The Effect of Zirconium and Yttrium Oxide on the Microstructure and Mechanical Properties of Ti-Si-Zr-Based Composites

Vitus Mwinteribo Tabie,^{1,2} Xiao-jing Xu,^{1*} Li Jian Wei¹ and Xiaodong Shi¹

Abstract

Ti-4Si-xZr-yY₂O₃/5TiO₂ matrix composites, containing various weight percentages (0 and 1.3) of zirconium and (0 and 0.3) yttrium oxide, were synthesized via ball milling and powder metallurgical route. Mechanical alloying of the powders was confirmed after ball milling using x-ray diffraction (XRD) and scanning electron microscope (SEM). The ball-milled powders were cold compacted at a pressure of 550 MPa. Sintering was done first at 600 °C for 2h, followed by 800 °C for 2h, then 1000 °C for 2h, and finally at 1250 °C for 3h, all in a controlled vacuum environment. The effect of the addition in weight percentages of Zr and Y₂O₃ on the microstructure, porosity, hardness, elastic modulus, and fracture toughness of the sintered composites, was studied. The results show improvements in the microhardness by 2.2 times, elastic modulus by 34.3%, porosity by 50%, and fracture toughness by 57%, when Zr and Y₂O₃ content was 1.3 wt% and 0.3 wt%, respectively. The improvements in the microstructure and mechanical properties can be attributed to the formation of more stable; Ti₅Si₃ and Ti₅Si₄ phases and the solid solutions of Ti₂Zr, Zr₃Si₂, and Ti₂Zr₃Si₃ compounds.

Keywords: Ti-Si alloy; Matrix composites; Microstructure; Mechanical properties.

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1. Introduction

Titanium matrix composites (TMCs) are used widely in automotive, aerospace, rail, marine, biomedical, and petrochemical structures because of their high strength, lightweight, high melting point, good biocompatibility, and excellent corrosion resistance.^[1-3] Over the past years, the development of TMCs relied on particulates and whiskers, notably SiC, TiC, TiB₂, TiB, TiN, ZrC, B₄C, and Al₂O₃.^[4-6] SiC, when used as reinforcement, produces titanium silicides (Ti-Si) compounds. These compounds maintain the strength of the composites at high temperatures, ensure low densities, high melting points, and good oxidation-resistant. Hence, the Ti-Si compounds are high-performance compounds, with many application prospects, especially in aviation.^[7,8] At high temperatures, its most stable state, Ti₅Si₃, acts as reinforcement in the α-Ti matrix phase.^[9-11] The addition of metallic elements to the Ti-Si system also increases the

performance of the composite materials significantly through the formation of M_xSi_y-type compounds.^[12] The M_xSi_y-type compounds change the structure and phase composition of the composite through improved grain refinements, reaction efficiency, density, and porosity.^[13,14] For example, elemental Zirconium (Zr) in the Ti-Si matrix forms a Ti-Si-Zr solid solution, which improves the creep resistance, hardness, and yield strength of the composite.^[7,15,16] Qiao *et al.*^[17] observe that Zr has little effect on the α and β phases of the Ti-Si system and maintains good high-temperature stability.

Oxides such as Aluminum oxide (Al₂O₃) and yttrium (Y₂O₃) have also been adopted widely as reinforcements in alloys because of their outstanding dimensional and structural stability.^[18-20] Y₂O₃, for example, is reported to have a potential in microalloying in titanium alloys and can be used as an agent for grain refinements.^[21] Zr and Y₂O₃ effectively refine grains of the base alloy structure by rare earth activity and uniformly distribute in and change the shape of this base structure.^[22,23] The present study selects Ti-4Si series composite material as the base sample because the Si content of 4 wt.% can effectively reduce the brittleness^[24] of the material and prolong the service life. The addition of nano-TiO₂ particles can form a solid solution, which helps improve the mechanical

¹ Institute of Advanced Forming Technology School of Mechanical Engineering, Jiangsu University, China.

² Department of Mechanical Engineering, Hilla Limann Technical University, Ghana.

