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Improvement in Tensile Property of Ti64 Composites with VGCFs Fabricated by Powder Metallurgy†

PRIPANAPONG Patchara*, MIMOTO Takanori*, LI Shu-feng, IMAI Hisashi**, UMEDA Junko***, KONDOH Katsuyoshi******

Abstract

Ti-6Al-4V (Ti64) composite reinforced with vapor grown carbon fibers (VGCFs) was fabricated by a powder metallurgy (P/M) route in this research. Ti64 powder was mixed with VGCFs in four compositions such as 0.1, 0.2, 0.3 and 0.4 wt%, and consolidated by spark plasma sintering (SPS), followed by a hot extrusion process. The microstructure observation was carried out by optical microscopy (OM) and scanning electron microscopy (SEM). The microstructure was changed from full lamellar to bimodal after the addition of VGCFs. Almost all VGCFs reacting with Ti and turned into $Ti_6C_{3.75}$ *compounds. Hardness was improved by the effect of solid solution strengthening of carbon in the Ti matrix and reached the maximum value of 495.8 Hv. 0.2%YS and UTS were also significantly increased after adding 0.1wt% of VGCFs, but never much improved when more VGCF content was applied. Ductility of the sample was affected by a change in microstructure when 0.1 wt% of VGCFs was added, bimodal microstructures showed higher ductility compared to full lamellar structures. The relationship between fracture morphology after tensile testing and original microstructure could also be observed. At 0.1wt% of VGCFs, a good balance between strength and ductility of Ti64/VGCFs composite can be improved simultaneously.*

KEY WORDS: (Ti64), (VGCFs), (Powder metallurgy), (Spark plasma sintering), (Solid solution strengthening)

1. Introduction

Ti and its alloys offer high specific strength, fracture toughness and good corrosion resistance as a hallmark properties. These materials have been utilized in many industrial fields such as automobile and aerospace components, gas condenser pipes in electrical power plants and human body prosthesis [1, 2]. Ti64 as an economical alloy grade has been used more than 50% among Ti alloys in many industries. Spark plasma sintering (SPS) is one of the promising hot pressing methods selected as a fabrication method in this research. High current and low voltage was applied for heat generation during sintering. In additional, oxide surface on powder particles and adsorbed gases in samples will be removed during SPS due to high applied pressure. These provide a good bonding between powder particles and high density samples could be obtained.

Hardness and mechanical properties of Ti or its alloys can be improved by composites with reinforced materials, this method is widely used in powder metallurgy field. Many researchers have focused on fabrication of metal matrix composites (MMCs) of Ti64 by composites with various reinforcing particles such as TiB, TiC or SiC [3-5] with a simple fabrication method and high strength improvement. VGCFs is a reinforcing material similar to CNTs but larger in size and much cheaper compared to CNTs. The cost effective composite material between VGCFs and Ti was successfully fabricated by Li et al. [6], and it was shown that mechanical strength of extruded materials was significantly improved with decreases in ductility. Furthermore, very few studies on Ti64 alloy composites reinforced with VGCFs have been reported. Normally, ductility will decrease with increasing in strength of material as a trade-off [7, 8]. However, this work could improve both properties simultaneously by the effect of solid solution strengthening and α stabilization by carbon element originating from the VGCFs.

2. Experimental

Ti-6Al-4V gas atomization powder (Osaka Titanium Technology Co. Ltd.,TILOP64-45) with mean particle diameter 45 μm (Fig. 1a) and vapor grown carbon fiber (Showa Denko K.K.,VGCFs™) with diameter and length

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of 0.15 and 8μm (**Fig. 1b**) were selected as raw materials in this research. The chemical compositions of Ti64 powder are listed in **Table 1**.

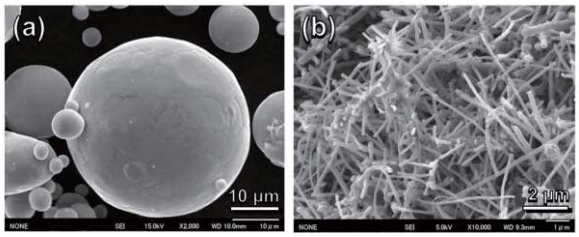

Fig. 1 SEM micrograph of (a) Ti-6Al-4V atomization raw powder and (b) vapor grown carbon fibers (VGCFs).

