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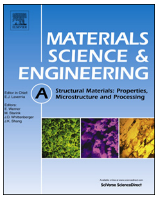

Investigation on particle strengthening effect in in-situ TiB_2/2024 composite by nanoindentation test

Jie Su, Yazhi Li*, Min-Ge Duan, Saiheng Liu, Ke Liu

School of Aeronautics, Northwestern Polytechnical University, Xi'an 710072, PR China

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ABSTRACT

Mechanical properties of in-situ 5.0 vol% TiB_2 particle reinforced 2024-T4 alloy matrix composite (in-situ TiB_2/2024-T4 composite) synthesized through exothermic reaction process are experimentally investigated using different approaches. Standard uniaxial tension tests are conducted, and SEM fractography is made to characterize the fracture mechanisms. The nanoindentation experiment and Oliver-Pharr method are employed to obtain the micro mechanical properties (indentation modulus and hardness). To carry out comparative study, all kinds of experiments have also been done for 2024-T4 alloy. The indentation results demonstrate that compared to 2024-T4 alloy, the dispersive in-situ TiB_2 nano particles do not evidently alter the Young's modulus of Al matrix of the composite, but elevate the hardness of it by 33.7%, which further means that the increase of Young's modulus of this composite over the 2024-T4 alloy is mainly due to the high stiffness of TiB_2 particle, and the increase of yield strength and ultimate strength of the composite can be attributed to microstructure strengthening to its Al matrix.

1. Introduction

With the increasing pursuit of light weight and high strength in structural applications, such as aviation and aerospace vehicles [1–3], automobiles [4] and sport equipments [5], particle reinforced aluminum matrix composites (PRAMCs) have attracted substantial attention of researchers and engineers. Taking the full advantages of excellent ductility of aluminum (or its alloys) and high stiffness of ceramic particles, PRAMCs have shown higher specific strength, better abrasion resistance and thermal conductivity and lower thermal expansion coefficient compared with their corresponding aluminum alloy. In perspective of manufacturing, superiority of PRAMCs also includes their good machining performance [6]. Moreover, compared to carbon fiber reinforced resin matrix composites, PRAMCs are easier to be moulded into complex geometry and recycled for reutilization [7].

In terms of material preparation methods, PRAMCs can be classified into two types, ex-situ and in-situ [8]. The main advantage of in-situ reaction process to prepare PRAMCs is the coherent or semi-coherent particle-matrix interface can efficiently transfer load from matrix to particles, thereby enhancing particle strengthening effect [9].

This paper studies the mechanical properties of in-situ 5.0 vol% TiB_2 particle reinforced 2024-T4 Al alloy matrix composite (in-situ TiB_2/2024-T4 composite). A variety of standard tests can be carried out to measure different aspects of mechanical properties, such as elastoplastic properties [10], fracture toughness [11], creep properties [12] and so on. However, for the PRAMCs such as the in-situ TiB_2/2024-T4 composite containing two or more heterogeneous phases, the mechanical properties of each phase cannot be directly measured by conventional techniques. Nanoindentation experiment is one of the effective means to implement such tough tasks [13,14]. As a rigid indenter (always made up of diamond) is stick into the flat surface of the material being tested, the indentation load-depth curves is recorded and then analyzed to obtain mechanical properties such as elastic modulus and hardness of different phases by using the appropriate methods [15–17].

To investigate the particle strengthening effect in in-situ TiB_2/2024-T4 composite, this research is undertaking a multiscale investigation of the material via the combination of conventional test methods with metallographic observation and nanoindentation experiment, and with the aid of elastoplastic finite element (FE) analysis. A comparison of mechanical properties between the Al matrix of the composite and the pure 2024-T4 alloy is carried out to reveal the strengthening mechanisms of TiB_2 particle to elastoplastic mechanical properties of the composite.

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* Corresponding author.
E-mail address: yazhi.li@nwpu.edu.cn (Y. Li).

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2. Experimental procedure

2.1. Material

The in-situ TiB_2/2024-T4 composite investigated in this paper is developed and provided by State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University of China. It is synthesized via an exothermic reaction of Al alloy melt and mixed salts composed of well-prepared K_2TiF_6 and KBF_4 [18]. The chemical reaction equation is shown in Eq. (1):

$$\begin{cases} 3K_2TiF_6 + 13Al = 3TiAl_3 + K_3AlF_6 + 3KAlF_4 \\ 2KBF_4 + 3Al = AlB_2 + 2KAlF_4 \\ AlB_2 + TiAl_3 = TiB_2 + 4Al \end{cases} \tag{1}$$

After as-cast ingot of this composite is obtained, homogenization treatment at 495 °C for 48 h and hot extrusion process at 450 °C with the 10: 1 extrusion ratio are successively carried on [19]. Three directions (extrusion, transverse and normal) of the hot extrusion thick plate are respectively represented as ED, TD and ND. The cross sectional dimension of the plate is 200 mm (TD) × 90 mm (ND). Finally the material is T4 heat treated with the sequent three procedures (solution treatment at 500 °C for 1 h, water quenching and natural aging for 4 days). It should be noted that the mechanical properties presented in this study are mainly in ED, although the extruded material usually exhibits appreciably orientation dependent.

