FABRICATION PROCESS OF 3D NETWORK STRUCTURED IN SITU (Al_3Zr_p+Al_2O_{3np})/2024AL METAL MATRIX COMPOSITE USING POWDER METALLURGY

B.Kaveendran, L.Geng, G.S.Wang*, L.J.Huang

Harbin Institute of Technology, School of Materials Science and Engineering, P. O. Box 433, Harbin 150001, P.R. China
**wangguisong@hit.edu.cn*

Keywords: metal matrix composite, 3D network microstructure, Al_3Zr_p+Al_2O_{3np} reinforcements, reactive sintering

Abstract

An approach for the fabrication of an in situ (Al_3Zr_p+Al_2O_{3np})/2024Al composite with a quasi-continuous 3D network reinforcement architecture using the powder metallurgical technique has been suggested. The network structure was obtained by an in situ reaction between 2024Al and ZrO_2, with subsequent synthesis of Al_3Zr and Al_2O_3 particulate around the boundaries of the large Al particles, using low energy ball milling and reactive sintering. With a reinforcement volume fraction of 10vol. %, the macrohardness and tensile strength of the composite were found to be 54.8 HRB (97HB) and 261 MPa, respectively.

1 Introduction

The combination of good properties, low cost, high workability and significant increase in performance over unreinforced alloys, have made aluminum metal matrix composites a commercially very attractive system [1]. The past few decades have seen the rapid development of homogeneously reinforced metal matrix composites, but in recent times there have been concerted efforts dedicated to the careful tailoring of three dimensionally networked microstructures such as bi-continuous, interpenetrating and quasi-continuous composites [2, 3]. The ability to design and fabricate networked three dimensional (3D) microstructures raises the possibility of developing materials with superior performance characteristics; each phase contributing its own characteristics to the overall properties of the composite [4]. However, composites with 3D continuous networks face certain difficulties such as, forming the matrix or reinforcement into a porous network structure following the impregnation of one material into the other, the complexity of varying the volume fraction of the phases and easy crack propagation along the continuous phase of the composite [5]. On the other hand, quasi-continuously networked composites have relatively simpler processing techniques and display exceptionally good properties[3, 5].

Transition metal tri-aluminides such as Al_3Zr and Al_3Ti having, thermal equilibrium existing between them and the matrix, high elastic moduli and low density, have made them good candidates for the in situ reinforcement of Al and Mg alloys. Al_3Zr exists in thermodynamic equilibrium, unlike other ceramic particulates, with the Al matrix - a real chemical bonding

between the Al and trialuminide reinforcement rather than an intermediate reaction zone. Its excellent resistance to oxidation and corrosion, high melting temperature (1580˚C) and thermal stability along with a relatively high elastic modulus (205 GPa) has made it a potential reinforcement to augment the properties of Al and its alloys. It has also been demonstrated that at a similar volume fraction of reinforcement the strengthening effect of Al_3Zr particles in a pure Al matrix is equivalent to that of 5 and 250 µm SiC particulates [6-8]. In order to enhance performance characteristics when compared to composites with homogeneous microstructures, and tackle the challenges posed by composites with 3D continuous networks, an attempt at fabricating a composite with a quasi-continuous network structure consisting of in situ synthesized Al_3Zr_p + Al_2O_{3np}, using powder metallurgy has been made.

2 Materials and Testing Methods

2.1 Materials

2024Al alloy powders (50-150 µm) and commercially pure ZrO_2 powders (0.5 -0.8µm) were used for the fabrication of this composite. The elemental composition of the 2024Al used is given in Table 1.

Cu	Mg	Si	Fe	Mn	Zn	Al
4.5	1.14	0.5	0.3	0.8	0.1	Balance

Table 1. Chemical composition of 2024Al (wt. %)

2.2 Low energy ball milling & reactive hot pressing

Initially, to produce a composite with reinforcement volume fraction of 10 vol. %, 2024Al (92.1 wt. %) and ZrO_2 (7.9 wt. %) powders, were weighed and mixed using a low energy ball milling process, according to the expected reaction equation [9]:

$$Al+ZrO_2 \rightarrow Al_3Zr+Al_2O_3 \quad (1)$$

The process was carried out under a high purity argon atmosphere, at 150 rpm and with ball to powder ratio of 5:1 for 2 hours. As a result of low energy ball milling, the ZrO_2 powders were evenly dispersed on the surface of the 2024Al particles as shown in Fig. 1.

