Production and mechanical properties of nano SiC particle reinforced Ti–6Al–4V matrix composite

G. SIVAKUMAR¹, V. ANANTHI², S. RAMANATHAN³

1. CISL, Department of Physics, Annamalai University, Tamilnadu 608002, India;
2. Physics Section, FEAT, Annamalai University, Tamilnadu 608002, India;
3. Manufacturing Engineering Department, Annamalai University, Tamilnadu 608002, India

Received 22 November 2015; accepted 31 May 2016

Abstract: Different mass fractions (0, 5%, 10%, and 15%) of the synthesized nano SiC particles reinforced Ti–6Al–4V (Ti64) alloy metal matrix composites (MMCs) were successfully fabricated by the powder metallurgy method. The effects of addition of SiC particle on the mechanical properties of the composites such as hardness and compressive strength were investigated. The optimum density (93.33%) was obtained at the compaction pressure of 6.035 MPa. Scanning electron microscopic (SEM) observations of the microstructures revealed that the wettability and the bonding force were improved in Ti64 alloy/5% nano SiCₚ composites. The effect of nano SiCₚ content in Ti64 alloy/SiCₚ matrix composite on phase formation was investigated by X-ray diffraction. The correlation between mechanical parameter and phase formation was analyzed. The new phase of brittle interfaced react synthesized nano SiC particle reinforced Ti64 alloy/SiCₚ matrix composite on phase formation was investigated by X-ray diffraction. The new phase of brittle interface reaction formed in the 10% and 15% SiCₚ composite specimens and resulted in no beneficial effect on the strength and hardness. The compressive strength and hardness of Ti64 alloy/5% nano SiCₚ MMCs showed higher values. Hence, 5% SiCₚ can be considered to be the optimal replacement content for the composite.

Key words: titanium alloy; nano SiC particle; powder metallurgy; hardness; compressive strength

1 Introduction

Metal matrix composites (MMCs) represent a new generation of engineering materials in which a strong ceramic reinforcement is incorporated into a metal matrix to improve its properties including specific strength, specific stiffness, wear resistance, excellent corrosion resistance and high elastic modulus [1,2]. MMCs combine metallic properties of matrix alloys (ductility and toughness) with ceramic properties of reinforcements (high strength and high modulus), leading to greater strength in shear and compression, and higher service-temperature capabilities, and control of physical properties such as density, coefficient of thermal expansion [3,4]. Thus, they have significant scientific, technological and commercial importance. During the last decade, because of their improved properties, MMCs have been used extensively for high performance applications such as in aircraft engines and more recently in the automotive industry [5,6]. Continuous fiber-reinforced titanium matrix composites (TMCs) are expensive due to high fiber costs and limited formality, and their properties are highly anisotropic. Thus, these materials have been restricted to very specialized applications. Discontinuous or particulate-reinforced TMCs are less expensive to fabricate and their properties are nearly isotropic. Thus, particulate-reinforced TMCs have been widely studied for structural applications [7,8]. Many studies have attempted to expend the range of applications and manufacturing techniques of these materials. TMCs, which have good wear resistance, high strength, and high strength stability through added reinforcement, exhibit advanced mechanical and chemical properties [9].

The poor tribological and thermo mechanical behaviors of titanium alloy were attributed to low hardness, relatively low tensile and compressive strengths. The addition of ceramic particle with TMCs matrix has been found to be an effective method for improving the tensile strength, shear strength, and hardness, and enhances wear resistance [10,11]. The particles investigated in previous works as reinforcements of titanium alloys are ceramics of TiC,
2.4 Density measurement and porosity

The experimental density (bulk density) of the composites was obtained by Archimedian method. The
theoretical density was calculated using the rule of mixture. Bulk density was calculated by using Eq. (1) [26]:

$$\rho_B = \frac{m_d}{m_{sat} - m_{sus}} \times \rho_{H2O}$$

(1)

where $\rho_B$ is the bulk density, $m_{sat}$ is the saturated mass, $m_d$ is the dry mass, and $m_{sus}$ is the suspended immersion mass.

