Spatial and microstructural dependence of mechanical properties and wear performance of functionally graded Al–TiAl₃ in situ composite

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Abstract

Effect of morphology and spatial distribution of TiAl₃ particles on the hardness, the tensile behavior and the wear performance of functionally graded Al–TiAl₃ in situ composite (FGC) was investigated. Initially, FGC was produced by interaction between solid Ti and liquid Al. Based on the spatial distribution and morphology of TiAl₃ particles in the microstructure of FGC, three distinctive regions including blocky-particle region, mixed-particle region (blocky and short plate particles) and lengthy plate-particle region were studied. Results of this study showed that the blocky-particle region had promoted tensile strength as well as highly improved wear resistance. In addition, this region with higher density of blocky particles and low interparticle spacing showed a brittle fracture. The mixed-particle region with a lower density of blocky particles had a ductile fracture mechanism while the plate-particle region showed cleavage fracture. The dominant wear mechanism for regions including blocky particles was determined to be mild oxidation while it was delamination wear for regions containing plate particles. Finally, correlation between mechanical properties, wear resistance and microstructure of FGC was discussed.

Keywords Microstructure · Mechanical properties · Wear resistance · Functionally graded Al–TiAl₃ in situ composite · Particle spatial distribution · Morphology

1 Introduction

TiAl₃ particles can significantly improve mechanical properties and wear resistance of Al-based composites due to their high melting point, Young's modulus and hardness values [1, 2]. However, microstructural features are mainly responsible for the resulted performances. Several investigations have studied the effect of TiAl₃ area fraction on wear and tensile properties without considering the effect of their morphology [3–5]. Since the results reported for the wear resistance of the Al–TiAl₃ composites were very scattered, authors believe that another parameter such as morphology of intermetallic particles can strongly affect the mechanical properties, in particular tensile behavior

as well as wear resistance. As a tangible example, comparison of results of the wear performances of the Al–TiAl₃ composites in two studies containing only plate-like particles [6, 7] and only blocky particles [5] showed completely different wear resistances. This might be due to not only particle area fractions, but also their morphologies.

Different morphologies of intermetallic particles may be achieved by adopting an appropriate production process. Authors of the present work have successfully produced functionally graded Al–TiAl₃ in situ composite (FGC) by interaction between liquid Al and solid Ti [8]. Thereafter, by a thermal treatment, a spatial gradient distribution of both morphology and area fraction of TiAl₃ particles in Al matrix was obtained. Afterward, wear and mechanical properties

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of three different regions of fabricated FGC containing different area fractions as well as various morphologies of intermetallic particles were investigated separately. Finally, correlation between wear behavior, mechanical properties and microstructural features of Al–TiAl₃ FGC was discussed.

2 Materials and methods

A functional gradient Al/Ti in situ composite (FGC) was fabricated by the interaction of solid Ti insert with surrounded Al melt. Details of the fabrication method have been reported previously by authors [8]. Fabricated samples underwent a thermal treatment in 1173 K for 180 min, resulting in distribution of two morphologies of TiAl₃ intermetallic particles in Al matrix. Blocky, mixed (blocky and small plates) and plate-like intermetallic particles were found in the three distinctive regions in Al matrix, respectively. Figure 1 shows the positions of these regions in the fabricated FGC. Based on the distribution of intermetallic particles in Al matrix, three samples including blocky-particle region, mixed-particle region (blocky and small

plate) and plate-particle region were cut and prepared for mechanical and wear evaluations. These samples will be referred as “blocky,” “mixed” and “plate,” respectively. The positions and dimensions of these samples are shown in Fig. 1.

The tensile specimens were prepared according to dimensions given in Fig. 1b. The tensile test was carried out by an Instron tensile machine at the strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$ and at the ambient temperature. The fracture surfaces were analyzed using scanning electron microscope (SEM) equipped with the energy-dispersive spectroscope (EDS). In addition, microhardness of each phase was measured using a Vickers indenter under a test load of 0.5 N in accordance with the ASTM standard E384-10.

Dry sliding wear test was conducted according to ASTM G99 standard using a pin-on-disk apparatus and an AISI 52100 carbon steel pin with a hardness of 64RC. The rotation speed, normal load and total sliding distance were 100 rpm, 2 N and 1000 m, respectively. For comparison, the wear behavior of a sample cut from pure aluminum was also investigated. Mass loss of specimens was measured using a balance with a sensitivity of $\pm 0.1 \text{ mg}$. Density of samples was measured using the Archimedes' method. The worn surface, debris and the subsurface microstructures were examined using a field-emission scanning electron microscope (FESEM).

