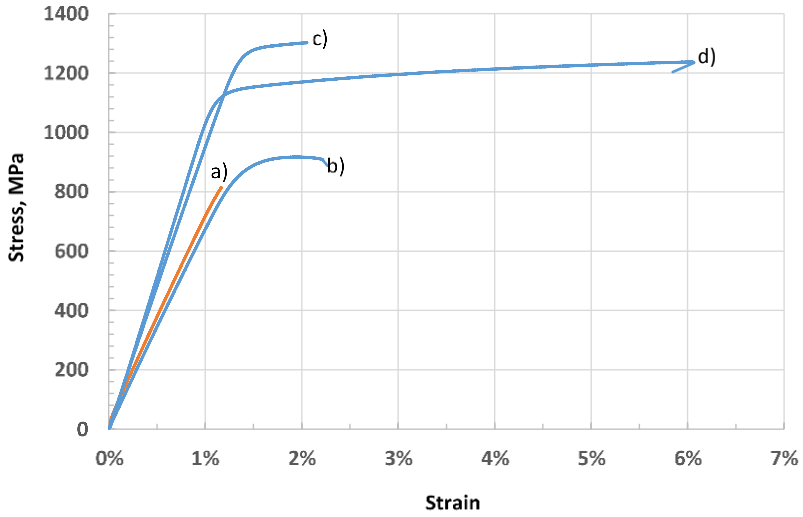
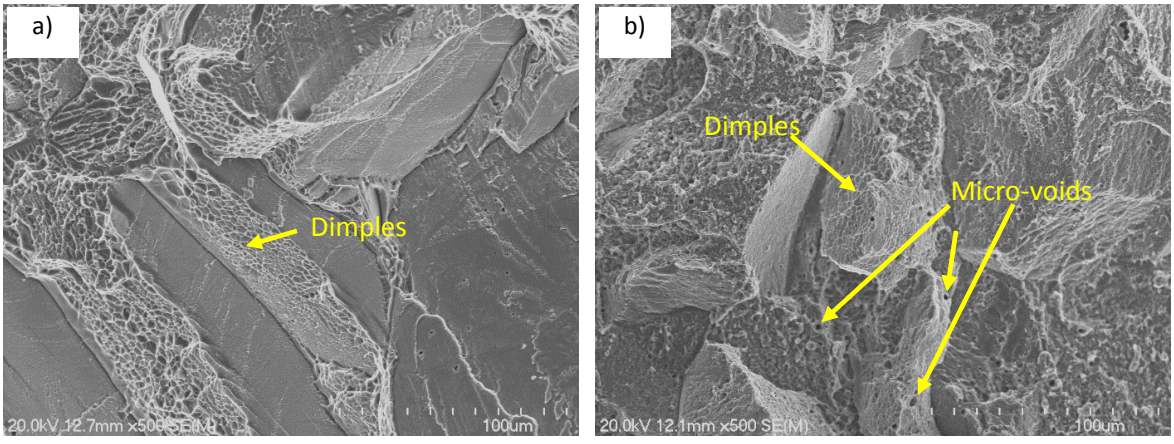


Fig. 6. Stress-strain curves of Ti-5553 alloy: a) hot-pressed at 1300 ºC after holding the temperature of 10 min, b) extruded from the hot-pressed billet at 1200 ºC in air, c) heat treated at 675 ºC for 2 h and d) heat treated at 780 ºC for 2 h.

Fig. 7 shows the fracture surface morphologies of the Ti-5553 alloy processed at different conditions. It showed that the as-extruded Ti-5553 alloy had a mixed mode of transgranular brittle fracture with facet surfaces besides minor ductile fracture with shallow dimples (Fig.7a), and a lot of micro-voids, dimples and intergranular brittle fracture are exhibited in Fig.7b. A mix mode of large percentage of ductile fracture, with fine and deep dimples, and small percentage of intergranular brittle fracture, can be observed in the 780 ºC-heat treated Ti-5553 alloy treated at 780 ºC, as shown in Fig.7c.



