Materials Physics and Mechanics. 2023;51(3): 29-37
Research article
http://dx.doi.org/10.18149/MPM.5132023_4

Submitted: December 26, 2022
Approved: February 21, 2023
Accepted: March 28, 2023

Microstructural characterization of SiC reinforced Ti−6Al−4V metal matrix composites fabricated through powder metallurgy route

Ashish Srivastava ✉**, Kadir Aajij Shaikh, Satish Bopanna**

School of Engineering, Presidency University, Bangalore-560064, Karnataka, India

✉ ashish.rs.mec14@iitbhu.ac.in

Abstract. Titanium alloy specifically Ti−6Al−4V is largely used in aerospace industry owing to its high specific strength and stiffness. Further improvement in the specific strength and stiffness of the alloy can be achieved by reinforcing it with hard ceramic material like SiC. In the present investigation, different proportion of SiC particles (0, 1.5, 3 and 4.5 %) was introduced into the Ti−6Al−4V alloy to fabricate the metal matrix composite. The MMC's were prepared through powder metallurgy route which involves mechanical alloying of different powder in the predefined proportion followed by compaction and sintering in the furnace. The microstructure of fabricated composite was analyzed using scanning electron microscope. Uniform distribution of SiC particles in the titanium matrix is observed due to better wettability between the reinforcement and the matrix. The bulk hardness of the MMC's was measured on Rockwell Hardness (C scale). The x-ray diffraction analysis and EDX spectroscopy is also performed to capture the phase transformation after the sintering. The result shows that with the increase in the mass fraction of the SiC in the MMC's, a continuous increase in the hardness is observed. A 13.15 % increase in hardness is observed with 1.5 % addition of SiC in base Ti alloy. However, this percentage increase is increased to 26 % with addition of 4.5 % of SiC in the matrix. The increase in the hardness is due to higher hardness of reinforced SiC. SEM micrograph shows the uniform distribution of reinforced particle into the matrix.
Keywords: Powder metallurgy, Ti-6Al-4V, Composite, SiC, Scanning electron microscope

Citation: Ashish Srivastava, Kadir Aajij Shaikh, Satish Bopanna. Microstructural Characterization of SiC reinforced Ti−6Al−4V metal matrix composites fabricated through powder metallurgy route. *Materials Physics and Mechanics*. 2023;51(3): 29-37. DOI: 10.18149/MPM.5132023_4.

Introduction

Titanium alloy (Ti−6Al−4V) is widely used in the manufacturing of aerospace component and other structural member due to its light weight, high specific strength, and stiffness [1]. However, properties such as young's modulus, wear resistance and thermal resistance of these alloys are inferior as compared to those of Nickel and steel-based alloy. The abrasion test performed on Ti–6Al–4V alloy andtool steel shows that the wear rate of titanium alloy is nearly 15 times higher than that of tool steel [2]. This inferior tribological response of titanium alloy is on account of lower hardness and lower values of tensile and shear strength [3]. The poor wear resistance associated with titanium alloys can be significantly improved by reinforcing hard ceramic particle (secondary particle) into the titanium matrix [4,5]. Particle reinforced TMC's are more economical as compared to that of continuous fibre reinforced TMC and their properties are nearly isotropic [6]. As of now, different types of secondaryparticles are used by various authors as a reinforcement in the fabrication of TMC's. The most commonly used