3 Results

3.1 Microstructure observation

Microstructure analysis of fabricated FGC showed that there were three main regions (from Ti insert to the periphery) in the Al matrix. Figure 2 shows a schematic illustration of these regions on the cross section of the FGC sample. It also shows the SEM images of three regions containing blocky, mixed and plate intermetallic particles distributed gradually in Al matrix. More specifically, particles close to the Al/Ti interface were blocky, thereafter, mixed particles including both blocky and short plates followed by lengthy plates which were close to the periphery of the FGC sample. The aspect ratio of lengthy plate particles was measured using Focus image analysis software [9], and their length-to-width ratio was about 15 times more than that of blocky particles.

The composition of all particles was determined by elemental analysis using energy-dispersive spectroscope (EDS) integrated with an SEM (Fig. 3a) as well as phase detection by X-ray diffraction analysis (XRD) which is shown in Fig. 3b. Both blocky and plate particles had similar composition and were determined to be TiAl₃.

Fig. 1 **a** Positions and dimensions of the evaluated regions in the fabricated FGC and **b** the dimensions of tensile specimen

Fig. 2 Schematic illustration and corresponding SEM images of the distinctive regions on the cross section of the FGC sample

Fig. 3 **a** Backscattered scanning electron microscope image of blocky and plate TiAl_3 particles together with energy-dispersive elemental analysis and **b** X-ray diffraction pattern containing both blocky and plate particles

3.2 Mechanical properties

3.2.1 Hardness and microhardness

Area fraction of TiAl_3 particles (determined using Focus [9] image analysis software) and hardness of FGC samples are listed in Table 1. As it can be noticed, increment in area fraction of TiAl_3 particles led to increment in hardness of samples.

The average value of six measurements of microhardness of each phase is shown in Fig. 4. As can be seen, the hardness value of intermetallic particles in both blocky

Table 1 Area fraction of TiAl_3 phase and hardness of FGC samples

Nomenclature of sample	Area fraction of TiAl_3 (%)	Hardness (HV)
Blocky	26 ± 0.9	48 ± 3.0
Mixed	11 ± 1.2	33 ± 3.4
Plate	6 ± 0.3	36 ± 0.9

and plate shapes was very similar but much higher than that of base materials (i.e., pure Al and Ti).

In addition to crystal structure, microhardness values also depend on chemical composition of each phase [10].

Fig. 4 Microhardness values of blocky TiAl₃, plate TiAl₃, Al and Ti phases

More specifically, TiAl₃ is a nonstoichiometric intermetallic compound with a range of composition. Therefore, a small difference in microhardness values between blocky (containing 37.8 ± 0.4 wt% Ti) and plate-like (containing 37.1 ± 0.9 wt% Ti) TiAl₃ particles could be due to small variation in Ti concentration and/or measurement errors (as shown in Fig. 4).

3.2.2 Tensile properties

The measured stress–strain curve of all samples (blocky, mixed, plate) and also that of the pure Al [11] are shown in Fig. 5. It was found that the blocky sample had the highest amount of yield strength and modulus of elasticity which were 230% and 117% higher than those for pure Al, respectively.

Fig. 5 Stress–strain curves for three tested FGC samples and pure Al adopted from Singh et al. [11]

In contrast, plate sample had both lower yield strength and Young's modulus compared to pure Al. The mixed sample showed the highest amount of ductility as well as tensile strength. Modulus of elasticity, 0.2% yield strength ($\sigma_{0.2}$), ultimate tensile strength (UTS) and elongation (El %) are given in Table 2.

Figure 6 shows the fracture surfaces of three FGC samples. The fracture surface of blocky sample revealed individual TiAl₃ particles. Moreover, several cracks on the surface of particles in blocky samples were found (Fig. 6a). However, fracture surface of mixed sample revealed a large number of dimples with different sizes (Fig. 6b). In addition, a small number of cracked, debonded and pulled-out particles were observed on this sample. Examples of cracks as well as debonded particles are shown in magnified parts of Fig. 6a, and dimples as well as voids are shown in Fig. 6b. Fracture surface of plate sample showed cleavage facets with some large cracks (Fig. 6c).

3.2.3 Wear performance

The wear mass loss of pure Al, blocky, mixed and plate samples as a function of sliding distance is presented in Fig. 7. The results demonstrated that for any given specimen, the mass loss increased with increasing sliding distance. In particular, by increasing sliding distance, the increment in rate of mass loss for blocky samples was the lowest while it was the highest for plate samples.

Figure 8 shows the wear rates (i.e., inverse of wear resistance) of pure Al and three FGC samples. It was found that blocky sample had the highest wear resistance (by two times compared to that of pure Al), followed by mixed sample. The wear rate of plate sample was observed to be even more than that of pure Al.