Porosity ($P$) was calculated by using Eq. (2) [27]:

$$P = \frac{m_{sat} - m_d}{m_{sat} - m_{sus}} \times 100\%$$

(2)

2.5 Hardness and compressive strength tests

The microhardness of the polished face of composite specimens was found at room temperature using an HMV–2T Schimadzu. The hardnesses of Ti64 alloy and composites were measured after polishing to a 1 µm finish. To evaluate the Vicker microhardness, several indentations were made on the faces of the composite at various loads and a dwell time of 10 s. The hardness values of Ti64 alloy and Ti64 alloy/nano SiCp composite specimens were taken at different places on the surface. The average of these values was considered in this result.

Compressive tests were used to assess the mechanical behavior of the composites and matrix alloy. The surface of the samples was polished with 600-grit sand paper. The compressive strength was tested and recorded by using a universal testing machine (UTM).

2.6 Microstructures and particle size

Ti matrix alloy and composite billets were polished with 600-grit sand papers and subsequently polished on cloth using diamond paste of 6 and 1 µm. Microscopic examinations of the composites and matrix alloy were carried out using a JSM–5610LV scanning electron microscope (SEM).

The phase identifications were performed by X-ray diffraction (XRD model: X’Pert PRO PAN) analytical technique using Cu Kα radiation. AFM images were recorded for the nano SiC particles by using AGILENT–N9410A series 5500 AFM.

3 Result and discussion

3.1 Particle size

The atomic force microscope image (3D) of the ball-milled SiC nano particles is shown in Fig. 1(a) and 2D representation of height image and line profile of the synthesized SiC nano particles is shown in Fig. 1(b). The average particle size of SiCp observed is 75 nm. The surface of the SiCp is found as irregular and rough shape.

![AFM images of ball-milled SiC nano particles](image)

Fig. 1 AFM images of ball-milled SiC nano particles: (a) 3D; (b) 2D

The increased surface roughness supports the higher surface energy of nano SiCp. The results reveal that the size reduction of SiCp from micron to nano level was achieved by high energy planetary ball milling (Retsch, PM 100) [28].

3.2 Density and porosity

The graphs of theoretical, bulk density and porosity of the composites according to the mass fractions of nano SiCp are shown in Fig. 2. It can be seen that the theoretical density of 5% nano SiCp composites increases and that for 10% and 15% SiCp composites (as expected from the rule of mixtures). The same trend was also observed in bulk density (Archimedes principle). The experimental densities are observed to be lower than the theoretical densities. The density measurements showed that the composites contained some porosities. The amount of porosity in the composites increased with increasing mass fraction of the SiC particles. Similar results have observed in previous investigations [29–31]. 5% nano SiCp composite has a very low porosity among the other three composites with different contents of SiCp. The result suggests that 5% of nano SiCp is effective, due to the removal of pores and it enhances the mechanical properties of the composites. More pores are filled by nano SiCp and therefore, the number of open pore decreases, at 5% nano SiCp composite.
The effects of low wettability and high agglomeration at high reinforcement content and pore nucleation at the matrix interface are the reasons for higher porosity for 10% SiCₚ and 15% SiCₚ. When the nano SiCₚ content increases more than 5%, the excess of unreacted SiC particles create pores, whose volume expands due to the increased pressure of the gas inside the closed pores. The reduction of porosity in the SiCₚ-reinforced alloy composite can be written in the following sequences, i.e., Ti alloy/5% nano SiCₚ>Ti alloy>Ti alloy/10% nano SiCₚ>Ti alloy/15% nano SiCₚ.