Publisher: Peter the Great St. Petersburg Polytechnic University

reinforcement is TiC [7], TiN [8], SiC [9], TiB [10], B [11] and CNT [12]. Titanium matrix composite (hereafter referred as TMC's) due to its high specific modulus and high specific strength has found potential application in the aircraft industry [13]. TMC's in the recent past are developed using melting route and powder metallurgy route. The fabrication of near net shape product using melting approach is difficult and is due to poor fluidity of the melts [14]. However, powder metallurgy route is most cost effective and can be used to produce near net shape components [15]. Ravindran et al. [16] fabricated hybrid composites, Al2024/(SiC + Gr), using powder metallurgy and have found significant enhancement in the wear properties of reinforced composite as compared to the base alloy. Kobayashi et al. [17] analyzed the performance of TMC's fabricated via mechanical alloying with different secondary particles such as TiB, MoB and CrB and observed that TMC fabricated with TiB as reinforcement lead to better mechanical and tribological performance. Yong-Jin Kim et al. [7] fabricated the in situ TMC with TiC as reinforcement and observed that hardness, yield strength, and tensile strength of the fabricated TMC significantly improved in comparison to parent titanium alloy. They also observed the continuous increase in the hardness of the fabricated TMC with increase in the amount of the TiC particle in the matrix. Poletti and Holtl [18] compared the properties of TMC's reinforced with TiC and SiC. They observed that the strengthening of SiC reinforced TMC is due to matrix strain hardening which is generated due to thermal mismatching of reinforcement and the matrix. On the other hand, strengthening of TiC reinforced TMC is due to the dissolution of C in the alpha phase. Finite element modelling of discontinuously reinforced TMC shows that strength hardening of TiB whisker reinforced TMC is mainly due to the matrix strengthening and not because of load transfer mechanism [6]. Sabri et al. [19] confirmed the improvement in mechanical properties with increasing reinforcement content, while Siva Surya et al. [20] successfully fabricated SiC reinforced Al7075 composite through P/M route.

From the above literature survey, it was observed that most of the researchers used melt-based approach to fabricate the composite material. This melt-based approach is not suitable to produce near net shaped product due to poor fluidity of the melt. The aim of the present experimental study is to fabricate the composite material by reinforcing hard SiC particle in the base Ti−6Al−4V alloy through powder metallurgy route. To analyze the effect of SiC on the mechanical properties of the fabricated composite, three different weight percentages (1.5, 3.0 and 4.5 %) of SiC are reinforced to the base Ti−6Al−4V alloy.

The metallurgical properties of the fabricated TMC's were evaluated using scanning electron microscopy and their relation with mechanical properties such as hardness is discussed in detail. The phase transformation and compositional change occurred during the sintering process is captured through x-ray diffraction and EDX technique respectively.

Materials and Methods

Raw material. The fabrication of TMC's is accomplished using Ti−6Al−4V alloy as the matrix and SiC particle of 100 mesh size as reinforcement material. Titanium alloy is first fabricated by mechanical alloying of titanium powder (90 % by mass), aluminum powder (6 % by mass) and Vanadium (4 % by mass). In the fabrication of TMC's, SiC particles of 97.43 % purity, titanium powder of 98.99 % purity, aluminum powder of 99.72 % purity and vanadium powder of 100 % purity are used. The purity of the powder in terms of elemental composition is confirmed through Energy dispersive spectroscopy (EDS) spectra before further processing.

Fabrication of TMC's. The specimen of TMC's is prepared by reinforcing 1.5, 3.0 and 4.5 % SiC (by mass) through powder metallurgy technique. Unreinforced alloy was also prepared in the same way. In this technique, the pre-calculated amount of different powders was milled in a high energy ball mill to get uniform mixture. The powder was milled using stainless steel ball of 0.5 cm diameter and is shown in Fig. 1(a). One clockwise and one anticlockwise rotation each of one hour is used to complete the milling process. Thereafter, the mixture was cold compacted into cylindrical shape with the help of die and punch system shown in Fig. 1(b). A load of 90kN is applied for cold compaction of powder using hydraulic press as shown in Fig. 1(c). The final compacted sample is shown in Fig. 1(d). After that the compact was immediately sintered at 1100 °C using high temperature furnace. The chosen temperature is due to the fact that at this temperature both phases are properly bonded and that the composite is fully densified. Also, at this temperature there is no formation of unwanted phases or defects that can negatively impact the mechanical properties of the composite. After that the compact was immediately sintered at 1100 °C using high temperature furnace. A time period of 2.5 h. is maintained as choking time followed by cooling in the furnace till the sample reaches to room temperature. The sample were then cut from fabricated composite for further characterization.