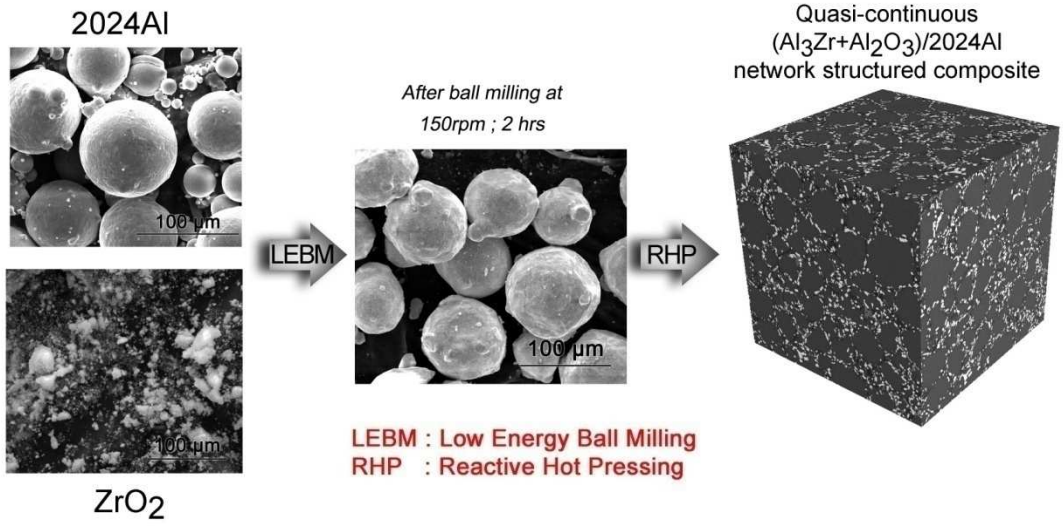

Figure 1. Fabrication process for (Al_3Zr_p+Al_2O_{3np})/2024Al networked composite.

The milled powders were then transferred to a graphite mould and sintering was commenced by applying a pressure of 25 MPa at 600˚C for 30 minutes to achieve good compaction. Following which the temperature was raised and held at 850˚C under no pressure for a period of 1 hour giving sufficient time for completion of the reaction Eq. (1). Subsequently a pressure of 25 MPa was reapplied during cooling at 600˚C for 30 minutes to effectively compact the reacted components. For meaningful comparison monolithic 2024 Al powders were also sintered at 570˚C under a pressure of 25 MPa for one hour. The sintering procedures were carried out under a high vacuum of 10^{-2} Pa and a heating rate of 10˚C/min.

2.3 Microstructural examination and testing

X-ray diffraction (XRD) analysis was done using Philips X'pert. Hardness tests were carried out using a Rockwell hardness testing machine - B scale 1/16 inch diameter (1.588 mm) steel sphere, a load of 100kgf and loading time of 60 seconds. The hardness value mentioned is an average of 5 indentations. Tensile tests were carried out on samples of gauge length 15mmx 5mmx2mm, using an Instron-5569 universal tensile testing machine with a constant crosshead speed of 0.5 mm/min. Scanning electron microscopy (SEM, Quanta 200FEG) and transmission electron microscopy (TEM, TECNAI F30) along with energy-dispersive x-ray spectroscopy (EDX) were used to perform microstructural examinations.

3 Results and Discussion

Fig. 2 shows the XRD pattern of the as-sintered composite which points out the sole presence of Al and Al_3Zr phases and the absence of ZrO_2, which indicates the completion of the in situ reaction between Al and ZrO_2.Zhu et al. [9] have indicated that the standard Gibbs free energy for Eq.(1) is negative and therefore the reaction can take place spontaneously. However, the anticipated Al_2O_3 phase according to Eq. (1) is not detected in the XRD pattern, the probable reason being the in situ synthesized Al_2O_3 phase is too fine and too little.

