Investigation on the elevated-temperature mechanical properties of TiB$_2$/Al-4.5\%Cu composites

Yanqing Xue$^1$, Ru Su$^2$, Dian Wei$^1$, Bo Li$^1$, Xinliang Wang$^1$, Han Zhang$^1$, Qitang Hao$^1$

$^1$State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi’an, Shaanxi, 710072, China
$^2$School of Materials Science and Engineering, Hebei University of Science and Technology, Shijiazhuang, Hebei 050018, China

*e-mail: yqxsue666@163.com, bemail: sxru2008@163.com, cemail: 2734595813@qq.com, demail: 1096516577@qq.com, eemail: 1054470459@qq.com, femail: laraine0222@163.com

*corresponding author; *email: haoqitang@nwpu.edu.cn

Abstract. Mechanical performance of TiB$_2$/Al-4.5\%Cu composites at elevated-temperature were evaluated by means of tensile behaviors and fracture mechanisms. Results indicated that the composite reinforced by nano-sized TiB$_2$ particles exhibited excellent mechanical ability during deformation at elevated temperatures, the optimum addition level for dispersion, refinement and modification was 5wt.\% TiB$_2$/Al-4.5\%Cu at 473K, i.e., yield strength is 358MPa and elongation is 4\%. The enhanced resistance of the composite was mainly attributed to Orowan strengthening and load transfer enhancement, additionally, interface debonding increasingly affects the damage and fracture of the composites.

1. Introduction

On account of green energy conservation and sustainable development, Lightweight and structural function integration particles reinforced aluminum-copper matrix composites (PRACMCs) have become a much-talked-about topic due to high specific strength, favourable fatigue resistance as well as clean and strong interfaces between reinforced particles and α-Al matrix[1]. Copious literatures further devote to study the grain refinement of composites aiming at producing high-quality components via metal forming operations, which are roughly divided into physical methods and chemical methods[2]. The physical method refines the grain by introducing physical factors into the solidification process, while the chemical method mainly refines the grain by adding grain refiner to the aluminum alloy and, TiB$_2$ particles have been previously investigated as one of the most common heteronuclear agents in industrial production, vast attempts have carried out to explore the effect of TiB$_2$ particles size, shape, and mass fraction on the mechanical behavior and damage mechanism of TiB$_2$/Al-4.5\%Cu composites. Shaik Mozammil et al.[3] found that TiB$_2$ is the most stable phase in the Al-4.5\%Cu composites because it requires lower free energy as compared to others, moreover, increasing the mass fraction of the reinforced particles can continuously improve the strength of the composite. P. Gurusamy et al.[4] investigated the mechanical properties of TiB$_2$/Al2219. Lei et al. [5] tested the properties of TiB$_2$/ZL205 composites and shown prominent performance. In the more recent literature, P. Mair et al.[6] produced the crack-free samples with TiB$_2$/Al-4.5\%Cu composites via laser powder-bed fusion, the
novel fine-grained microstructure results in the as-built state in a tensile strength of 401 ± 2 MPa and total elongation at fracture of 17.7 ± 0.8%. Numerous documents have committed to studying the mechanical properties at atmospheric temperature. Nevertheless, researches on elevated-temperature performance properties, e.g., mechanical behaviors and fracture mechanisms, of in-situ PRACMCs are scarce, and the effects of temperature on the formability and fracture have not been fully understood. In this paper, elevated-temperature mechanical properties of TiB2 /Al-4.5%Cu composites was conducted, i.e., temperature range of 473K and 493K, relationship between microstructure and mechanical properties was analyzed, along with the strengthening mechanism.