(a) (b) (c) (d)

Fig. 1. The steps followed in fabrication of TMC's (a) ball milling of powder (b) die and punch used (c) compaction of powder (d) P/M sample

Microstructural Characterization. For microstructural characterization, titanium alloy and fabricated TMC's specimen are polished using fine emery paper. Thereafter, specimen was cloth polished on auto-disc polishing machine with diamond paste to achieve shiny surface and then were cleaned with acetone to remove any unwantedparticle from the surface. Finally, the sample were observed under scanning electron microscope.

Hardness. The bulk hardness of the TMC's and alloy is measured through Rockwell hardness tester. A steel ball of diameter 1/8 inch is used as an indenter. A load of 60 kgf is used for a time of 10 sec (dwell time) to perform the test. For better accuracy, five indentations were taken at different locations and the average value of them is taken for further analysis.

X-ray diffraction study. The phase identification of the TMC's is done through X-ray diffraction technique. The monochromatized Cu-Kα radiation with Ni filter is used to obtain the diffraction pattern.

Energy dispersive spectra analysis. Energy dispersive spectroscopy technique is used to identify the elemental composition and proportion present in the titanium alloy and fabricated TMC's specimen. The beam energy in the range 0-20 keV is used to emit the characteristics X-rays from the sample. The acceleration voltage is set at 15 kV for the purpose.

Results and Discussions

Microstructure. The SEM micrograph of monolithic titanium alloy and fabricated TMC's with 1.5, 3.0 and 4.5 % SiC is shown in Fig. 2. Figure 2(c) shows the clean interface between the SiC particle and the titanium matrix without the presence of any void. The uniform distribution of SiC particles in the titanium matrix can be observed from Fig. 2(b). This homogeneous distribution is on account of better wettability between the reinforcement and the matrix. The increase in amount of SiC in the matrix will lead to more interfaces between the reinforcement and the matrix. Moreover, larger amount of reinforcement increases the porosity which in turn reduces the strength of the sample [1]. Also, larger mass fraction of reinforcement favors the solid-state reaction leading to the formation of intermetallic compound such as Ti_5Si_3 and TiC. These intermetallic being brittle in nature can cause initiation of crack and under the presence of tensile stress; these cracks can grow and lead to catastrophic failure of the component.

