Available online at www.sciencedirect.com

ScienceDirect

Scripta Materialia 78–79 (2014) 1–4

Scripta MATERIALIA

www.elsevier.com/locate/scriptamat

Influence of flake thickness on the shape and distribution of Al_2O_3 particles in Al matrix composites fabricated by flake powder metallurgy

Ning Guo,[a,b] Baifeng Luan,[a,*] Fangfang He,[a] Zhiqiang Li[c] and Qing Liu[a,*]

[a] *School of Materials Science and Engineering, Chongqing University, Chongqing 400030, People's Republic of China*
[b] *Faculty of Materials and Energy, Southwest University, Chongqing 400715, People's Republic of China*
[c] *State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai 200240, People's Republic of China*

Received 14 October 2013; revised 3 December 2013; accepted 24 December 2013
Available online 3 January 2014

Microstructure of in situ Al_2O_3/Al composites fabricated by flake powder metallurgy (FPM) has been characterized in the current paper. The coarsening behavior of the ball-milled Al flake powders is influenced greatly by the thickness of the flakes, which in turn affects the shape and distribution of the Al_2O_3 reinforcement particles within the Al_2O_3/Al composites. Intragranular Al_2O_3 nanoparticles with a lamellar shape in the Al matrix could be obtained by the FPM method through refining the thickness of the ball-milled Al flakes.

Keywords: Al matrix composites; Flake powder metallurgy; Nanoparticles; Intragranular particles

Al matrix composites have good prospects for application in the automobile, aerospace and electronic industries due to their lighter weight and greater strength and stiffness over conventional metals and alloys [1]. The majority of high-strength/high-hardness reinforcement additives, such as Al_2O_3, carbon nanotubes (CNTs) and SiC [2–4], could be used to enhance the strength of the Al matrix composites. Many techniques, like stir casting, powder metallurgy and even severe plastic deformation (accumulative roll bonding, high pressure torsion, friction stir processing, etc.) [5–9], have been developed in order to disperse such reinforcement additives throughout the Al matrix. However, the challenge is how to obtain well-dispersed reinforcing particles with high volume fractions in the composites, and balance the strength and ductility for high performance.

Recently, a simple and scalable methodology called flake powder metallurgy (FPM) has been proposed for fabricating Al matrix composites [10–12]. Al flake powders with reinforcing lamellae could be used as building blocks to assemble into biomimetic nanolaminated structures via compacting, sintering and extrusion. FPM is considered to be a high-efficiency mass-producing method, which can disperse reinforcement additives homogeneously in the Al matrix and balance the strength and ductility of the composites. For example, Al_2O_3/Al composites [10] and CNTs/Al composites [12] fabricated by FPM show tensile strengths of 262 and 435 MPa and plasticities of 22.9 and 6%, respectively. In this study, the microstructure of the in situ Al_2O_3/Al composites fabricated by FPM was investigated, mainly focusing on the shape and distribution of the Al_2O_3 reinforcement in the composites.

Spherical Al powders (10 µm in diameter and 99.5 wt.% in purity) were ground into flake powders by ball milling (wet milling) in an attritor at 423 rpm at room temperature for 1, 2, 4 and 5 h, respectively. The as-prepared flake powders were heated in a flowing Ar atmosphere at 400 °C for 1 h, then kept in air at room temperature for several days to obtain Al_2O_3 skins/lamellae in the surface of the flakes by in situ oxidation reaction of the Al flake powders. The Al_2O_3/Al flake powders were subjected to compacting (500 MPa), sintering (630 °C for 2 h) and hot extrusion (400 °C with an extrusion ratio of 20:1 and a speed of 0.5 mm min^{-1}) in sequence to obtain bulk Al_2O_3/Al composites (more details about the procedure are

---

[*] Corresponding authors. Fax: +86 23 65111295; e-mail addresses: bfluan@cqu.edu; qingliu@cqu.edu.cn

1359-6462/$ - see front matter © 2014 Published by Elsevier Ltd. on behalf of Acta Materialia Inc.
http://dx.doi.org/10.1016/j.scriptamat.2013.12.019

provided in Ref. [10]). The as-received bulk Al_2O_3/Al composites were fabricated and provided by State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University (Shanghai, China). The as-received composites are hereafter denoted as Al_2O_3/Al-1 h, Al_2O_3/Al-2 h, Al_2O_3/Al-4 h and Al_2O_3/Al-5 h samples according to their ball milling time. The microstructures were characterized by electron channeling contrast (ECC) imaging using a field emission gun scanning electron microscope (Zeiss Auriga). A high-resolution field emission gun transmission electron microscope (Zeiss Libra 200) was used to identify the Al_2O_3 particles. The content of Al_2O_3 particles in the composites was expressed by the area fraction of Al_2O_3 particles within the ECC images (calculated by ImageJ software). The grain width of the Al matrix was determined by electron backscattered diffraction through the intercept measurement of the Channel5 software. The specimens used for tension testing were dog-bone shaped, with the gage dimensions of 5 mm diameter and 25 mm length. The tension testing was performed at room temperature using a Shimadzu AG machine at a strain rate of 10^{-3} s^{-1}.

