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

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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 SiC_p composites. The effect of nano SiC_p content in Ti64 alloy/SiC_p 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 reaction formed in the 10% and 15% SiC_p composite specimens and resulted in no beneficial effect on the strength and hardness. The compressive strength and hardness of Ti64 alloy/5% nano SiC_p MMCs showed higher values. Hence, 5% SiC_p 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 fiberreinforced 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,

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TiO₂, SiC, TiB, and TiB₂[11]. The mechanical properties of MMCs can be further enhanced by decreasing the size of ceramic particulate and/or matrix grains from micrometer to nanometer level [12–14]. The nano SiC particle is better choice for reinforcement of TMCs matrix. Among various fabrication processes, powder metallurgy (PM) is the most diverse manufactured approach. One attraction of PM is the ability to fabricate high quality complex parts to close tolerances in an economical manner. In essence, PM makes a net shape, high performance and less prone to porosity and defects [15].

The purpose of the present work is to produce nano SiC_p by using high energy ball milling, fabricate SiC_p -reinforced metal matrix composites by powder metallurgy method and investigate the effect of nano SiC particle content on the porosity and mechanical properties of nano SiC_p -reinforced TMCs.

2 Experimental

2.1 Materials

Ti-6Al-4V alloy was used as the matrix material and SiC particles with average particle size of 20 µm were used as the reinforcement. The SiC powder was supplied by Alfa Aesar A13561, German. In this work, titanium powder of 99% purity (average particle size of 44 µm, supplied by Kemphasol, Mumbai), aluminium fine powder of 99.7% purity (average particle size of 44 µm, supplied by Lobachemi, Mumbai) and vanadium powder of 99.5% purity (average particle size of 44 um, supplied by Aesar Alfa) were used as raw materials for this study. An industrial standard titanium alloy mixture, titanium powder 90% (mass fraction), aluminium powder 6% (mass fraction) (α -phase stabilizer) and vanadium 4% (mass fraction) (β -phase stabilizer) were mechanical alloyed to make Ti6Al4V alloy (Ti64 alloy). All the powders were milled in a high energy ball mill to obtain homogeneous alloy [16].

2.2 Production of nano SiC_p

The size reduction of SiC from micron to nano level (<75 nm) was achieved by high energy planetary ball mill (Retsch, PM 100). The following parameters were the milling parameters such as ball to powder mass ratio (BP ratio 40:1), constant milling time (8 h) and constant rotational speed (300 r/min) [17,18]. The wet milling method was used to mill the powder. The organic compound toluene (C_6H_6 —CH₃) was filled in the ball mill chamber in adequate quantity [19] and its level was frequently checked. After the end of one complete cycle time, the milled powders with toluene were poured into a pyrex container, dried in an oven at 70 °C for 24 h, de-agglomerated in a mortar and sieved.

2.3 Production of composites

Different mass fractions (0, 5%, 10% and 15%) of nano SiC particle-reinforced Ti64 alloy metal matrix composites were produced by using powder metallurgy technique. The basic steps in powder metallurgy (PM) include mixing, compacting, and sintering of mixtures of powders constituting the matrix and reinforcements. After the mixing operation, mixtures of powders are pressed (compacted) in a die at the pressure that makes the particle adhere at contacting points. 100 t hydraulic pressing machine [20,21] was used for compaction. The blended powder mixtures were cold compacted by using the suitable punch and dies set assembly [22]. Several designated pressures of 4.365, 5.432, 5.82 and 6.305 MPa [23] were used to make cylindrical specimens. The maximum density was obtained to be about 93.33% in compaction pressure of 6.305 MPa with a diameter of 31 mm and a length of 22.85 mm.

The compacts were heated at 125 °C for 2 h for the vaporization of solvent and decomposition of stearic acid. They were then placed in the vacuum chamber and heated to 250 °C at vacuum of 5.33×10^4 Pa for 4 h to remove the residual gases.

The green compacts having the maximum density were subsequently sintered at 1200 °C in high temperature tubular furnace with argon atmosphere. Choking time of 2 h was maintained and followed by cooling to room temperature in the furnace itself [24,25]. Similar procedure was adopted for preparing Ti64 alloy/ nano SiC_p composites. The hot extrusion was carried out in the 150 t hydraulic press with argon atmosphere. Alloy and composite billets were loaded into the container and the die assembly was heated to 850 °C using a split type resistance furnace for 30 min. The billet temperature was measured by using a chromel/alumel thermocouple located at 2 mm away from the inner wall of the container. When the desired temperature was reached, the billets were soaked for 30 min and extruded in the ratio of 2. Liquid paraffin with graphite mixture (mass ratio of 5:1) was used as a surface lubricant. The extrusion process was carried out not only for producing structural shapes but also to get more homogeneous particle distribution with the refinement of the particle size. Also, the extrusion process minimized the compaction defects in the composites.