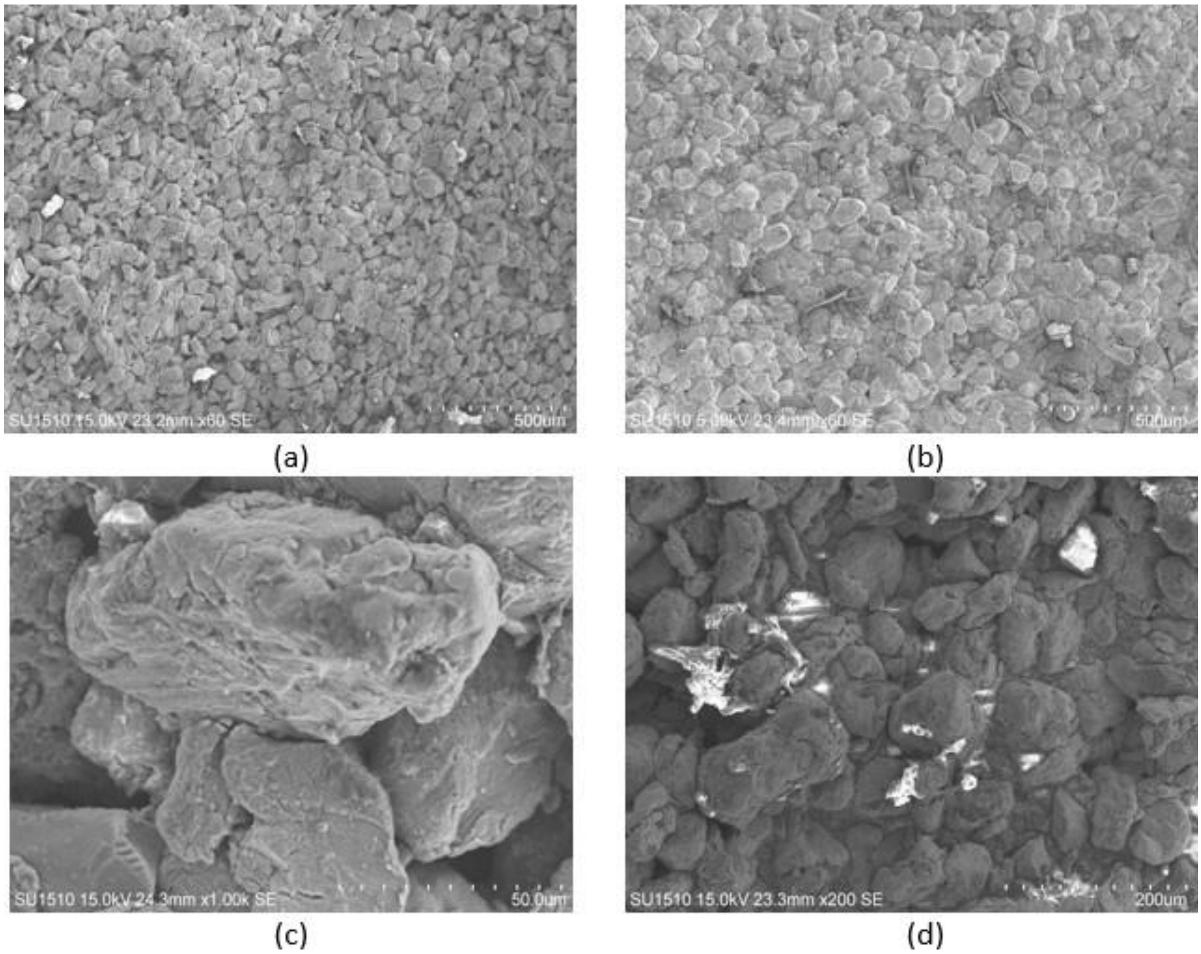


Fig. 2. SEM images of (a) Monolithic Ti alloy, (b) TMC with 1.5 % SiC, (c) TMC with 3.0 % SiC, and (d) TMC with 4.5 % SiC

Hardness. The hardness of the Ti alloy and the fabricated TMC's was measured in terms of Rockwell hardness (HRC) and is shown in Fig. 3. It can be seen from the figure that hardness of Ti alloy is 39 HRC and with the addition of SiC, the hardness of the TMC is found to increase with maximum hardness being achieved at 49 HRC with 4.5 % SiC. The continuous increase in the hardness with the increase in amount of reinforcement in the TMC is due to fact that larger amount of hard ceramic particle offers greater resistance to the plastic deformation. Also, harder reinforcement particle act as strong pinning sites which restrict the dislocation motion and thus TMC with higher amount of SiC shows higher hardness value. Moreover, the clean interface with minimum porosity between the reinforcement and the matrix leads to better bonding strength and thus helps in improving the hardness of the TMC's.

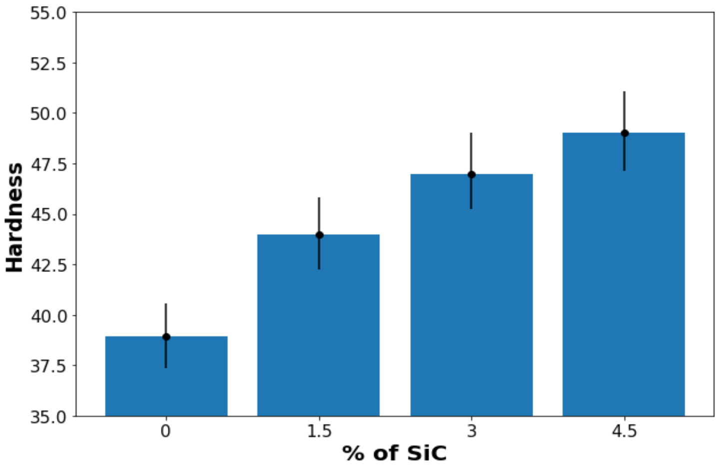


Fig. 3. Variation in hardness of fabricated TMC's with amount of SiC reinforcement

Energy dispersive spectral analysis. The elemental composition of as supplied powder needs to be verified as it significantly affects the final properties of the fabricated TMC's. Thus, the EDS analysis of the as supplied powder (Ti, Al, V and SiC) is shown in Fig. 4.