2. Methods and experimental procedures

TiB2 /Al-4.5%Cu composites with various mass fraction of TiB2 are fabricated by in-situ salts-metal reaction (SMR). Firstly, Commercial pure K2TiF6, KBF4 (99.6%) and other ingredients in precise proportion are used as raw materials and synthesized at 860°C in an electrical resistance furnace by following strict procedures[7], and then, the composite melt is casted into a steel mould for cooling after the chemical reaction completed. Secondly, Standard fatigue sample plates are cut from equal-axis of ingot and subjected to T6M heat treatments (450°C /4h+540°C /3h+water quenching+300°C /12h+470°C /30min+170°C /18h). Microstructure and fractography of conditioned samples are examined by scanning electron microscope (SEM) with MLA FEG650, operated at 25kV, more details are characterized using Double Spherical Aberration Corrector Transmission Electron Microscope (TEM) equipped with energy dispersive spectroscopy (EDS) system to acquire (HR) TEM, operated at 200kV. The average particle size is measured by using particle size analyzer. The tensile test is performed with a constant tensile speed of 1 mm /min in electronic universal testing machine (GNT100) according to ASTM E8 standards.

3. Results & Discussion

3.1 Microstructure characterization

Fig. 1 presents microstructure of the TiB2 /Al-4.5%Cu under T6M heat treatment. It can be observed that the size of α-Al significantly decrease with the TiB2 mass fraction increase, gathered TiB2 has been observed along grain boundary, this demonstrates that with the increase of TiB2, its distribution in matrix appears inhomogeneous. Fig 1 (a) shows the distribution of TiB2 in the 2 wt.% TiB2 /Al-4.5%Cu, As can be observed, TiB2 nanoparticles disperse in matrix uniformly. Fig 1 (b) presents microstructure of 5 wt.% TiB2 /Al-4.5%Cu. Still, some TiB2 distribute in the matrix uniformly, as marked by arrows. However, continued to increase TiB2, as seen in Fig.1 (c-d), α-Al grain refinement is more pronounced along with the serious TiB2 agglomeration, and more TiB2 are also found gathering along grain boundary, which is due to the pushing of crystallization front for TiB2 during solidification [5].

![Fig. 1. Macrostructures of composites](image-url)
As observed in Fig. 2, SEM images show discontinuous TiB₂ reinforcement, non-uniform in size and uniformly dispersed. Fine particles exhibit better mechanical properties compared to coarse ones. Further high nucleation and sluggish development rate of TiB₂ result in the finer size as the TiB₂ increase, Fig. 2 (b) clearly show the decent interfacial attachment among the α-Al matrix and reinforcement which has also been explained through TEM results in Fig. 3. The existence of a clear interface can be assigned to the thermodynamic stability of TiB₂ particles especially at elevated-temperature. More critically, the ratio of Ti:B is strictly controlled as 2.2:1 in the work, owing to the excess amount of Ti increases the formation of Al₃Ti and in the similar note, more amount of B will increase the chance of formation of Al₃B, which will be coarsening during the high-temperature circumstances.

Fig. 2. Distribution, shape, size of TiB₂ particles and interfacial attachment with the α-Al matrix under T6M heat treatment. (a) A1; (b) A2; (c) A3; (d) A4, respectively.

To understand the exact morphology and interfacial behaviour of TiB₂ with Al matrix, TEM have been carried out in Fig. 3 (5 wt.% TiB₂ /Al-4.5%Cu). As observed, majority of TiB₂ particles are hexagonal and cubic prisms structure, on account of favoured excessive growth along its own axis. Except for the inerratic nanoscale TiB₂ particles, Fig. 3(a) shown the high density dislocation, which could offer more crack propagation path during stretching exercise. The outstanding homogeneous distribution and thermodynamic stability of TiB₂ particles in Al matrix are of key contributions.

Fig. 3. (a) TEM images of dislocations around TiB₂ nanoparticles under T6M heat treatment, (b) homogeneous distribution of TiB₂ and selected area electron diffraction (SAED) pattern.