Table 1 Chemical compositions of Ti-6Al-4V atomization raw powder (wt%).

Material	Al	V	Fe	C	O	N	H	Ti
Ti-6Al-4V	6.12	4.48	0.03	0.01	0.13	0.014	0.006	Bal.

The monolithic Ti64 and Ti64/VGCFs composites were fabricated by the P/M process. Ti64 powder was mixed with 0.15g of cleasafe oil by table milling equipment at 90 rpm for 1hr and subsequently mixed with VGCFs by a rocking mill for 2hr. Mixed powders were prepared in four compositions as 0.1, 0.2, 0.3 and 0.4 wt%. Ti64/VGCFs mixed powder was pre-compacted at 20 MPa with a hydraulic hand press before SPSed (Syntech CO. SPS103S) at 1000ºC for 1hr under an applied pressure of 30 MPa in vacuum atmosphere. The SPS billets were pre-heated at 1150ºC for 10 min before extrusion by a 200 ton press machine (SHP-200-450, Shibayamakikai Co.) from diameter of 42 to 12 mm. SPSed and extruded samples were ground with SiC paper, polished with Al_2O_3 solution and etched with Kroll's etchant (H_2O:HNO_3:HF = 100:5:1). Microstructure observation and phase analysis on SPSed and extruded samples were carried out by optical microscope, SEM (JEOL JSM6500F) equipped with EDS and XRD (Lab X XRD-6100 Shidmazu). Hardness of SPSed and extruded samples were measured by a Vickers hardness tester with 20 points of testing per sample. Tensile specimens with 20 mm of gauge length and 3 mm in diameter were fabricated from extruded rods, and their tensile properties were evaluated using a universal tensile testing machine (Autograph AGX 50kN, Shidmazu) at a strain rate 5×10^{-4} s^{-1}. The fractured surfaces of tensile samples were also investigated by SEM.

3. Results and Discussion

3.1 Microstructure and Phase analysis

Figure 2a shows VGCFs which were coated on Ti64 particles, and a higher magnification photo indicates individual VGCFs exist at powder surface homogeneously (**fig. 2b**). However, VGCFs were starting to agglomerate as some local particles when the VGCFs content reached 0.2 wt% (**fig. 2c**) because of its large volume. Monolithic Ti64 and Ti64/VGCFs composites show high relative density with over 97% by applying high temperature and pressure.

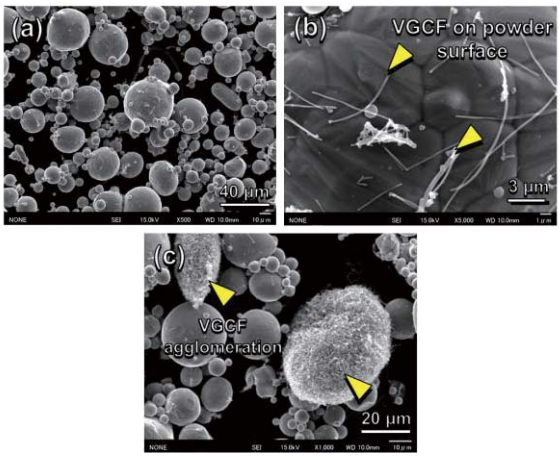

Fig. 2 Ti-6Al-4V atomization raw powder coated with 0.4 wt% of VGCFs (a) Ti64 coating with VGCFs (b) un-bundled VGCFs on Ti64 powder surface and (c) VGCFs agglomeration as some local particles.

XRD results of monolithic Ti64 and Ti64 composites are shown in **fig. 3**. The monolithic Ti64 shows only a peak of Ti and Al, and Ti64 composites clearly reveal carbide phases identified as $Ti_6C_{3.75}$ by EDS. Diffraction pattern of $Ti_6C_{3.75}$ can be observed in samples in which VGCFs content is over 0.1 wt% because no $Ti_6C_{3.75}$ peak can be observed in Ti64+0.1wt%VGCFs. A ratio of intensity between main peak of $Ti_6C_{3.75}$ and α-Ti was calculated (**Table 2**).