### 3.3 X-ray diffraction analysis

The XRD patterns of Ti64 alloy and Ti64 alloy/nano SiCₚ composites are shown in Fig. 3. ANDERSSON and WARREN [32] reported that, in reactive systems, SiC reacts with the metal to form silicides and carbides. Such reactions are thermodynamically possible between SiC and Ti. Ti reacts with SiC rapidly to produce TiC and Ti₅Si₃. The intensity of α-Ti phase (peak) significantly decreased in 10% and 15% SiCₚ specimens and subsequently some new peaks (Ti₅Si₃ and TiC) with high intensity grew up at 2θ=36.92° (210), 37.63° (102), 42.14° (300) which belong to reflections due to Ti₅Si₃ phase (JCPDS card No. 08–0041, 78–1429). TiC phase is observed at 2θ=62.19° (220) in 10% SiCₚ specimen. The high intensity of TiC is formed at 2θ=41.80° (104) formed in 5% SiCₚ specimen due to the hexagonal structure of silicon carbide (JCPDS card No. 29–1128).

The Ti64 alloy consists of an α-Ti phase as a major phase and it is observed for all the patterns nearly at 2θ=35°, 40.16°, and β-Ti phase as a minor phase observed at 2θ=38.45° which corresponds to reflection (100), (101) of HCP structure and (110) of BCC structure with d spacing values of 2.249 and 2.350 Å, respectively. The observed values well coincide with the standard JCPDS cards No. 89–2762 and 89–3726.

The α-Ti and β-Ti phases observed at 2θ=40.19° (101) and 2θ=38.37° (110) are coexistent with a new peak at 2θ=41.80° (104) formed in 5% SiCₚ specimen.

### 3.4 Microstructures

SEM images of the 0, 5%, 10% and 15% SiC particle-reinforced Ti64 alloys are shown in Fig. 4. The microstructure of Ti64 alloy is shown in Fig. 4(a). It can be seen that Ti64 alloy has distinctive Widmanstatten structure. It consists of two phases: the phase began to appear in the form of the plates as the specimen is slowly cooled near the β transus temperature [33]. Some β phases (thin dark region) are observed between α platelets in monolithic alloy, which is in good agreement with the report [34] where approximately 95% (volume fraction) α phase and only 5% (volume fraction) β phase are observed for furnace cooled Ti64 specimen. Figure 4(b) shows good bonding between matrix and reinforcement. Addition of ceramic nano particles into the titanium matrix sustains the crystallite size reduction and better particle size distribution. The surface of Ti64 alloy with 5% nano SiCₚ was the smoothest, and most uniform layer formation was achieved. The homogeneous distribution of composition is attributed to good wettability between Ti64 alloy and reinforcement.

It can be concluded that 5% nano SiCₚ (Fig. 4(b)) particles are bonded well with the Ti64 alloy matrix. The nano particle size in the present work is about 75 nm, the Orowan strengthening mechanism works more...
significant than the micro-sized ceramic particles. It clearly showed that, there were no voids and discontinuities, when nano SiC was dispersed in the titanium matrix. The inhibitory effect is more remarkable with the increase in the nano SiC [35].

Increasing the content of nano SiC (10% to 15%) in the matrix will result in more Ti64 alloy/nano SiC interfaces, and consequently large numbers of void formation sites are observed and shown in Figs. 4(c) and (d). This indicates that the weak bonding is observed between the SiC and Ti64 alloy particle. Addition of nano SiC more than 5% can increase the porosity, resulting in the decline in strength of the corresponding specimen. This is because the pore can nucleate at SiC sites and the contact surface area increases, which would result in higher porosity level [36]. Increasing the mass fraction of nano SiC leads to the formation of TiC and Ti3Si phases. These porosities and formation of reaction zone will lower the bonding of interface between the reinforcement and matrix phase.