3.2 Tensile properties

Fig. 4 presents the tensile properties of the TiB₂ /Al-4.5%Cu composites at 453K (Fig. 4(a)) and 493K (Fig. 4(b)), the results show that composites under high temperature mechanics performance is excellent, not only strength increased significantly, elongation remain in a level and it, is evidently, the yield strength (YS) improve continually to some extent as the TiB₂ increases both at 453K and 493K. Nonetheless, in the 10 wt.% TiB₂ /Al-4.5%Cu composites, the value significantly decrease to 241 MPa and 197 MPa respectively, leading cause is the badly TiB₂ agglomeration along the grain boundary, as
the Van Der Waals Force between TiB$_2$ is tiny, agglomeration break could form a lot of voids, which is the source of crack. As shown in Fig.3 (a), this dislocation entrapped between the TiB$_2$ particles and felt like pinning effect in the dislocation dynamics played a critical role towards enhancing the mechanical property when exposing the materials during T6M heat treatment at elevated temperature.

![Fig. 4 Tensile properties of the TiB$_2$ /Al-4.5%Cu composites at 453K (a) and 493K (b)](image)

Fig. 4 Tensile properties of the TiB$_2$ /Al-4.5%Cu composites at 453K (a) and 493K (b)

Fig. 5 shows the fracture surfaces of the tested specimens. Under the considered deformation condition, all specimens show the typical features of ductile fracture, i.e., the fracture surfaces are of equiaxial dimples and cavities, as well as obvious necking before fracture, the least dimple size was observed in 5 wt.% TiB$_2$ /Al-4.5%Cu composites (Fig. 5(c) because of refinement in reinforced TiB$_2$ particles, whereas contradictory observation discerned in Fig. 5(d), markedly shown brittle fracture surface. As observed, TiB$_2$ particles are seen mostly inside the dimples, and exist good bonding with matrix, so it still maintains a certain performance at high-temperature.

![Fig. 5. Fracture surface of composites at 493K, (a) A1; (b) A2; (c) A3; (d) A4, respectively.](image)

3.3 Strengthening mechanism

It is well known that increased YS in PRACMCs could be attributed to combination of the factors: (i) Grain refinement strengthening, (ii) Orowan strengthening, (iii) Load-bearing strengthening, (iv) Coefficient of thermal expansion mismatch strengthening (CTE).

In this research, the average grain size exceed 50 μm, Researches indicated [8] that since Al alloys have a low k value, grain sizes of 10 μm and less will be required to influence the yield strength significantly in Al MMCs. Thereby, the effect of Hall-Petch strengthening[9]is not added.

Load-bearing strengthening results directly from the load transfer from the α-Al matrix to hard TiB$_2$ particles, and the value can be calculated by[10]:

$$\Delta \sigma_{\text{load}} = \frac{1}{2}V_p \sigma_m$$

(1)

where $V_p$ is the volume fraction of reinforced particulates, and $\sigma_m$ is the YS of matrix.

CTE mismatch between α-Al and TiB$_2$ particles can lead to the enhanced dislocation density in the matrix, which makes plastic deformation of composites more difficult, and can be presented by[10]:

$$\Delta \sigma_{\text{CTE}} = \frac{\alpha L}{L_0}$$

(2)

where $\alpha$ is the CTE of matrix, $L$ is the length of specimen, and $L_0$ is the original length of specimen.
\[ \Delta \sigma_{\text{CTE}} = 1.25G_m b \sqrt{\frac{12(\Delta \text{CTE})}{b d_p (1-\nu)}} \]  
\[ (2) \]

where \( G_m = 0.5E_m(1+\nu)^{-1} \), \( E_m \) and \( \nu \) are the Young’s modulus and Poisson’s ratio of the matrix; \( b \) is the Burgers vector of the dislocations; \( (T_{\text{melt}} - T_{\text{room}}) \) is deviation between melt and room temperature; \( (\alpha_m - \alpha_p) \) is the difference between the CTE of matrix and TiB₂, and \( d_p \) is particle size.