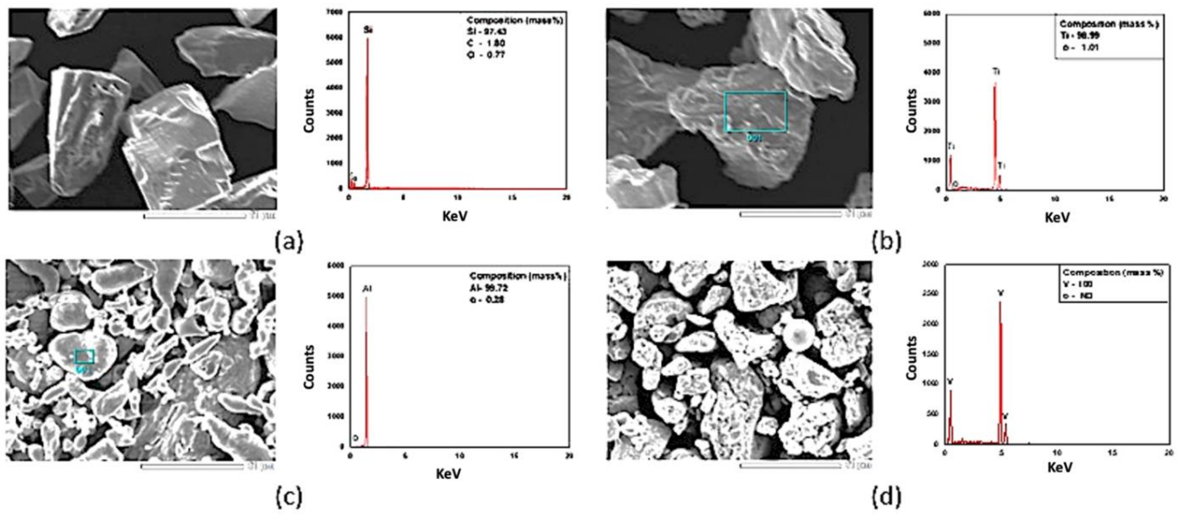
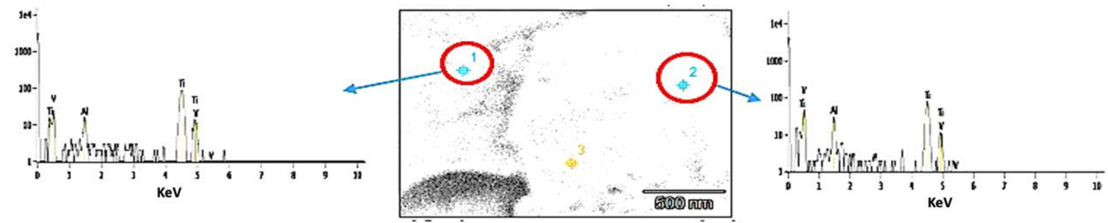


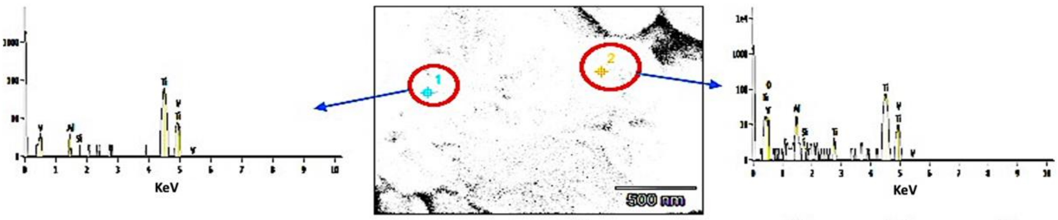
Fig. 4. EDS results of as supplied powder (a) SiC, (b) Titanium, (c) Aluminum, and (d) Vanadium

It can be observed from the figure that sample of Ti powder consists of 98.99 % of Ti and 1.01 % of O. The elemental composition (mass %) of Al powder consists of 99.72 % of Al and 0.28 % of O; V powderconsists of 100 % of V and SiC consists of 97.43 % of Si, 1.80 % of C and 0.77 % O respectively. The EDSanalysis of TMC's with different mass % of SiC is shown in Fig. 5. The EDS details were examined at two different locations; point 1 and point 2. The presence of a very small amount of oxygen is observed in the TMC's. However, the significant increase in the carbon element is observed in Fig. 5(d) as compared to other fabricated TMC's and this is due to greater amount of SiC reinforcement.