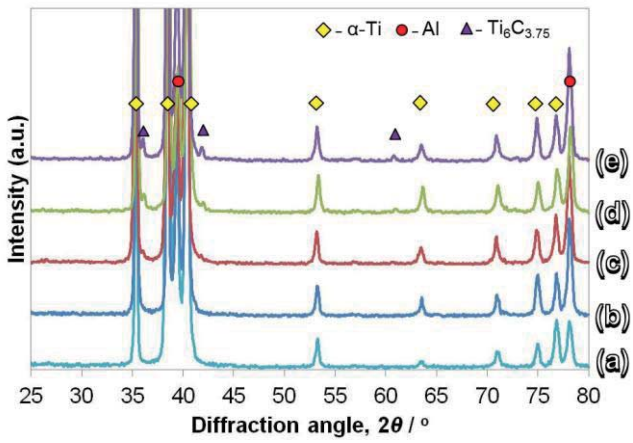

Fig. 3 XRD diffraction patterns of Ti64 and Ti64 composites at various compositions (a) monolithic Ti64, (b) Ti64+0.1wt%VGCFs, (c) Ti64+0.2wt%VGCFs, (d) Ti64+0.3wt%VGCFs and (e) Ti64+0.4wt%VGCFs after hot extrusion.

Table 2 Ratio of intensity between main peak of $Ti_6C_{3.75}$ and α-Ti of Ti64+VGCF composites.

Composition	Intensity (a.u.)		Intensity ratio ($I_{Ti_6C_{3.75}}$/ $I_{\alpha\text{-Ti}}$)
	α-Ti matrix (1 0 1)	$Ti_6C_{3.75}$ (0 1 4)	
0.1VGCF	1710	224	0.13
0.2VGCF	1822	431	0.24
0.3VGCF	1954	645	0.33
0.4VGCF	1957	846	0.43

This ratio is related to an increasing of VGCFs content by proportion. Microstructures of Ti64 and Ti64+VGCFs after SPS are shown in **fig. 4**, no pores can be observed in each sample. Microstructure of monolithic Ti64 shows large prior β grains of 290 μm (**fig. 4a**), and high magnification image (**fig. 4b**) shows an α stripe at grain boundaries and lamellar colonies inside grains [9, 10]. Microstructures of Ti64 composite sintered at the same temperature are shown in **fig. 4c-f**. It shows different microstructures compared to the monolithic Ti64. Microstructure of Ti64 composite is a bimodal composes with primary α lamellar and secondary α equiaxed. This microstructure is observed when sintering temperature is lower than the β transition temperature (1273 K). This implies that changes in microstructures occur by the effect of α-stabilization from additive VGCFs [11]. Size of primary α lamellar and secondary α equiaxed is approximate 10 μm. Ti64+0.1wt%VGCF shows no $Ti_6C_{3.75}$ or VGCFs particle in the matrix (fig. 4c) corresponding to the XRD result. It indicates that the solubility limit of carbon in Ti64 is approximately 0.1 wt%, and this content was also confirmed by F. Perdrix et al [12]. Increasing VGCFs content over 0.1 wt%, $Ti_6C_{3.75}$ compounds are observed in the matrix. Amount of $Ti_6C_{3.75}$ compound was increased by increasing VGCFs content (fig. 4d-f). Microstructures of Ti64 and Ti64 composite after hot extrusion in the transversal direction are shown in **fig. 5**. The white phase and gray matrix phase observed clearly by SEM is β-Ti and α-Ti, respectively. Microstructure of the monolithic Ti64 after hot extrusion shown in fig. 5a is fully lamellar, the same as before extrusion. However, the grain size of prior β was drastically decreased from 295 to 115 μm. In the same manner as monolithic Ti64, microstructure of Ti64 composite was still bimodal after extrusion. Size of primary α lamellar and secondary α equiaxed were decreased from 10 to 3 μm. The agglomeration of VGCFs could be observed in Ti64+0.2wt%VGCFS and samples which contained VGCFs higher than 0.2 wt% (**fig. 5d**). VGCFs agglomeration after powder mixing as some local particles took responsibilities for VGCFs agglomeration after SPS.