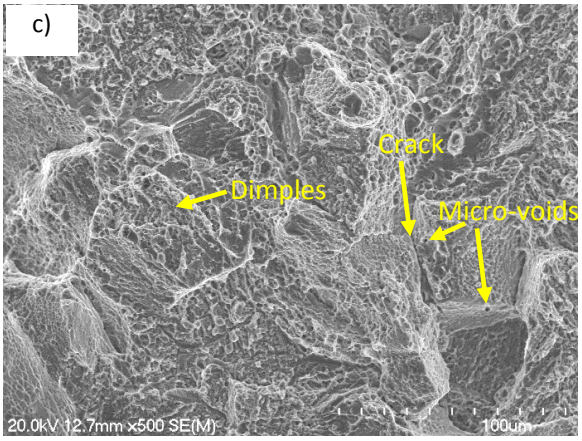


Fig. 7. Fracture surface morphologies of Ti-5553 alloy: a) extruded from the hot-pressed billet at 1200 ºC in air, b) heat treated at 675 ºC for 2 h and c) heat treated at 780 ºC for 2 h.

The low mechanical properties for the hot-pressed Ti-5553 alloy were supposed to originate from the equiaxed microstructures and unstable single β phases caused by fast cooling after hot pressing. The flake-shaped structure which located at the grain boundaries also deteriorated the mechanical properties for the hot-pressed billet and acted as a crack origin due to a high strain concentration was introduced during the deformation. For the as-extruded Ti-5553 alloy, although the properties were better than that of the hot-pressed billet, they were still quite low because of the equiaxed microstructure with single β phase which was soft and did not have effective pinning effect on the dislocation motion. α precipitations in β titanium matrix had significant influence on the mechanical properties of the heat treated Ti-5553 alloy, and it will contribute to both of the strength and ductility because [3, 6, 11, 25] (1) fine α phase precipitations, such as fine particles and/or acicular structure, were harder than β phase and hardly to be deformed; and (2) α/β interfaces acted as dislocation barriers, causing pinning effect on the dislocations; (3) larger α phases, such as lath α, grain boundary α and globular α, could be deformed by slipping and shearing mechanisms so that the dislocations could be activated and accumulated within them; (4) twinning could be also activated in large α phase during the deformation; and (5) α precipitating from β phase made the β matrix relatively stable. Thereby, the Ti-5553 alloy treated at both 675 ºC and 780 ºC exhibited much higher strength than that of the as-extruded alloy. Especially for the Ti-5553 alloy treated at 675 ºC, it had the highest strength, with a value of 1250 MPa of yield strength and 1300MPa of ultimate strength, because the very fine acicular structured α, particle α and thin discontinued grain boundary α phases were hard to be deformed and they hindered the movement of the dislocation within β matrix, making the Ti-5553 alloy stronger. Comparing to the Ti-5553 alloy treated at 675 ºC,

the strength of 780 ºC-heat-treated Ti-5553 alloy was decreased, but the elongation was significantly increased. This was because the coarsened α phases, such as lath α and globular α, could be deformed and even the mechanical twining could be active in them, so that producing more ductility for the alloy.

Since the as-extruded Ti-5553 alloy was composed of β phase and had an equiaxed microstructure, crystal was easy to be separated along a certain plane during deformation, cleavage fracture was the primary fracture characteristic for this type of microstructure, and the fracture surface was flat, smooth and featureless. Because the deformation in α phase and β phase was different, strain concentration was introduced, and this led to form micro-voids at the α/β interfaces. The micro-voids could coalescence and grow into the micro-cracks [11, 14], eventually causing the Ti-5553 alloy fractured along the grain boundary α phase to form intergranular fractures. Due to the coarsened α phase could be introduced more deformation before fracture than that of fine α phase, more tear and pulling phenomenon would be occurred in the Ti-5553 alloy treated at 780 ºC than that at 675 ºC, so that more ductile fracture surfaces (dimples) could be observed in Fig.7c than that in Fig.7b.

4. Conclusions

A multi-compositional titanium alloy, Ti-5Al-5Mo-5V-3Cr, with homogeneous composition distribution and microstructures, was successfully and rapidly prepared by powder compact extrusion of elemental powder mixtures, this expanded the thermomechanical powder consolidation process to complex compositional systems. The yield strength and ultimate strength for the as-extruded alloy were about 875 MPa and 905 MPa, respectively, and the elongation was about 2.2%. Heat treatment could significantly increase the mechanical properties of the as-extruded alloy, the strength of Ti-5553 alloy treated at 675 °C for 2 h could reach 1250 MP of yield strength and 1300 MPa of ultimate strength, and the ductility of the 780 ºC-heat-treated Ti-5553 alloy was significantly improved, with a value of about 6.1%.

5. Acknowledgements

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