*Email: xjxu67@126.com (X. Xu)

properties and high-temperature oxidation resistance of the material.^[25] Besides the many processing techniques employed in processing Ti-Si-based composite, mechanical alloying (MA) followed by reaction sintering best improve hardness and mechanical properties.^[15,26-28]

This paper selects the technology of ball milling and cold pressing sintering to prepare four composites: Ti-4Si/5TiO₂, Ti-4Si-1.3Zr/5TiO₂, Ti-4Si-0.3Y₂O₃/5TiO₂, and Ti-4Si-1.3Zr-0.3Y₂O₃/5TiO₂ hereafter labeled M1, M2, M3, and M4, respectively. An earlier study by authors^[29] showed that M4 composites possess excellent high-temperature corrosion and oxidation resistance. The present study examines the phase composition, microstructure, apparent porosity, hardness, modulus of elasticity, and fracture toughness of these composite materials. This study will provide further insight into this material as a possible candidate material for high-temperature application.

2. Experimental procedure

2.1 Preparation of Ti-4Si-xZr-yY₂O₃/5TiO₂ composites

The starting powders used in the study were titanium (99.0%, 250 mesh), silicon (99.9%, 200 mesh), zirconium (92.0%, 300 mesh), nano yttrium oxide (99.99%), and nano titanium dioxide (99.99%), all obtained from the Sinopharm Chemical Reagent Company Limited, China. The raw materials were weighed in appropriate quantities and used to prepare the various composites, as shown in Table 1.

Table 1. Composite powder number and distribution ratio.

Serial number	Composition / (wt.%)					Composite
	Ti	Si	Zr	Y ₂ O ₃	TiO ₂	
1	91	4	-	-	5	M1
2	89.7	4	1.3	-	5	M2
3	90.7	4	-	0.3	5	M3
4	89.4	4	1.3	0.3	5	M4

The mixed powders were ball-milled at room temperature using a YXQM-4L planetary ball mill, which contained Al₂O₃ balls with diameters of 5 mm and 10 mm. A single mill selected was for 55 min, with an interval of 5 min, and a speed of 300 rpm. The total milling time was 48 h. The grinding ball-to-powder mass ratio was 8:1. After milling, the slurry was taken and dried in a vacuum oven at a temperature of 80 °C for 8 h. The heating rate was at 5 °C/min. A uniformly refined alloyed powder was obtained after sieving with a 200 mesh sieve. The alloyed powder was dried in a vacuum oven at 80 °C for 3 h, quantitatively weighed, and put in a 30 mm diameter preform mold. The cold pressing was done using a hydraulic machine model YB32-100 under a pressure of 550 MPa and held for about 3 min. The pre-compressed samples were sintered in a WZS-20 double-chamber vacuum sintering furnace. The furnace was set at a vacuum pressure of 1×10^{-1} Pa and a heating rate of 10 °C/min. The step-temperatures

placed were at 600 °C for 2 h, 800 °C for 2 h, 1000 °C for 2 h, 1250 °C for 3 h, and finally cooled.

2.2 Characterization

The microstructure and grain size of the composites were observed under scanning electron microscopy S-3400N model with a variable vacuum tungsten filament. The microstructure of the composites was measured using US energy dispersive spectroscopy (US.EDS). The samples were machined to the size 10mm × 10mm × 3mm, and polished. Hydrofluoric acid, nitric acid, and distilled water were used, in turn, to treat the samples. The treatment helped to remove the corroding effect before the microstructural characterization. The X-ray diffraction (XRD) patterns and phase compositions of the powder and composite materials were recorded with a D8-ADVANCE diffractometer. Its parameters Cu K α radiation 40 kV, 200 mA within the 20-90° range (2 θ), a step of 0.01° and a scanning speed of 5 °/min, were set in place. The apparent porosity of the composite materials was measured using the Archimedes principle. The samples were dried in an oven at 120 °C for 2 hours and removed and weighed in an air and vacuum box (1×10^{-1} MPa pressure). Fig. 1 illustrates the preceding steps after taking the sample weight in a vacuum. The apparent porosity was calculated using appropriate formulas.