Wear tracks, worn width and location of TiAl₃ particles are clearly visible on the worn surfaces shown in Fig. 9. The reported value for each track width (measured by image analysis) is the average value of ten measurements taken at different regions of track. The values decreased from 3.37 mm for the pure Al to 2.61 and 2.75 mm for blocky and mixed samples, respectively, while, the value was increased to 3.70 mm for plate sample.

Table 2 Tensile data obtained from stress–strain curves

Sample	Modulus of elasticity (GPa)	$\sigma_{0.2}$ (MPa)	UTS (MPa)	El. (%)
Blocky	148 ± 1.0	66 ± 2.3	103.0 ± 2.0	11.9 ± 0.7
Mixed	92 ± 0.9	58 ± 2.4	116.1 ± 1.3	26.3 ± 1.3
Plate	26 ± 1.5	9 ± 3.1	12.9 ± 0.8	11.7 ± 0.7
Pure Al [11]	68	20	28	30

Fig. 6 Secondary (SE) and backscattered (BS) SEM images of fracture surfaces of **a** blocky, **b** mixed and **c** plate samples

Fig. 7 Wear mass loss of pure Al, blocky, mixed and plate samples

Fig. 8 Wear rate of pure Al, blocky, mixed and plate samples

Figure 10 illustrates the SEM micrographs of the worn surfaces at higher magnification. Some voids are observed on the relatively smooth worn surface of blocky sample. There are some large cracks and some plowing perpendicular to the sliding direction on the worn surfaces of mixed sample, while plate sample showed a higher number of large cracks as well as abrasion and delamination signs. In the case of pure Al, poor delamination, abrasion and massive plastic deformation were observed.

After the wear test, debris were collected and are shown in Fig. 11. There were two types of debris morphology including fine and flake particles.

Fine and some agglomerated debris were observed in blocky sample, where EDS results (Table 3) showed that they included high oxygen content. The detected oxygen amount in blocky sample (33.8 wt%) was higher than that of mixed (8.7 wt%) and plate (0.8 wt%). It is

noteworthy that the debris from mixed sample consisted of agglomerated fine particles and also a low amount of flake particles. Plate sample showed more flake debris with high amounts aluminum detected by EDS (Table 3). In addition, wear debris of pure Al were mostly flakes composed of Al element.

Figure 12 shows the SEM micrographs of the subsurface (vertical to the sliding direction) of blocky and plate samples. An obvious difference was observed in wear depth of two samples. Moreover, voids, cracks and fragments were detected on the surface of a plate particle shown in Fig. 12b.

4 Discussion

Blocky sample with the microstructure shown in Fig. 2 has the highest yield strength and Young's modulus values among all samples (Fig. 5). Since the chemical composition of the matrix and intermetallic particles in all three composite samples was the same, the significant difference in tensile properties of these three composites can be ascribed to the differences in their microstructural parameters (including morphology and area fraction of the TiAl_3 particles).

It has been shown in previous studies that in aluminum–matrix composites, second phase particles can

act as load-bearing components and improve mechanical properties [12, 13]. The results of this study showed that the blocky and mixed samples containing 26 and 11 vol% intermetallic particles have higher yield strength values than those of pure aluminum. Moreover, Young's modulus increased by 60% with increasing the area percentage of particles from 11% (mixed sample) to 26% (blocky sample). A similar trend has also been reported for increasing intermetallic particles in other metallic samples [14]. As area percentage increases, the inter-particle spacing decreases, which in turn causes an increase in the load bearing capacity and therefore Young's modulus of samples.

In addition to the area fraction of the second phase, morphology or aspect ratio of the TiAl_3 particles (length-to-width ratio) had a significant effect on the tensile behavior. It was observed while plate sample had lower area fraction of TiAl_3 particles, it had lower yield strength but lower ductility compared to mixed sample. It was expected that by decreasing area fraction of intermetallic particles, yield strength decreases and meanwhile ductility increases. However, this was not the case for plate sample. Thus, it can be concluded that in addition to area fraction of intermetallic particles, morphology of them had a significant effect on tensile properties of samples.