Orowan strengthening is induced by the interaction between TiB₂ particles and the dislocations in the matrix and, can be presented by[10]:

\[ \Delta \sigma_{\text{Orowan}} = \frac{0.13G_m b}{d_p[(1/2\nu)^{3/2} - 1]} \ln \frac{d_p}{2b} \]  
\[ (3) \]

Where \( G_m \), \( b \), \( d_p \), and \( \nu \) are the same as the above-mentioned parameters.

Good agreement between the theoretical and experimental YS was obtained[8], suggesting that the strengthening formula was available for predicting increased YS. In this research, the T6M treatment could improve the dispersion of TiB₂ particles at the grain boundaries. Introducing an external-field into the preparation process might be an effective method, and related work will be carried out in the future.

4. Conclusions

TiB₂/Al-4.5%Cu composites were successfully fabricated via SMR, main conclusions are as following:

(1) With the increase of TiB₂ mass fraction, α-Al dendrites have been significantly refined, and the TiB₂ particles were small in size and homogeneously distributed in the matrix around by tight dislocations as confirmed through scanning and transmission electron microscopy.

(2) Tensile properties of the composite under high temperature mechanics performance is excellent, not only strength increased significantly, elongation remain in a level, which were much affected by the reinforcement content and T6M heat treatment process, the optimal was found in the 5wt.% TiB₂/Al-4.5%Cu at 473K,i.e., YS is 358MPa and elongation is 4%.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (51375391, 51701064), Natural Science Foundation of Hebei Province (E2018208126), the Key Research and Development Plan of Shaanxi Province (2020GY-117), Special Fund for Research and Development of Hebei Province.

References

[1] C. Bartels, D. Raabe, G. Gottstein, U. Huber, (1997)Investigation of the precipitation kinetics in an Al6061/TiB₂ metal matrix composite Mat. Sci. Eng. a-Struct. 237(1) 12-23.
[2] Z. Chen, K. Yan, (2020)Grain refinement of commercially pure aluminum with addition of Ti and Zr elements based on crystallography orientation Sci Rep 10(1) 16591.
[3] S. Mozammil, R. Verma, J. Karloopia, P.K. Jha, (2020)Investigation and measurement of porosity in Al+4.5Cu/6wt% TiB₂ in situ composite: optimization and statistical modelling J Mater Res Technol 9(4) 8041-8057.
[4] P. Gurusamy, C. Breever Asington, K. Selvam, P. Arun Kumar, (2020)Mechanical properties and dry sliding wear behaviour of Al2219 alloy reinforced with TiB2 composites by stir casting route Materials Today: Proceedings.
[5] S. Lei, X. Li, Y. Deng, Y. Xiao, Y. Chen, H. Wang, (2020)Microstructure and mechanical properties of electron beam freeform fabricated TiB₂/Al-Cu composite Mater. Lett. 277.
[6] P. Mair, L. Kaserer, J. Braun, N. Weinberger, I. Letofsky-Papst, G. Leichtfried, (2021)Microstructure and mechanical properties of a TiB₂-modified Al-Cu alloy processed by laser powder-bed fusion Mater. Sci. Eng. A 799.
[7] J.W. Geng, G. Liu, T.R. Hong, M.L. Wang, D. Chen, N.H. Ma, H.W. Wang, (2019)Tuning the microstructure features of in-situ nano TiB₂/Al-Cu-Mg composites to enhance mechanical properties J. Alloys. Compd 775 193-201.
[8] J. Xue, W. Wu, J. Ma, H. Huang, Z. Zhao, (2020)Study on the effect of CeO₂ for fabricating in-situ TiB₂/A356 composites with improved mechanical properties Mater. Sci. Eng. A 786.
[9] N. Hansen, (2004) Hall–Petch relation and boundary strengthening Scr. Mater. 51(8) 801-806.
[10] Z. Liu, Z. Dong, X. Cheng, Q. Zheng, J. Zhao, Q. Han, (2018) On the Supplementation of Magnesium and Usage of Ultrasound Stirring for Fabricating In Situ TiB2/A356 Composites with Improved Mechanical Properties Metallurgical and Materials Transactions A 49(11) 5585-5598.