2.2. SEM and TEM experiments

A sample of in-situ TiB_2/2024-T4 composite is prepared for SEM observation. The surface of the sample is polished by waterproof abrasive paper from #800 to #5000. Zeiss SUPRA 55 field emission SEM is used to observe the microstructure in ND-TD plane under secondary electron imaging mode. Another for TEM experiment is a thin slice which is polished to reduce the thickness to about 40 μm and then thinned by precision ion thinning instrument. FEI TEM is employed to provide more detailed observation on microstructure.

2.3. Uniaxial tension test and fractography

Uniaxial tension tests are carried out by using CRIMS DDL100 test machine (with full load range of 100 kN) to obtain the macro elasto-plastic mechanical properties of in-situ TiB_2/2024-T4 composite, according to ASTM E 8 M standard [20]. The stress vs. strain curves, the elongation and the section shrinkage of the composite are acquired. To figure out the fracture mechanism of this composite, SEM fractography is carried out. For contrast, the same kinds of test are also conducted on 2024-T4 alloy.

2.4. Nanoindentation experiment

A cylindrical nanoindentation specimen is cut out of the hot extrusion thick plate of in-situ TiB_2/2024-T4 composite. The same polishing process as SEM samples is conducted on its top surface. To prevent the polished surface from being scratched and oxidized during handling, it is kept in pure alcohol before the indentation test.

The nanoindentation experiment is conducted using the Agilent Nano Indenter G200 System attached with a standard Berkovich, the nominal curvature radius of which is 20 nm. The test is implemented using the custom method "G-Series Basic Hardness, Modulus, Tip Cal, Load Control". The maximum indentation load (P_{max} = 20 mN) is chosen to reduce the adverse effect of surface roughness of the test material. In view of the distinct difference of mechanical properties between Al matrix and TiB_2 particle, it is necessary to acquire the nanoindentation response of matrix and particles separately. In considering the serious heterogeneity of in-situ TiB_2/2024-T4 composite at micrometer scale and to obtain sufficient valid data, indentation array (m × n = 3 × 11) shown in Fig. 1 is dotted on the polished surface. The dent spacing D = 70 μm are set to prevent mechanical interference of the adjacent indentations. The location of the array on the test surface is randomly selected. In addition, the peak load is held for 20 s to eliminate the possible creep interfering and the allowable drift rate (0.08 nm/s) is set to avoid the error induced by the considerable thermal drift effect. Nanoindentations are made on 2024-T4 alloy accordingly for comparison. The scheme of all nanoindentation experiments is listed in Table 1.

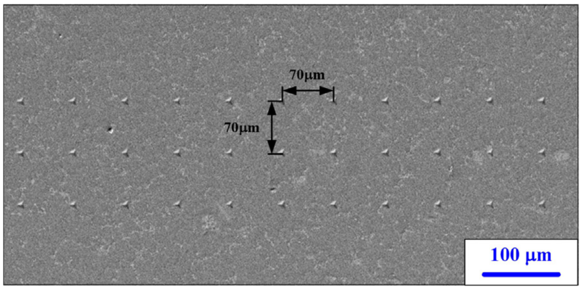

Fig. 1. Nanoindentation array (3 × 11) of in-situ TiB_2/2024-T4 composite.

The indentation load-depth curves are recorded during experiment, and the indentation modulus and hardness can be obtained using Oliver-Pharr (O-P) method [17], inspired upon Doerner and Nix's work [21] and widely used to obtain indentation modulus and hardness of the film attached to substrate [22,23] and bulk material [24,25] according to the unloading portion of indentation load-depth curve. The solution procedure is as follows:

1) Fitting the unloading curve with power function. The form of the function is

$$P = \alpha(h - h_f)^m \tag{2}$$

where, α, h_f and m are the three fitting parameters; P and h are the indentation load and depth respectively.

2) Calculating contact stiffness. The contact stiffness (S) is defined as the ratio of ΔP and Δh at the very beginning of the unloading stage and can be represented as

$$S = \left.\frac{dP}{dh}\right|_{h=h_{max}} = \alpha m(h_{max} - h_f)^{m-1} \tag{3}$$

where, h_{max} is the maximum indentation depth.

3) Determining contact depth and projected area of contact region. Taking the advantage of Sneddon 's solution [26] depicting the contour profile outside the contact region, the contact depth (h_c) and the ideal projected area of contact region (A_i) can be written as

$$h_c = h_{max} - \gamma\frac{P_{max}}{S} \tag{4}$$

$$A_i = 3\sqrt{3}h_c{}^2\tan^2\theta = 3\sqrt{3}h_c{}^2\tan^2 65.27^\circ = 24.49h_c{}^2 \tag{5}$$

where, P_{max} is the maximum indentation load; γ is a parameter related to the shape of indenter (γ = 0.75 for Berkovich indenter); θ is the angle between pyramidal face and indenter axis (θ = 65.27° for

Table 1
Scheme of nanoindentation experiments.

Material	P_{max} /mN	m × n	D /μm
in-situ TiB_2/2024-T4	20	3 × 11	70
2024-T4	25	2 × 1	70
2024-T4	26	3 × 1	70