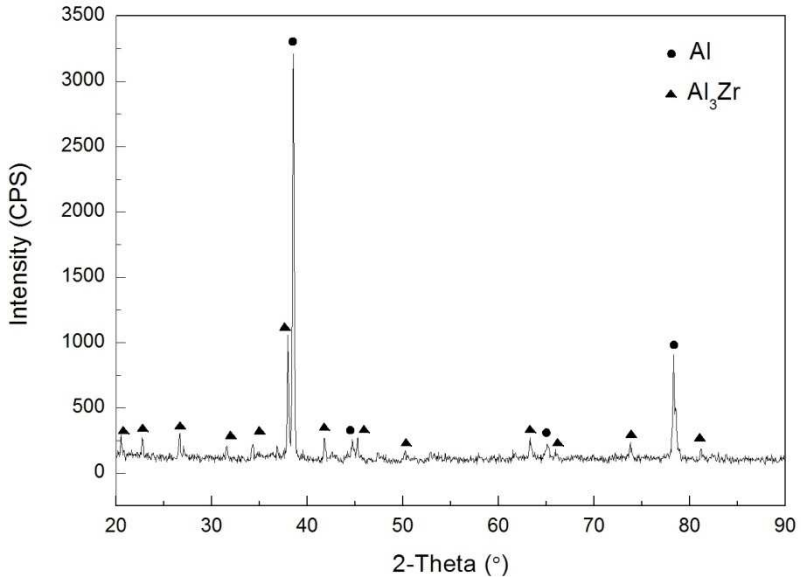

Figure 2. XRD spectra of $(Al_3Zr_p+Al_2O_{3np})$/2024Al composite

Microstructural examination on the composite using SEM clearly reveals the presence of two distinct regions as shown in Fig. 3(a) and (b). Firstly the particle lean matrix region (R1) and secondly, the particle rich reinforcement network (R2). R2 forms the skeletal structure of the composite, which is a 3D quasi-continuous network of dispersed components. The possible

formation of the network structure can be explained as follows; a chemical reaction as described in Eq. (1) takes place only on the periphery of the large 2024Al particles resulting in the formation of a network made up of dispersed Al_3Zr and Al_2O_3 particles. However, a large portion of the Al particles are undisturbed by the chemical reaction thereby making it to be reinforcement scarce regions. This phenomenon is clearly illustrated in Fig. 3(b). As shown in Fig. 4(a) the morphology of the Al_3Zr particles in R2 are mostly polyhedron and rectangular which are in agreement with Zhao et al. [10] The size of the Al_3Zr particles along the network varies between 5-20µm.

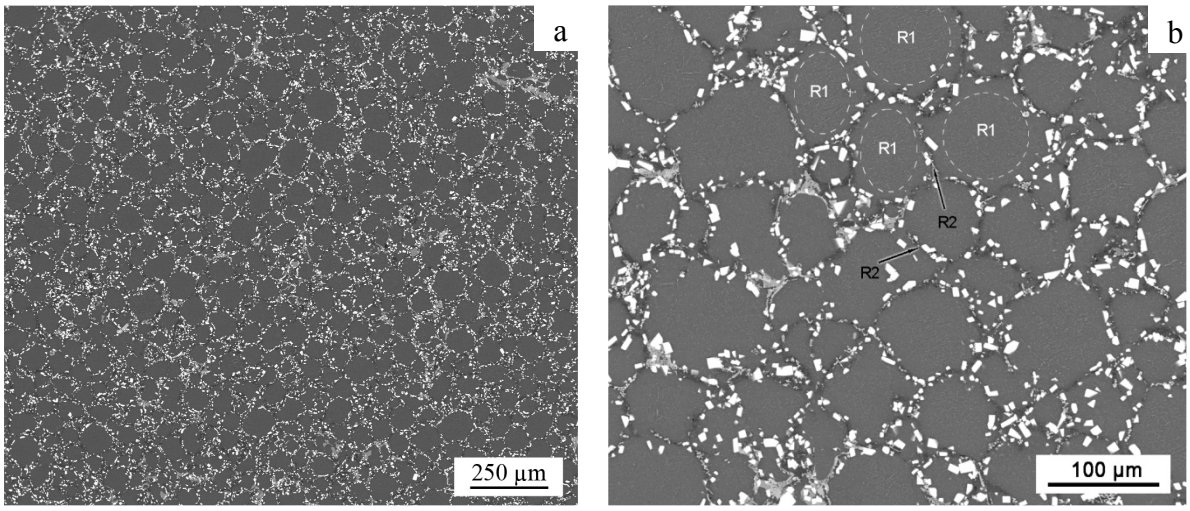

Figure 3. Microstructure of the as-sintered (Al_3Zr_p+Al_2O_{3np})/2024Al networked composite (a) low magnication (b) high magnication