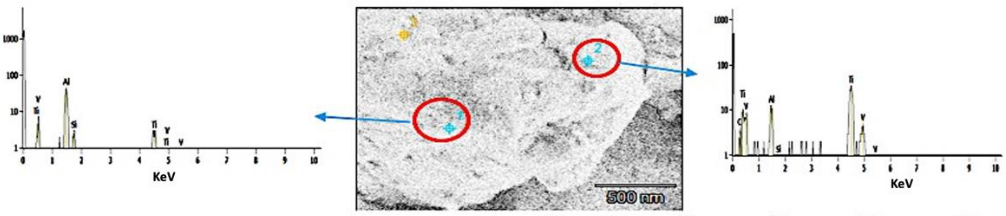
	O	Al	Ti	V
Point 1	-	3.53	94.94	1.53
Point 2	-	7.92	89.43	2.65
Average	-	5.72	92.18	2.09

(a)



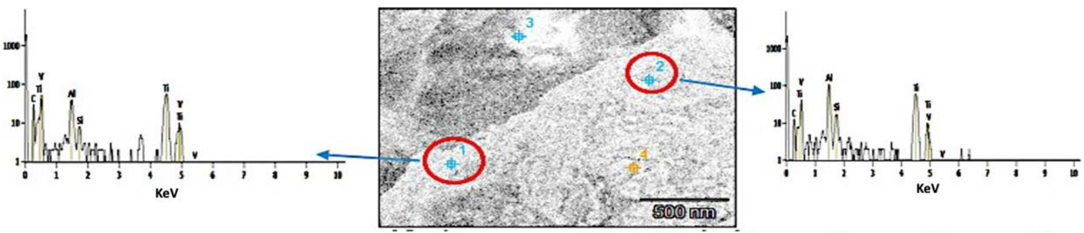
	O	Al	Si	Ti	V
Point 1	-	0.45	0.51	94.66	4.38
Point 2	2.10	3.78	0.44	92.40	1.28
Average	1.05	2.11	0.47	93.53	2.83

(b)



	C	O	Al	Si	Ti	V
Point 1	-	-	63.80	6.29	21.00	8.91
Point 2	2.23	-	6.41	0.09	88.50	2.77
Average	1.11	-	35.10	3.19	54.75	5.84

(c)



	C	O	Al	Si	Ti	V
Point 1	6.67	-	11.03	1.90	78.07	2.34
Point 2	4.65	-	23.25	4.36	67.06	0.69
Average	5.66	-	17.14	3.13	72.56	1.51

(d)

Fig. 5. EDS spectrum of TMC's with (a) 0 % SiC(unreinforced alloy), (b) 1.5% SiC, (c) 3 % SiC, (d) 4.5 %SiC

X-ray diffraction. Identification of different phases formed due to solid state reactions at higher temperature (as in case of powder metallurgy) between the reinforcement (SiC) and matrix (Ti−6Al−4V) is important as it may lead to the formation of brittle intermetallic compound which can deteriorate the in-service performance of the fabricated composite [21]. The diffraction peaks for Ti_6Al_4V and SiC will appear at different angles due to their different crystal structures. By analyzing the position and intensity of these peaks, it is possible to identify the phases present and determine their crystal structure. The X-ray diffraction pattern of titanium alloy without SiC and fabricated TMC's with 4.5 % SiC is shown in Fig. 6. From the figure, it can be seen that the Ti alloy (without SiC) consists of a major phase. The shown value in the diffraction pattern well match with the data reported [22]. In TMC with 4.5 % SiC, the intensity of α-Ti peak considerably reduced and two more new peaks of TiC and SiC appear in the diffraction pattern. The formation of TiC is due to diffusion mechanism along the grain boundary. In the present investigation, no peaks corresponding to Ti_5Si_3 is observed. Also, there is no clear phase separation because of reaction between SiC/Ti as expected rather the reaction zone typically consists of TiC and is due to nearly equal diffusion coefficient of carbon and silicon in titanium. Among C, Si and Ti, none of the element are mobile elemental species and therefore the growth of the reaction [23] is governed by diffusion of all elements together.