Fig. 1 Steps in measuring apparent porosity: (I) Take the weight of water and beaker (II) completely immerse the sample in water and take weight (III) shear the thin line and allow the sample to sink to the bottom. Take the total weight.

2.3 Mechanical performance testing

The Vickers hardness tester model HV-1000 was selected to measure the microhardness and fracture toughness of the composite employing appropriate formulas. Experimental parameters were 0.98N force and loading time 10s. The elastic modulus of the composites after sintering at 1250 °C was measured using the Impulse Excitation Technique (IET). The setup of the experiment is illustrated in Fig. 2. The Young's modulus (E) of the rectangular-shaped solid specimen was calculated using equations (1)^[30,31]

$$E = 0.946 \left(\frac{mf_f^2 l^3}{bh^3} \right) \left(1 + 6.585 \left(\frac{h}{l} \right)^2 \right) \quad (1)$$

where f_f is the natural frequency, b is the width, h is the sample thickness, m is the mass, and l is the length of the sample.

3. Results and discussions

3.1 Phase and Microstructure of milled powders

Fig. 2 Setup of IET-01 experiment and working principle.

Figures 3(a)–(d) show the SEM images of milled composite powders. The powders after ball milling are mainly in the form of small particles, block flakes, and some agglomerated pellets. They are evenly refined, and mechanical alloying occurred with Ti_5Si_3 , Ti_5Si_4 , and TiSi_2 phases appearing in all four formulations, as shown in the XRD pattern in Fig. 4. The addition of the Zr element enhances the peak of Ti_5Si_4 and introduces solid solutions such as Ti_2Zr and Zr_3Si_2 . Improvements in the Ti_5Si_3 , Ti_5Si_4 , and TiSi_2 phases were recorded when Y_2O_3 was also added. While Zr is known to promote the formation of stable Ti_5Si_3 compound,^[32] Y_2O_3 , on the other hand, promotes Si and Ti elements reactions.^[33] Muthaiah and Mula^[34] also reported the improved mechanical alloying effect of Zr in nanocrystalline aluminum alloy. The combined effect of Zr and Y_2O_3 was powder agglomeration and improved alloying, as seen in Fig. 3d. Besides, the phases of TiSi_2 and TiSi compounds decreased while the stable Ti-Si got enhanced.

3.2 Phase and microstructure of composites

X-ray diffraction analysis of the sample sintered at 1250 °C (see Fig. 5) shows that the four composites mainly contain diffraction peaks Ti_5Si_3 and Ti_5Si_4 and solid solutions of Ti_2Zr , Zr_3Si_2 , and $\text{Ti}_2\text{Zr}_3\text{Si}_3$ when the elemental Zr was added. The intensity of the diffraction peaks of these phases increased with the addition of elemental Zr and Y_2O_3 oxide in composite M4. It indicates that elemental Zr can promote the conversion of Ti-Si compounds into the Ti_5Si_3 phase and induce solid solution strengthening.^[35] The Y_2O_3 oxide also promotes the reaction of Ti and Si elements and brings about dispersion strengthening.^[36] From the SEM morphology in Fig. 6 and EDS results in Table 2, Ti and Si were detected throughout the matrix in composite 1, as the uncorroded dark grey region marked 1. The Ti_5Si_3 compounds are pure white platelets numbered 3 and uniformly distributed in the matrix of composites 3 and 4. The gray areas marked 2 are mainly titanium-zirconium solid solutions of Ti_2Zr , Zr_3Si_2 , and $\text{Ti}_2\text{Zr}_3\text{Si}_3$.

Fig. 3 SEM morphology of ball milled composites powders (reproduced with permission from^[29] copyright @ IOP Publishing Ltd) (a) M1 (b) M2 (c) M3 (d) M4.

Fig. 4 XRD diagrams of ball-milled composite powders.

Table 2. EDS element analysis of regions (reproduced with permission from^[29] copyright @ IOP Publishing Ltd).