Fig. 9 Backscattered FESEM images of the wear track of **a** blocky, **b** mixed, **c** plate and **d** pure Al samples

Fig. 10 FESEM images of the worn surface of **a** blocky, **b** mixed, **c** plate and **d** pure Al samples

There are methods for estimation of elastic modulus of composites. When reinforced particles are lengthened plates or fibrous, Young's modulus depends not only on loading but also on the orientation of particles. The transverse Young's modulus, E_C , can be determined by following equation [15]:

$$E_C = \frac{E_p E_m}{E_m V_p + E_p V_m} \quad (1)$$

where E_p , E_m and E_C are Young's modulus of the reinforcement particle, the matrix and the composite, respectively. Thus, the transverse Young's modulus can be lower (especially if the volume fraction of particles is too low) or higher than matrix depending on volume fraction and Young's modulus of both particles and matrix. In the current study, based on E_C relation, the Young's modulus of plate sample has only 6% of plate particle which is calculated to be lower than that of pure Al. This was also confirmed experimentally.

A stress concentration in the matrix-containing plates or fibers causes its transverse strengths to be much lower than those measured from its monolithic specimens [16]. As a result, plate sample containing large plate particles with high stress concentration factor showed lower yield strength than that of pure Al.

As noted, the aspect ratio of the plate particles in the plate sample is 15 times higher than that of the blocky particles found in blocky and mixed samples. Increasing the aspect ratio of particles leads to an increase in the stress concentration factor (Eq. 1 [17]) and, finally, an easier failure:

$$K_n = 2a/b \quad (2)$$

where a and b are the length and the width of particles, respectively. Furthermore, defects such as pores, cracks and impurities are more likely to exist in coarser particles; this leads to decrease in ductility as well as fracture toughness and increase in the probability of brittle fracture [18, 19]. Therefore, plate sample was more prone to fracture due to the presence of large plate particles compared to blocky and mixed samples.

Micrographs of fracture surfaces might be used to determine fracture mechanism of samples (Fig. 6). The presence of individual particles on the fracture surface and the low elongation percentage of blocky sample (11.9%) indicate that the fracture is more likely brittle. Moreover, plastic deformation was reduced due to higher area fraction of TiAl_3 particles, resulting in lower inter-particle spacing in Al matrix compared to mixed sample.

Considering the fracture surface of mixed sample as well as strain–stress diagram, it is more likely that fracture of this sample was ductile. In other words, the presence of

Fig. 11 FESEM images of the **a** blocky, **b** mixed, **c** plate and **d** pure Al wear debris

Table 3 EDS results of wear debris

Sample	O (wt%)	Al (wt%)	Ti (wt%)
Blocky	33.8 ± 5.2	60.6 ± 4.4	5.6 ± 0.9
Mixed	8.7 ± 2.5	77.0 ± 1.8	14.3 ± 1.1
Plate	0.8 ± 0.6	97.5 ± 0.3	1.7 ± 0.5
Pure Al	0.6 ± 0.3	99.4 ± 0.3	–

several dimples and voids in fracture surface was indicator of this type of fracture. In addition, due to large difference in Young's modulus values of particles and matrix and also larger inter-particle spacing, Al matrix had possibility to undergo plastic deformation. As a result, debonding occurred at the matrix/particle interface. In fact, with decreasing the percentage of the second phase (compared to blocky sample), the inter-particle spacing in Al matrix increases; and thus, the plastic deformation increases. This is in agreement with the model presented by Babout [20].

In general, increment in volume fraction of particles in the composites decreases the dislocation slip distance and as a result elongation of the composites decreases [21]. Accordingly, blocky sample having higher area fraction of particles compared to mixed sample showed lower elongation value.

However, the elongation of plate sample (having the lowest area fraction of particles among the composite samples) is unexpectedly lower than that of mixed sample. Authors believe that easy fracture of large plate particles in plate sample was responsible for this abnormal behavior. Figure 12b demonstrates fracture of plate particles during loading.

Because the cleavage facets appeared on the fracture surface of the plate sample, it seems that its fracture is of brittle transgranular type. Low percentage of elongation (11.7%), as well as the appearance of large cracks on the surface, indicates the sudden fracture of the sample.

In order to determine the dominant wear mechanism, investigating the results of wear test (weight loss, wear rate, worn surface and collected wear debris) simultaneously is necessary. Considering the low amount of weight loss, high percentage of oxygen in wear debris and relatively smooth and dark worn surface (shown in Fig. 9) of blocky sample, it seems that the dominant wear mechanism was mild oxidation wear. Oxidation wear occurs when the rate of oxide formation is greater than its removal rate [22]. The main kinetic factor in determining the results of competition between these two processes is temperature. The major source of temperature rise in the wear process is the heat generated by friction. Due to relatively low heat conductivity of TiAl_3 particles (1.15 W/m K [23], increasing

Fig. 12 FESEM images of worn subsurface of **a** blocky and **b** plate samples

the area fraction of particles reduces heat dissipation rate, increases local temperature of sample and finally increases the rate of oxide layer formation. The presence of agglomerates in the wear debris is also an evidence of increasing the temperature.