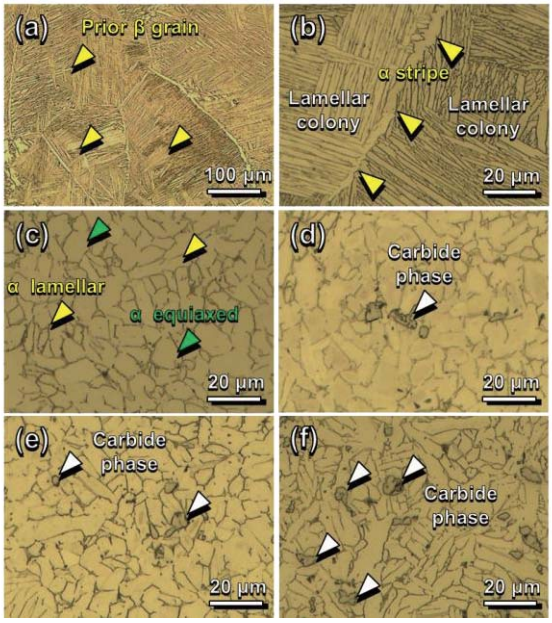

Fig. 4 Microstructure of samples after SPS at 1273 K for 0.5 hr: (a) monolithic Ti64 (low magnification) (b) monolithic Ti64 (high magnification) (c) Ti64+0.1wt%VGCFs (d) Ti64+0.2wt%VGCFs (e) Ti64+0.3wt%VGCFs (f) Ti64+0.4wt%VGCFs.

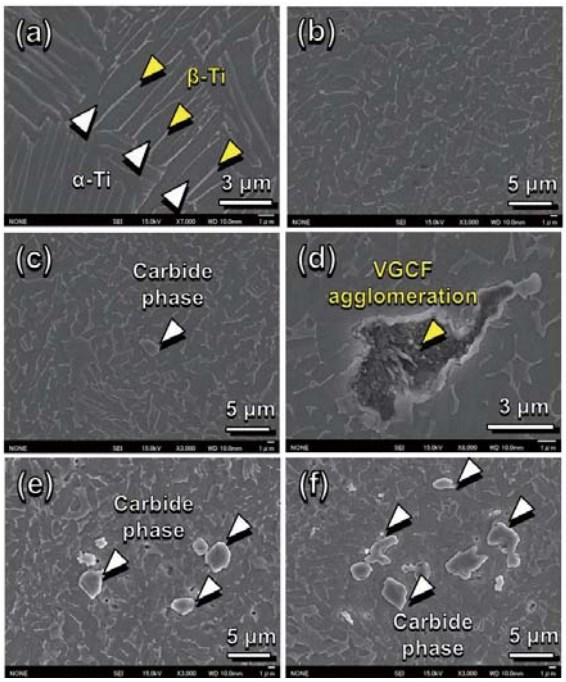

Fig. 5 SEM image of samples after hot extrusion in transversal to extrusion direction (a) monolithic Ti64 (b) Ti64+0.1wt%VGCFs (c) Ti64+0.2wt%VGCFs (d) agglomeration of VGCFs in Ti64+0.2wt%VGCFs (e) Ti64+0.3wt%VGCFs and (f) Ti64+0.4wt%VGCFs.

3.2 Mechanical properties

Hardness results of Ti64 and Ti64 composites are shown in a histogram (**fig. 6**), where testing area was selected in the matrix because test data on $Ti_6C_{3.75}$ compounds or VGCFs agglomeration area was unstable and irregular. Hardness of the monolithic Ti64 after SPS is 454.8 Hv. It significantly increased to 487.1 Hv when 0.1wt% of VGCFs was added. Increment in hardness after adding VGCFs possibly caused by two factors; changes in microstructures from full lamellar to bimodal and solid solution of carbon element into Ti matrix. In order to investigate the effect of changes in microstructures on hardness, the monolithic Ti64 was fabricated by SPS at 1173 K (below the β transition temperature), and bimodal structures were obtained. Hardness of the monolithic Ti64 which is full lamellar or bimodal structures was similar, and this indicated that changes in microstructures had no effect on hardness. The only important effect on improvement in hardness was carbon solid solution [12, 13]. Increasing of VGCFs content from 0.1 wt% to higher content showed no improvement in hardness. This corresponded well with the previous results that a maximum solubility limit of carbon in Ti matrix was 0.1 wt%. Hardness data of Ti64 and Ti64 composite before and after extrusion were similar, and this implied that an effect of grain refinement on hardness wasn't so effective.