3.5 Energy dispersive spectroscopy (EDS) spectra

EDS spectra of Ti64 alloy and Ti64 alloy/nano SiC reinforced composite specimens are shown in Fig. 5. EDS analysis taken from sintered Ti64 alloy sample exhibited that only Al, V and Ti elements present in the mass fractions of 5.5%, 4.0% and 90.5%, respectively. Ti64 alloy/5% nano SiC composite sample exhibited Al, V, Ti, Si and C elements present in the mass fractions of 5.51%, 3.70%, 85.64%, 2.40% and 2.75%, respectively. Ti64 alloy/10% nano SiC composite sample exhibited Al, V, Ti, Si and C elements present in the mass fractions of 5.35%, 3.55%, 80.50%, 5.15% and 5.45%, respectively. Ti64 alloy/15% nano SiC composite sample exhibited aluminium, vanadium, titanium, silicon and carbon elements present in the mass fractions of 4.90%, 3.13%, 76.19%, 7.26% and 8.52%, respectively.

The peaks of Si and C indicate the presence of SiC and the peaks of Ti, Al, and V are attributed to the matrix of Ti64 alloy. The mass fractions of Si and C increased with increasing reinforcement content. EDS spectra confirm the presence of SiC in the reinforced Ti64 alloy composite specimen.

It is evidently seen from all the EDS spectra and it is inferred that there is no oxygen present in the extruded sample since the hot extrusion was carried out in the argon atmosphere.

3.6 Hardness and compressive strength

The Vickers hardness of the composites is shown in Fig. 6. Vickers hardness of Ti64 alloy specimen is HV 380 [37,38] and the hardness increased significantly (20%) for 5% nano SiC reinforced Ti64 alloy specimen to HV 460, which could be attributed to the presence of
Fig. 5 EDS analysis of experimental composites: (a) Ti64 alloy; (b) Ti64 alloy/5% nano SiC<sub>p</sub> composite; (c) Ti64 alloy/10% nano SiC<sub>p</sub> composite; (d) Ti64 alloy/15% nano SiC<sub>p</sub> composite

Fig. 6 Vickers hardness of Ti64 alloy and Ti64 alloy/nano SiC<sub>p</sub> composites

harder ceramic particulate in the matrix. The presence of such hard surface area of the specimen offers more resistance to plastic deformation which leads to the increase in the hardness of the specimen, which is directly related to the forces that exist between atoms in the solid. The decrease of hardness for 10% (HV 369) and 15% (HV 315) SiC<sub>p</sub> specimens is due to greater agglomeration of particles and higher degree of defects and micro porosity present in the specimens during processing.

The higher hardness value of 5% nano SiC<sub>p</sub> can provide its inherent properties to the Ti64 alloy matrix and can act as a hindrance to the dislocation motion, which is attributed to the improvement of hardness value, and reduction of porosity was another reason behind the improvement of hardness [39]. The matrix deforms plastically to accommodate the volume expansion due to incorporation of hard reinforcement particles. Similar behavior was observed by SINGLA et al [40]. MURTY et al [41] reported that the stronger the reinforcement particles bond to alloy matrix, the higher the Vickers hardness becomes.

Further increase of 10% and 15% SiC<sub>p</sub> with Ti64 alloy resulted in decreasing the Vickers hardness. The results also revealed that a large scattering of hardness was observed for each individual composite, which may be due to the presence of residual internal stresses resulting from the thermal expansion mismatch between Ti64 alloy and nano SiC<sub>p</sub>. The Vickers hardness is known to be very sensitive to the residual internal stresses [42].

The compressive strength of the composite was investigated by determining their failing load. The compressive strength of the composites is shown in Fig. 7. The 5% nano SiC<sub>p</sub> reinforced Ti64 alloy
composite (1483 MPa) has improved (17%) compressive strength than the Ti64 alloy (1263 MPa) [43,44]. The values of 10% and 15% nano SiC_p reinforced Ti64 alloys are found to be 1189 and 1064 MPa, respectively. The tendency of 5% nano SiC_p towards higher compressive strength with higher density and lower porosity is established. This finding for the 5% nano SiC_p composites leads to the increase of vitrification level and a reduction of internal cracks and voids formation. Also, good bonding between 5% nano SiC_p particle and matrix phase can be considered to be responsible for the increase in strength. The nano particulate improves the mechanical properties in the monolithic system because of Orrowen strength mechanism.