Due to lower area fraction of particles in mixed sample (compared to blocky sample), the oxide layer was unlikely to be compact enough to be protective. This might increase the contact between the abrasive agent and the surface of the sample, which eventually increased the wear rate of mixed sample compared to blocky sample.

The increase in wear rate of plate sample was due to the morphology of plate particles. As described previously, the plate particles were easily cracked and fractured. The pores and cracks created on a plate particle under the worn surface are shown in Fig. 12b. Subsurface cracks grew and coalesced, causing a part of the surface to be peeled off as flakes (the delamination process). The edges that were formed on the worn surface of the sample as a result of delamination process and the flake-like particles in the collected wear debris are visible in Figs. 10c and 11c. Chemical analysis of the wear debris (Table 3) showed that these particles contain low amounts of titanium and high percentages of pure aluminum. It has also been reported in previous studies that the fractured pieces of intermetallic particles can act as abrasive agents due to their high hardness values [19] which could accelerate and intensify the wear process. Therefore, produced fragments diminished the wear resistance of plate sample. On the contrary, blocky particles because of having a strong bond with Al matrix as well as high tensile strength improved the wear resistance of Al matrix.

In summary, there were three different regions with the difference in particle morphology as well as their volume fraction of these particles. Comparing samples gives a good proof for surveying the effect of morphology and area fraction of particles on mechanical properties. Blocky sample having 26% of TiAl_3 with blocky

morphology showed the best combination of wear resistance and mechanical properties, whereas mixed sample having 11% of TiAl_3 with both blocky and small plate-like morphology showed maximum values of ductility, but the wear behavior was comparable with blocky sample. So, the fabricated Al- TiAl_3 FGCs in this study have a potential application where there is a need for higher wear resistance at the surface and higher ductility in the core.

Comparing plate sample with pure Al, the negative effect of long plate particles is noteworthy. It can be improved by either increasing the volume fraction of particles or changing plate-like to harmless blocky morphology.

According to the results of this study, including effect of area fraction and morphology of TiAl_3 particles on yield strength, ductility and fracture type, by a desired distribution of particles in the matrix, desired properties can be achieved. In this regard, controlling the fabrication parameters of primary FGC samples is of great importance. Therefore, the prerequisite to predict and achieve desirable tensile properties and wear behavior of FGC samples can be achieved through control of microstructure parameters. Hence, a desired microstructure can be achieved through control of production parameters such as heat treatment time and temperature during liquid Al and solid Ti interaction.

5 Conclusion

This paper studied the effect of spatial distribution and morphology of TiAl_3 particles on the mechanical properties and the wear resistance of Al- TiAl_3 functional gradient composite. Based on the morphology and spatial distribution of TiAl_3 particles, three distinctive regions in microstructure of FGC were studied and the following major conclusions were obtained:

1. Based on the spatial distribution of TiAl_3 particles in Al matrix, hardness, tensile properties and wear performance were varied.
2. Increment in area fraction of blocky TiAl_3 particles in the microstructure resulted in increment in yield strength, UTS and wear resistance. However, blocky-particle region showed a decreased ductility compared to the mixed-particle region.
3. Mechanism for the fracture of blocky-particle region was determined to be brittle due to low interparticle spacing while it was ductile fracture for mixed-particle region due to higher interparticle spacing. The fracture mechanism for plate-particle region was cleavage.
4. Morphology of TiAl_3 particles had a considerable effect on tensile and wear behavior of Al– TiAl_3 functional gradient composite. High aspect ratio of particles including high length to width (i.e., lengthy plate particles) was significantly deteriorating mechanical and wear performance of FGC.
5. The blocky-particle region possessed the highest yield strength and the lowest wear rate values. The wear performance of this region was highest among other regions.
6. The mixed-particle region in FGC with both blocky and short plate TiAl_3 particles exhibited the highest ductility with rather high tensile strength and wear performance.
7. The plate-particle region with high-aspect-ratio lengthy plate particles showed the minimum tensile strength as well as lowest wear performance compared to blocky and mixed regions.
8. The dominant wear mechanism for blocky and mixed-particle regions was determined to be mild oxidation while it was delamination wear for plate-particle region.

Author's contribution Hamid Doostmohammadi contributed to conception and main idea. Maryam Yousefi designed and performed the experiments. Maryam Yousefi was involved in literature survey. Hamid Doostmohammadi was involved in supervision. Maryam Yousefi collected the data. Hamid Doostmohammadi analyzed and interpreted the data. Hamid Doostmohammadi was involved in major parts of writing. All authors reviewed and approved the final manuscript.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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