According to Eq. (1) Al_2O_3 phase must be present in the composite but the XRD spectra and the SEM images do not verify the presence of the Al_2O_3 particles. However, the TEM and EDX analysis performed on the microstructure, as shown in Fig. 4(b) confirms the presence of nano particles comprising of Al and O. Therefore it can be concluded that this Al and O rich phase corresponds to Al_2O_3. It is to be noted that as the matrix used is an Al alloy, there are certain remnants consisting of precipitates and intermetallics formed during this process.

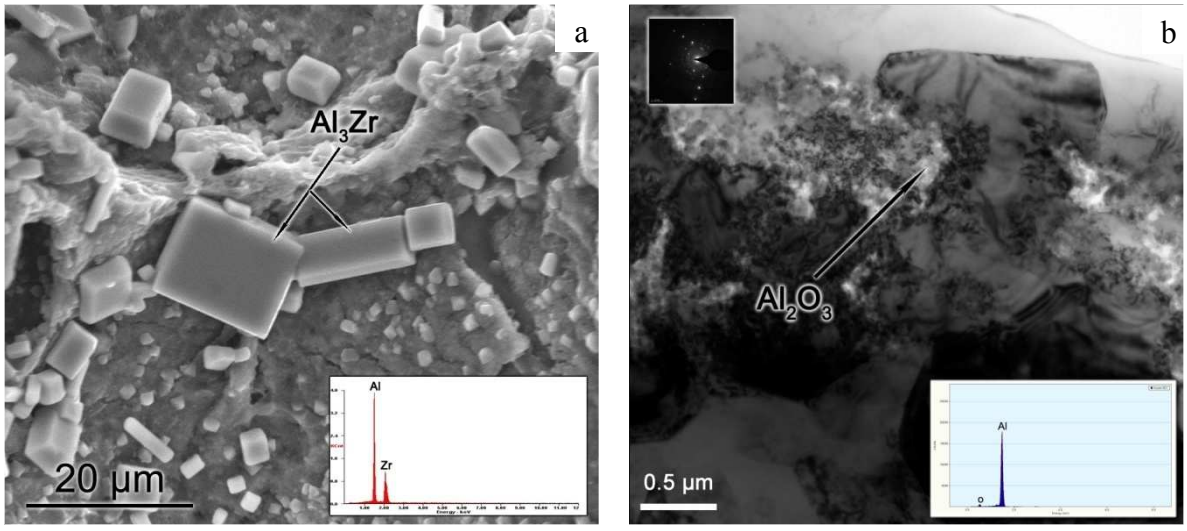

Figure 4. (a) SEM and EDX showing the morphology of Al_3Zr particles and (b) TEM image and EDX showing the morphology and distribution of the Al_2O_3 nano particles.

This has not been discussed in detail because it has been demonstrated that homogeneously reinforced composites consisting of a 2024Al alloy matrix can be suitably heat treated to further enhance its properties [11].

With a reinforcement volume fraction of 10vol. % the rockwell hardness of the composite is 54.8 HRB. The tensile characteristics of the composite are shown in Fig.5. The composite exhibits yield strength (YS) of 175 MPa, ultimate tensile strength (UTS) of 261 MPa and an elongation of 2.3%, whereas the monolithic alloy has a YS of 105 MPa, UTS of 220MPa and elongation of 12%. In other words the YS and UTS of the composite depicts an increase by 65.71% and 18.18% relative to that of the monolithic 2024 Al alloy.