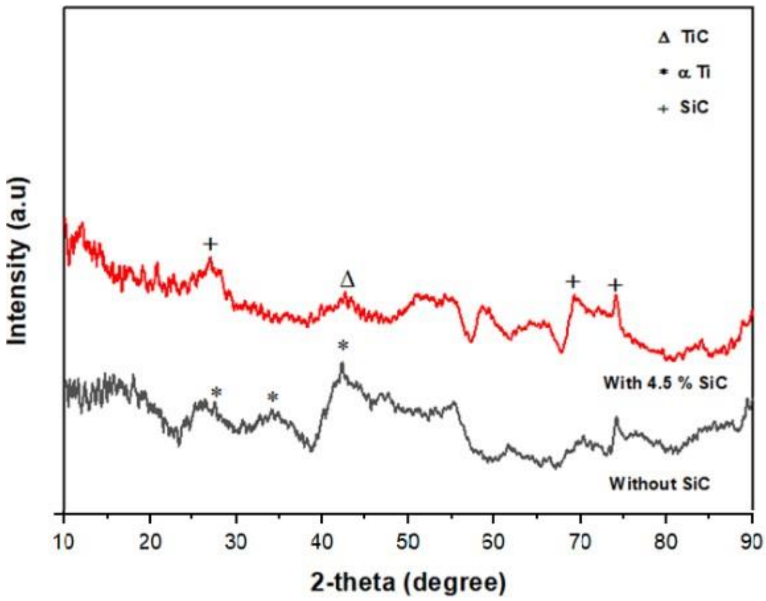


Fig. 6. X-ray diffraction profile of TMC's with (a) 0 % SiC (unreinforced alloy) and (b) 4.5 % SiC

Conclusions. In the present investigation, the ceramic particle (SiC) was reinforced in industry standard Ti-6Al-V alloy to fabricate Titanium matrix composites through powder metallurgy technique. The effect of different amount (mass %) of reinforcement on microstructure and hardness were studied in detail. The results show that SiC reinforced TMC's fabricated through P/M technique offer better hardness values in respect to the parent Ti alloy. The hardness of 4.5 % SiC reinforced TMC is found to be 49 HRC which is 25.64 % higher than the unreinforced Ti alloy. The phase analysis carried out through XRD shows the formation of harder TiC phase during the sintering process. Also, it is recommended from the current study that the P/M technique can be effectively used to fabricate Ti based composites; The P/M technique is cost effective technique for the fabrication of TMC's.