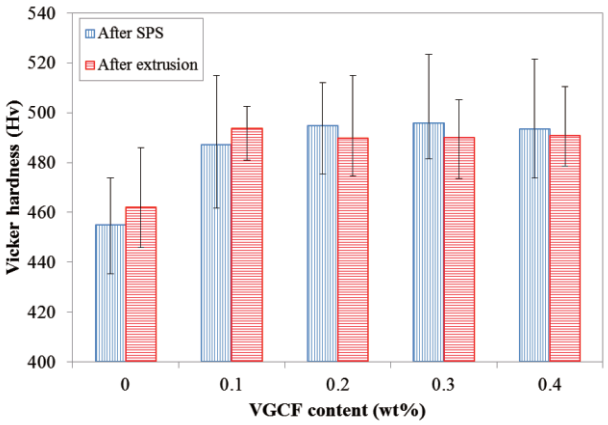

Fig. 6 Histogram of relationship between hardness and VGCFs content (wt%) of SPSed and extruded Ti64 powder materials.

Figure 7 shows plots of the relationship between tensile properties (0.2%YS, UTS and elongation) and VGCFs content of extruded samples. Their values are also summarized in **table 3**. The 0.2%YS and UTS were significantly increased by 155.6 and 73.2 MPa when 0.1 wt% of VGCFs was added. The main strengthening mechanism at this stage was a solid solution strengthening of carbon element in the Ti matrix. The effect of changes in microstructures wasn't so significant in strengthening which was reported in some research papers [14, 15]. Another strengthening mechanism should be obtained from VGCFs reinforcements. However, the microstructure shows that almost all of VGCFs were reacted with Ti and some remain in agglomeration area which made this strengthening mechanism not prominent. Increasing VGCFs content more than 0.1 wt% resulted in increment of $Ti_6C_{3.75}$ compounds quantity (fig. 5), but didn't affect much on strengthening of material.

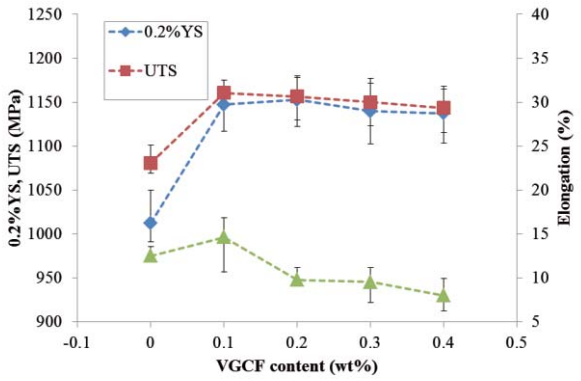

Fig. 7 Plot of relationship between tensile properties (0.2%YS, UTS and elongation) and VGCFs content (wt%) of extrusion samples.

Table 3 0.2%YS, UTS, elongation and hardness after SPS and hot extrusion of Ti64 and Ti64+VGCF composites.

VGCF content (wt%)	0.2%YS (MPa)	UTS (MPa)	Elongation (%)	Hardness (HV)	
				After SPS	After Extrusion
No VGCF	991.6	1087.5	14.1	456.3	471.1
0.1	1140.7	1160.7	15.8	459.1	484.8
0.2	1152.6	1159.9	10.9	476.0	492.1
0.3	1139.5	1150.0	10.6	485.1	498.6
0.4	1137.3	1144.5	8.8	488.5	499.5

Figure 8 shows fracture surfaces of Ti64 and Ti64 composites after tensile testing. It can be seen that fracture morphology of monolithic Ti64 was lamellar as its microstructure has large dimple size and low fracture boundary area (**fig. 8a**). On the other hand, Ti64 composites show equiaxed fracture morphology with small dimple size related to it microstructure (**fig. 8b-e**). Fracture of $Ti_6C_{3.75}$ compounds on fracture surfaces can be observed in sample, these compounds were acting as load carriers during tensile testing and cracked as a brittle phase. However, larger VGCFs content also increased VGCFs agglomeration area which was harmful to material strength as materials defects (**fig. 8f**). This area would be a pore causing no improvement of 0.2%YS and UTS when VGCFs content was over 0.1 wt%.