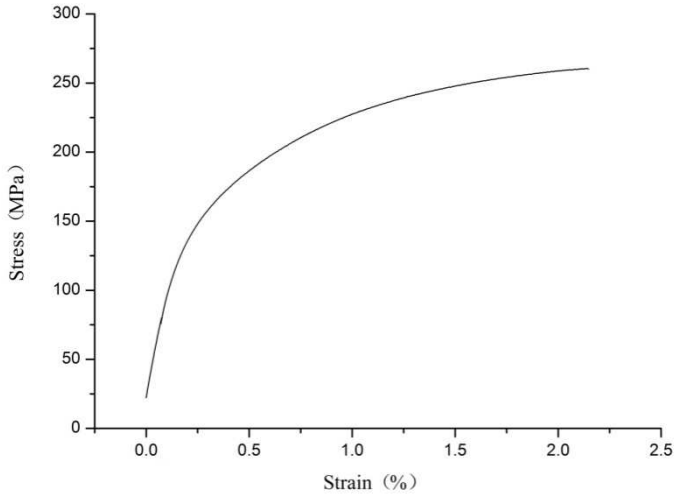

Figure 5. Tensile characteristics of the as-sintered (Al_3Zr_p+Al_2O_{3np})/2024Al networked composite

4 Conclusions

This work demonstrates the successful fabrication of a 3D quasi-continuous network structured composite using powder metallurgy. As a result of the low energy ball milling and reactive hot pressing between 2024Al and ZrO_2, particle Al_3Zr and Al_2O_3 were synthesized around the larger 2024Al particles to form the reinforcement network. A superior strengthening effect has been explored by tailoring a (Al_3Zr_p+Al_2O_{3np})/2024Al metal matrix composite with a novel network microstructure.

References

[1]T.W.Clyne and P.J.Withers. *An introduction to metal matrix composites*. Cambridge University Press, (1993).

[2]F. Scherm, R. Völkl, S. van Smaalen, S. Mondal, P. Plamondon, G. L'Espérance, F. Bechmann and U. Glatzel. Microstructural characterization of interpenetrating light weight metal matrix composites. *Materials Science and Engineering: A*, **518**, pp. 118-123 (2009).

[3]L. J. Huang, L. Geng, H. X. Peng and J. Zhang. Room temperature tensile fracture characteristics of in situ TiB_w/Ti6Al4V composites with a quasi-continuous network architecture. *Scripta Materialia*, **64**, pp. 844-847 (2011).

[4]H. X. Peng, Z. Fan and J. R. G. Evans. Bi-continuous metal matrix composites. *Materials Science and Engineering: A*, **303**, pp. 37-45 (2001).

[5]Katsunori Yamada and N. Kamiya. Composite material having discontinuous three-dimensional network structure and production thereof. USA, **US6,228,481 B1**. (2001)
[6]R.A.Varin. Intermetallic-reinforced light-metal matrix in-situ composites. *Metallurgical and Materials Transactions A*, **33A**, pp. 193-201 (2002).
[7]S. El-Hadad, H. Sato and Y. Watanabe. Wear of Al/Al_3Zr functionally graded materials fabricated by centrifugal solid-particle method. *Journal of Materials Processing Technology*, **210**, pp. 2245-2251 (2010).
[8]M. S. Zedalis, M. V. Ghate and M. E. Fine. Elastic moduli of Al_3Zr. *Scripta Metallurgica*, **19**, pp. 647-650 (1985).
[9]H. Zhu, J. Min, J. Li, Y. Ai, L. Ge and H. Wang. In situ fabrication of (α-Al_2O_3+Al_3Zr)/Al composites in an Al–ZrO_2 system. *Composites Science and Technology*, **70**, pp. 2183-2189 (2010).
[10]Y. T. Zhao, X. N. Cheng, Q. X. Dai, L. Cai and G. X. Sun. Crystal morphology and growth mechanism of reinforcements synthesized by direct melt reaction in the system Al–Zr–O. *Materials Science and Engineering: A*, **360**, pp. 315-318 (2003).
[11]N. E. Bekheet, R. M. Gadelrab, M. F. Salah and A. N. Abd El-Azim. The effects of aging on the hardness and fatigue behavior of 2024 Al alloy/SiC composites. *Materials & Design*, **23**, pp. 153-159 (2002).