References

1. Poletti C, Balog M, Schubert T, Liedtke V, Edtmaier C. Production of titanium matrix composites reinforced with SiC particles. *Composite Science and Technology*. 2008;68(9): 2171-2177.
2. Budinski KG. Tribological properties of Titanium alloys. *Wear*. 1991;151: 203-217.
3. Kutas FM, Misra MS. Friction and wear of Titanium alloys. *ASM Handbook*. 1992;18: 778-784.
4. Alman DE, Hawk JA, Simmons JS. Abrasive wear behavior or P/M titanium metal-matrix composites. *Advances in Powder Metallurgy and Particulate Materials*. 1997;7: 3-18.
5. Oh JC, Yun E, Golkovski MG, Lee S. Improvement of hardness and wear resistance in SiC/Ti-6Al-4V surface composites fabricated by high-energy electron beam irradiation. *Material Science and Engineering A*. 2003;351(1-2): 98-108.
6. Wang P, Nian G, Qu S, Shan Y, Huang L, Peng HX. Numerical study on Mechanical properties of discontinuously reinforced titanium matrix composite with network reinforcement architecture. *International Journal of Applied Mechanics*. 2017;9(5): 1750073.
7. Kim YJ, Chung H, Kang SJ. Processing and mechanical properties of Ti-6Al-4V/TiC in situ composite fabricated by gas-solid reaction. *Material Science and Engineering A*. 2002;333(1-2): 343-350.
8. Cui ZD, Zhu SL, Man HC, Yang XJ. Microstructure and wear performance of gradient Ti/TiN metal matrix composite coating synthesized using gas nitriding technology. *Surface and Coatings Technology*. 2005;190(2-3): 309-313
9. Selamat MS, Watson LM, Baker TN. XRD and XPS studies on surface MMC layer of SiC reinforced Ti-6Al-4V alloy. *Journal of Materials Processing Technology*. 2003;142(3): 725-737.
10. Godfrey TMT, Goodwin PS, Ward-Close CM. Production of titanium particulate metal matrix composite by mechanical milling. *Material Science and Technology*. 2000;16(7-8): 753-758.
11. Lu H, Zhang D, Gabbitas B, Yang F and Matthews S. Synthesis of a TiBw/Ti6Al4V compositeby powder compact extrusion using a blended powder mixture. *Journal of Alloys and Compounds*. 2014;606: 262-268.
12. Kondoh K, Threrujirapapong T, lmai H, Umeda J, Fugetsu B. Characteristics of powder metallurgy pure titanium matrix composite reinforced with multi-wall carbon nanotubes. *Composites Science and Technology*. 2009;69(7-8): 1077-1081.
13. Liu Y, Chen LF, Tang HP, Liu CT, Liu B, Huang BY. Design of powder metallurgy titanium alloys and composites. *Material Science and Engineering A*. 2006;418(1-2): 25-35.
14. Kim IY, Choi BJ, Kim YJ, Lee YZ. Friction and wear behavior of titanium matrix (TiB+ TiC) Composites. *Wear*. 2011;271(9-10): 1962-1965.
15. Alman DE, Hawk JA. The abrasive wear of sintered titanium matrix- ceramic particle reinforced composites. *Wear*. 1999;225-229: 629-639.
16. Ravindran P, Manisekar K, Rathika P, Narayanasamy P. Tribological properties of powder metallurgy-Processed aluminium self lubricating hybrid composites with SiC additions. *Materials & Design*. 2013;45: 561-570.
17. Kobayashi M, Funami K, Suzuki S, Ouchi C. Manufacturing process and mechanical properties of fine TiB dispersed Ti-6Al-4V alloy composites obtained by reactive sintering. *Material Science and Engineering A*. 1998;243(1-2): 279-284.
18. Poletti C, Holtl G. Mechanical properties of particle reinforced titanium and titanium alloys. *Kovove Materials*. 2010;48(2): 87-95.
19. Sabry, I, Ghafaar MA, Mourad, AHI, Idrisi AH. Stir casted SiC-Gr/Al6061 hybrid composite tribological and mechanical properties. *SN Appl. Sci.* 2020;2: 943.

20. Siva Surya M, Prasanthi G. Physical and mechanical characterisation of Al7075/SiC functionally graded materials fabricated by powder metallurgy route. *Advances in Materials and Processing Technologies*. 2020;8(1): 1007-1022.
21. Gotman E, Gutmanas Y, Mogilevsky P. Interaction between SiC and Ti powder. *Journal of Material Research*. 1993;8(10): 2725-2733.
22. Sivakumar G, Ananthi V, Ramanathan S. Production and mechanical properties of nano SiC particle reinforced Ti-6Al-4V matrix composite. *Transaction of Non-ferrous metal society of China*. 2017;27(1): 82-90.
23. Tripathi H, Bharti A, Saxena KK, Kumar N. Improvement in mechanical properties of structural AZ91 magnesium alloy processed by friction stir processing. *Advances in Materials and Processing Technologies*. 2020;8(3). 1543-1556.

THE AUTHORS

Srivastava Ashish
e-mail: ashish.rs.mec14@iitbhu.ac.in

Aajij Shaikh Kadir
e-mail: kadirshaikh27@gmail.com

Satish Babu Boppana
e-mail: satishbabu3@yahoo.co.in