![Fig. 7 Compressive strength of Ti64 alloy and Ti64 alloy/nano SiC_p composites](image)

When the increase of the nano SiC_p reinforcement content is above the optimum value (5%), brittle interfacial reaction products TiC and Ti_2Si_3 are formed [11] and the debonding would be expected to occur as a result of processing condition and act as critical flaws, which lower the strength of the 10% and 15% SiC_p specimens. It is observed that, nano SiC_p grains embedded in the matrix phase which has a detrimental effect on the compressive strength mainly because of the occurrence of porosity and agglomeration [45,46]. It may be one of the reasons for a low strength in 10% and 15% nano SiC_p reinforced Ti64 alloys.

4 Conclusions

1) Nano SiC particle-reinforced Ti64 alloy composites were successfully fabricated by the powder metallurgy method. Density, porosity, hardness, compressive strength, phase formation and SEM microstructures of MMCs were investigated.

2) The optimum density (93.33%) was obtained at the compaction pressure of 6.305 MPa. SEM observation of the microstructures showed that the wettability and the bonding force were improved in Ti64 alloy/5% nano SiC_p composites.

3) The new phase formation of brittle interfacial reaction formed at the 10% and 15% SiC_p composite specimens and resulted in no beneficial effect on the strength and hardness. Hence, 5% composite specimens can be considered to be the optimal replacement content.

4) The compressive strength and hardness of Ti64 alloy/5% nano SiC_p MMCs showed higher value. Porosity of the composites increased with increasing mass fraction of nano SiC particles.

Acknowledgement

The authors thank the professor and head, Manufacturing Engineering Department, Annamalai University for providing fabrication and mechanical testing facilities. The authors also thank CDSL, Department of Physics, Annamalai University for the support in using AFM and SEM for experimentation. The authors are gratefully acknowledging Prof. and Dr. N. SELVAKUMAR, the director, Nano Centre of MepcoSchlenk Engineering College, Sivakasi, Tamil Nadu, India.

References


纳米 SiC 颗粒增强 Ti–6Al–4V 基复合材料的制备和力学性能

G. SIVAKUMAR, V. ANANTHI, S. Ramanathan

1. CISL, Department of Physics, Annamalai University, Tamilnadu 608002, India;
2. Physics Section, FEAT, Annamalai University, Tamilnadu 608002, India;
3. Manufacturing Engineering Department, Annamalai University, Tamilnadu 608002, India

摘要：采用粉末冶金技术制备含不同质量分数(0, 5%, 10%和 15%)纳米 SiC 颗粒增强的 Ti–6Al–4V(Ti64)金属基复合材料(MMCs)，研究添加纳米 SiC 颗粒对复合材料力学性能如硬度和抗压强度的影响。结果表明，当压缩载荷为 6.035 MPa 时，复合材料具有最佳的相对密度(93.33%)。SEM 显微组织观察结果表明，Ti64/5%SiCp 复合材料的润湿性和结合力得到了提高。采用 X 射线衍射技术研究 Ti64/SiCp 复合材料中纳米 SiC 颗粒含量对其相组成的影响，并分析其力学性能与相组成的关系。含 10%和 15% SiCp 的复合材料由于脆性界面反应产生了新相，此新相对复合材料的强度和硬度不利。Ti64/5%SiCp 金属基复合材料的抗压强度和硬度较高，因此 5% SiCp 是此复合材料的最佳含量。

关键词：钛合金；纳米 SiC 颗粒；粉末冶金；硬度；抗压强度

(Edited by Wei-ping CHEN)