From the hot-extruded billets (31 mm in diameter and 22.85 mm in length), the cylindrical specimens of required dimensions (9 mm in length and 6 mm in diameter) were machined (wire cut) using wire cutting machine and used for further study.

2.4 Density measurement and porosity

The experimental density (bulk density) of the composites was obtained by Archimdian method. The

theoretical density was calculated using the rule of mixture. Bulk density was calculated by using Eq. (1) [26]:

$$\rho_{\rm B} = \frac{m_{\rm d}}{m_{\rm sat} - m_{\rm sus}} \times \rho_{\rm H_2O} \tag{1}$$

where $\rho_{\rm B}$ is the bulk desity, $m_{\rm sat}$ is the saturated mass, $m_{\rm d}$ is the dry mass, and $m_{\rm sus}$ is the suspended immersion mass.

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

$$P = \frac{m_{\rm sat} - m_{\rm d}}{m_{\rm sat} - m_{\rm sus}} \times 100\%$$
(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 μ m 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 SiC_p 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 μ m. 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 K_{α} 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 SiC_p observed is 75 nm. The surface of the SiC_p is found as irregular and rough shape.



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 SiC_p . The results reveal that the size reduction of SiC_p 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 SiC_p are shown in Fig. 2. It can be seen that the theoretical density of 5% nano SiC_p composites increases and that for 10% and 15% SiC_p composites (as expected from the rule of mixtures). The same trend was also observed in bulk density (Archimedies 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 SiC_p composite has a very low porosity among the other three composites with different contents of SiC_p. The result suggests that 5% of nano SiC_p is effective, due to the removal of pores and it enhances the mechanical properties of the composites. More pores are filled by nano SiC_p and therefore, the number of open pore decreases, at 5% nano SiC_p composite.



Fig. 2 Physical properties of experimental composites

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% SiC_p and 15% SiC_p. When the nano SiC_p 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 SiC_preinforced alloy composite can be written in the following sequences, i.e., Ti alloy/5% nano SiC_p> Ti alloy>Ti alloy/10% nano SiC_p>Ti alloy/15% nano SiC_p.

3.3 X-ray diffraction analysis

The XRD patterns of Ti64 alloy and Ti64 alloy/nano SiC_p 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 Ti_5Si_3 .



Fig. 3 XRD patterns of Ti64 alloy (a) and Ti64 alloy/5% nano SiC_p (b), Ti64 alloy/10% nano SiC_p (c) and Ti64 alloy/15% nano SiC_p (d) composites

$$SiC+Ti \rightarrow Ti_xSi_v+TiC$$
 (3)

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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% SiC_p specimen due to the hexagonal structure of silicon carbide (JCPDS card No. 29–1128).

The intensity of α -Ti phase (peak) significantly decreased in 10% and 15% SiC_p 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% SiC_p specimen. The high intensity of TiC is formed at 2θ =41.43° (200), and 60.30° (220) (JCPDS card No. 06–0614) in 15% SiC_p specimen. The reinforcement material SiC_p is formed at 2θ =73.39° (203) (JCPDS card No. 29–1128) in 10% SiC_p specimen and higher intensity is observed at 2θ =35.62° (102) and 72.40° (202) in 15% SiC_p 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 SiC_p 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 SiC_p (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



Fig. 4 SEM images of experimental composites: (a) Ti64 alloy; (b) Ti64 alloy/5% nano SiC_p composite; (c) Ti64 alloy/10% nano SiC_p composite; (d) Ti64 alloy/15% nano SiC_p composites

significantly than the micro-sized ceramic particles. It clearly showed that, there were no voids and discontinuities, when nano SiC_p 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_p (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_p and Ti64 alloy particle. Addition of nano SiC_p 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_p sites and the contact surface area increases, which would result in higher porosity level [36]. Increasing the mass fraction of nano SiC_p leads to the formation of TiC and Ti₅Si₃ 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_p 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_p 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_p 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_p 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_p 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_p 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_p composite; (c) Ti64 alloy/10% nano SiC_p composite; (d) Ti64 alloy/15% nano SiC_p composite



Fig. 6 Vickers hardness of Ti64 alloy and Ti64 alloy /nano SiC_p 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_p 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_p 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_p 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_p. 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_p 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

When the increase of the nano SiC_{p} reinforcement content is above the optimum value (5%), brittle interfacial reaction products TiC and Ti₅Si₃ 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 detritus 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 interfaced 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.

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纳米 SiC 颗粒增强 Ti-6Al-4V 基复合材料的制备和力学性能

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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%SiC_p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/SiC_p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% SiC_p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%SiC_p金属基复合材料的抗压强度和硬度较高,因此 5% SiC_p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

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