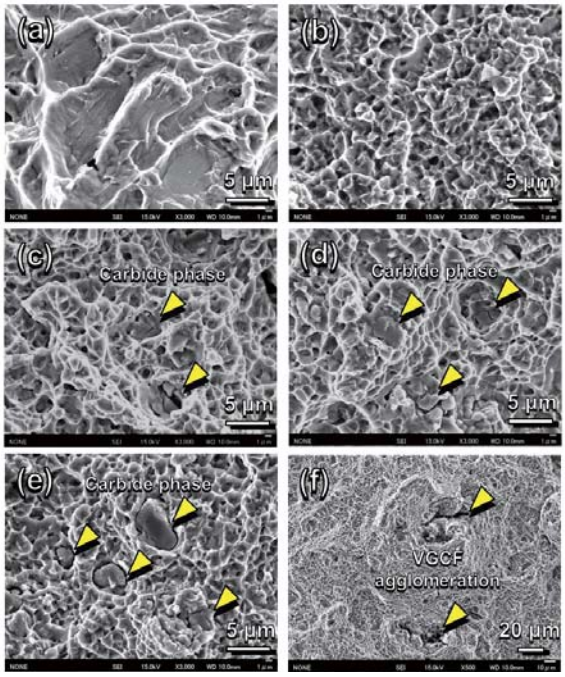

Fig. 8 Fractured surfaces of tensile specimens in transversal direction to tensile testing direction: (a) Ti64 (b) Ti64+0.1wt%VGCFs (c) Ti64+0.2wt%VGCFs (d) Ti64+0.3wt%VGCFs (e) Ti64+0.4wt%VGCFs and (f) VGCFs agglomeration zone.

Ductility of sample was increased from 12.5 to 14.6% when added 0.1 wt% of VGCFs, this can be explained by changing in morphology of microstructure from full lamellar to equiaxed that increased fracture boundary area [13]. Ductility of Ti64+0.2wt%VGCFs was significantly decreased compared to Ti64+0.1wt%VGCFs from 14.6 to 9.8 wt% caused by first precipitate of brittle $Ti_6C_{3.75}$ compounds and VGCFs agglomeration in the sample. Ductility was gradually decreased when increased VGCFs content higher than 0.2 wt% because more brittle $Ti_6C_{3.75}$ compounds and VGCFs agglomeration area were formed.

4. Conclusions

The improvement of hardness, 0.2%YS, UTS and also ductility of Ti64+VGCFs composites can be achieved by solid solution strengthening and α stabilization of carbon. The conclusions of this study are as follows:

(1) Changes in the microstructures from full lamellar to bimodal were affected by VGCFs addition. Amount of $Ti_6C_{3.75}$ compounds was also increased by increasing VGCFs content. Agglomeration of VGCFs and formation of $Ti_6C_{3.75}$ compounds could be initially observed in Ti64+0.2wt%VGCFs.

(2) Hardness improvement can be achieved by solid solution of carbon element in Ti matrix in which solid solubility of carbon was limited at 0.1wt%.

(3) 0.2%YS and UTS were improved by the solid solution strengthening of carbon in the Ti matrix

(4) Change in fractured morphology from full lamellar to equiaxed by adding 0.1 wt% of VGCFs improve ductility. However, increasing VGCFs contents over 0.1wt% caused formation of brittle $Ti_6C_{3.75}$ compounds and VGCFs agglomeration area which were harmful to ductility.

According to the above conclusion, Ti64+0.1wt%VGCFs exhibit interesting mechanical properties such as the highest UTS and ductility of 1160.7 MPa and 14.6%, respectively, and shows high hardness of 493.5 Hv after hot extrusion. Tensile strength and ductility of this material can be increased simultaneously by addition a small amount of VGCFs.

5. Acknowledgement

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