Composite	Region	Composite (wt.%)			
		Ti	Si	Zr	Y ₂ O ₃
M1	1 Dark gray	98.6	1.4	-	-
	2 Gray	96.5	3.5	-	-
	3 Pure White	74.6	25.4	-	-
M2	1 Dark grey	95.07	3.84	1.09	-
	2 Grey	96.25	2.89	0.86	-
	3 Pure White	77.29	22.42	0.31	-
M3	1 Dark Grey	93.27	4.36	-	2.37
	2 Grey	92.89	6.85	-	0.26
	3 Pure White	73.58	26.34	-	0.08
M4	1 Dark Grey	96.66	2.36	0.86	0.12
	2 Grey	94.78	3.68	1.22	0.32
	3 Pure White	74.99	24.84	0.11	0.06

3.3 Mechanical properties

Table 3 summarizes the mechanical properties of the Ti-4Si-xZr-yY₂O₃/5TiO₂ composites. Both elastic modulus and the hardness of the samples containing Ti₅Si₃, Ti₅Si₄, Ti₂Zr, Zr₃Si₂, and Ti₂Zr₃Si₃ phases are higher than the initially formulated composite. The elastic modulus of the M4 composite is about 120.68 GPa, which is about 34% higher than that of the M1 composite. M4 composite also has a maximum microhardness of 1478 HV, which is 2.2 times (119.9%) higher than the M1 composite. Similarly, the composite with Zr and Y₂O₃ additions also recorded higher elastic modulus and microhardness values. The improved mechanical properties of Ti-4Si-xZr-yY₂O₃/5TiO₂ composite may be attributed to the following two factors according to the SEM images shown in Fig. 6. Firstly, because of the increase in Ti₅Si₃, and Ti₅Si₄

peaks compared to the base composite, as seen in Fig. 5. These phases enhance the microhardness and wear resistance of alloys. For example, Tian *et al.*^[37] observed that the Ti₅Si₄ in Y-Ce modified silicide coating possessed a higher microhardness than the pure silicide coating. Secondly, the formation of solid solutions of Ti₂Zr, Zr₃Si₂, and Ti₂Zr₃Si₃ compounds strengthens the material and increases the microhardness. The effects of Zr solid solutions on microhardness have been reported by Suntharavel *et al.*^[34] A combination of these factors may result in the experimentally observed increase in hardness with the addition of Zr and Y₂O₃ oxide. The microhardness of the M3 composite is lower than the M2 composite because Y₂O₃ oxide stabilizes the β phase by binding the oxygen in the composite and reducing the hardness.^[38]

Fig. 5 XRD diagrams of sintered composites.

Table 3. Summary of mechanical properties of Ti-4Si-xZr-yY₂O₃/5TiO₂ composites.

composite	Porosity (%)	Youngs Modulus at RT (GPa)	Microhardness (HV)	Fracture Toughness (MPa. m ^{1/2})
M1	5.8	89.88	672	55.3
M2	6.7	108.71	1048	62.5
M3	3.8	95.85	859	58.9
M4	2.9	120.68	1478	86.8

The M2 composite has a higher apparent porosity (6.7%) than the M1 composite (5.8%) because the Zr element during vacuum sintering promotes the reaction, and releases a large amount of heat which causes the gas retained in the sintered body to expand with the increase in temperature. The grain structure gets pressed against each other, and voids and other defects occur. M4 composite material exhibits the lowest porosity of only 2.9%, which is about 50% of the apparent porosity of M1 composite and 56.7% lower than that of M2 composite.

The fracture toughness K_{IC} values were calculated using equation (2).^[39]

$$K_{IC} = 0.015 \frac{P_C}{a_c^{3/2}} \left(\frac{E}{H} \right)_I^{1/2} \quad (2)$$

where, K_{IC} is fracture toughness, unit: MPa/m^{1/2}; E is elastic modulus, unit: GPa; H is Vickers hardness value, unit: HV; P_C is load, unit: N; a_c is crack radius length, Unit: mm; d is the diagonal length of the indentation, unit: mm.