Figure 1 presents the microstructures of the Al_2O_3/Al composites fabricated by FPM. Typical extrusion structures featured by equiaxed and elliptical shape grains (marked in Fig. 1a) from the transverse section and elongated grains (layered structures) from the longitudinal section (Fig. 1b) can be observed. The authors [10] previously attributed such layered structures of the Al matrix to the self-assembly of the original Al flakes and considered the grain width to represent the thickness of the original Al flakes. However, it should be emphasized that the ball-milled Al flake powders were subjected to microstructure coarsening (grain growth), which is inherent to sintering [13]. This microstructure coarsening during sintering produces a dimensional change in the original Al powders. The layered structure of the Al matrix in Figure 1b is in fact the result of hot extrusion rather than the morphology of the original Al flakes. Importantly, the layered distribution of Al_2O_3 particles was retained throughout the manufacturing FPM process and the Al_2O_3 particles introduced are well dispersed throughout the Al matrix.

The distribution of Al_2O_3 particles in the Al_2O_3/Al composites is shown in Figure 2. It illustrates that the area fraction of the Al_2O_3 particles increases with increasing ball milling time, and this is responsible for the thickness of the Al flakes. Due to the repetitive microrolling during the milling process [10], the longer the ball milling time, the thinner the Al flakes will be. The original Al_2O_3 particles are introduced by the in situ method, and fine flakes have a large specific surface area (Table 1), leading to high oxidation reaction efficiency. It is important to note that the value shown in Table 1 is theoretically calculated for a single Al ball or a single Al flake according to its shape under conditions of constant volume (without fragmentation during milling). Nevertheless, the fragmentation of Al flakes is considered to occur during the milling process. Therefore, in practice, the total specific surface area of the Al flakes is much larger than that of the original Al balls.

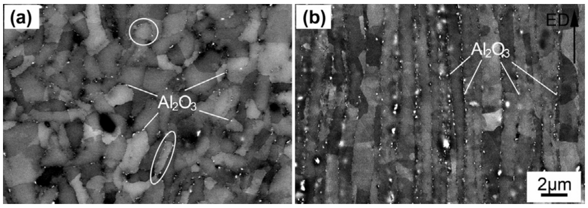

Figure 1. ECC images showing microstructures of the Al_2O_3/Al-1 h sample observed from a transverse section (a) and a longitudinal section (b), respectively.

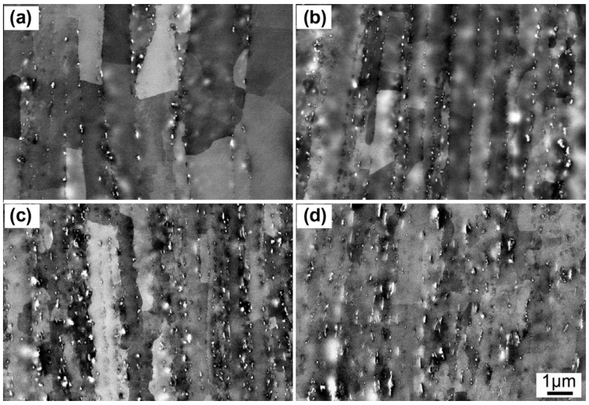

Figure 2. Distribution of Al_2O_3 particles in the composites made of Al flake powders with different thickness: (a) Al_2O_3/Al-1 h; (b) Al_2O_3/Al-2 h; (c) Al_2O_3/Al-4 h; (d) Al_2O_3/Al-5 h.

The reinforced particles of Al_2O_3 with a skin/lamella shape were formed in situ by oxidation reaction in the surface of the Al flakes. Surprisingly, as shown in Figure 2a and b, the majority of the original Al_2O_3 skins/lamellae suffered spheroidization (coarsening) during the sintering process and changed into spherical particles in the Al_2O_3/Al-1 h and Al_2O_3/Al-2 h samples obtained from coarse flake powders (thickness ranging from 1 to 2 μm). Moreover, such spherical particles of Al_2O_3 are mainly distributed in the grain boundaries of the Al matrix. In contrast, as shown in Figure 2c and d, most of the Al_2O_3 particles display a rod-like rather than spherical morphology in the Al_2O_3/Al-4 h and Al_2O_3/Al-5 h samples obtained from fine flake powders (thickness ranging from 0.2 to 0.5 μm). This indicates that the spheroidization of Al_2O_3 skins/lamellae is greater in the composites obtained from coarse flake powders. Moreover, some lamellar nanoparticles are observed inside the grains of the Al_2O_3/Al-4 h and Al_2O_3/Al-5 h samples, as shown in Figure 3a. Such intragranular lamellar Al_2O_3 nanoparticles are identified as Al_2O_3 by transmission electron microscopy (TEM) at a high resolution (see Fig. 3b). This reveals that, for the Al_2O_3/Al-4 h and Al_2O_3/Al-5 h samples obtained from fine flake powders, some of the Al_2O_3 skins/lamellae could maintain their original lamellar shape in the inside of the grain of the Al matrix during the subsequent high-temperature sintering and hot extrusion processes.

From Figure 3b, it is inferred that the thickness of the original Al_2O_3 skins/lamellae is less than 50 nm. However, the original Al_2O_3 skins/lamellae have been changed into different shapes in the final bulk Al_2O_3/Al composites (Fig. 2). Overall, FPM processing sintering needs the highest temperature and longest time. The