Table 4 shows the mechanical properties of M4 and some titanium alloys with similar contents of Zr of yttrium and the

Table 4. Mechanical properties of M4 and some titanium alloys.

Condition	Alloy	Elastic Modulus (GPa)	Fracture Toughness (MPa. m ^{1/2})	Microhardness (HV)	Reference
-	Ti-6Al-4V	110-140	-	300-400	[40]
-	Ti-6Al-4V	110	78.3	-	[41]
sintered	Ti-6Al-2Zr-1Mo-1V	141	74-90	357	[42]
Forged + HT	Ti-6Al-2Zr-1Mo-1V	-	-	-	[43]
sintered	Ti-4Si-1.3Zr-0.3Y ₂ O ₃ /5TiO ₂	120	86.8	1478	This work
Forged	Ti-5.9Al-2.7Sn-4Zr-0.45Mo-0.35Si-0.22Y	120	-	320-350	[44]
As-cast	Ti-6Al-2.5Sn-4Zr-0.7Mo-0.3Si-0.5Y	-	-	590.8	[45]

*HT – heat treatment

Fig. 6 SEM morphology of the surface of sintered composites (a) M1 (b) M2 (c) M3 (d) M4.

commercial Ti-6Al-4V alloy. The M4 composite displays excellent mechanical properties in comparison with the other alloys. It has the highest microhardness, which can be attributed to the addition of zirconium. Zirconium's hardness ranges between 1350 HV and 1470 HV.^[46] Besides, it has comparatively good tensile and fracture toughness properties. The M4 composite is thus a possible substitute for the other alloys which are already in a commercial application.

4. Conclusion

The strengthening mechanism of titanium matrix composites is mainly through solid solution hardening, dispersion strengthening, and grain refinement strengthening. Among these, solid-solution strengthening plays a significant role in the properties of the Ti-4Si-xZr-yY₂O₃/5TiO₂ composites. Four composites, which contain various weight percentages (0 and 1.3) of Zirconium and (0 and 0.3) yttrium oxide, were successfully produced via powder metallurgical routes. With the addition of elemental Zr and Y₂O₃ oxide, the mechanical properties of the composites got enhanced. The reinforcements promoted the formation of solid solutions of Ti₂Zr and Zr₃Si₂ and the enhancement of the Ti₅Si₃ phase. The M4 composite material has the lowest porosity of 2.9%. There was an improvement in its microhardness (1478 HV) which is 2.2 times that of the M1 composite (672 HV). It also has the highest elastic modulus of 120.68 GPa. The additions also led to an improvement in the fracture toughness of the composites. The KIC value of M4 composites was the highest and best, reaching 86.8 MPa.m^{1/2}.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

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Authors information

Vitus Mwinteribo Tabie is a Lecturer at the Department of Mechanical Engineering, Hilla Limann Technical University, Ghana. His research focuses on Powder metallurgy, Materials Characterization, alloys and composites materials. He received his PhD degree from the Institute of Advanced Forming Technology, School of Mechanical Engineering, Jiangsu University, China.

Xiaojing Xu is a distinguished Professor and leader of the Institute of Advanced Forming Technology, School of Mechanical Engineering, Jiangsu University. Prof. Xu does research in advanced large strain technology and nanomaterials by severe plastic deformation, advanced forming technology, equipment design and manufacturing, Biomedical metallic materials and surface modification, Materials characterizations theories and technologies of microstructures and properties etc. His current research is in new generation super aluminum alloy.

Jianwei Li currently works at the Institute of Advanced Manufacturing and Modern Equipment Technology, Jiangsu University. Jianwei does research in Materials Engineering. Their current project is 'Interfacial design for high thermal conductivity composite material'.

Xiaodong Shi is a master student at the Institute of Advanced Forming Technology, School of Mechanical Engineering, Jiangsu University. Xiaodong does research in Materials Engineering. His current project is into the development of high-temperature Titanium alloy.

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